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1. Artículo 1. Pellet manufacturing potencial of forest residuals of *Cupressus lusitanica* and *Tectona grandis* in Costa Rica.

Pellet manufacturing potential of forest residuals of *Cupressus* lusitanica and *Tectona grandis* in Costa Rica

Pellet fabricated with C. lusitanica and T. grandis

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Summary

The process of transformation and sawmilling of sawlog generates residuals that can be used as sources of raw material for the production of pellets. The aim of this study was to evaluate the efficiency, pellet quality for x-ray photographic and the energetic, physical and mechanical properties of pellets fabricated from residuals obtained from processing sawlogs of *Cupressus lusitanica* and *Tectona grandis*. The results showed a percentage of low efficiency for both species, 25% for *C. lusitanica* and 20% for *T. grandis*. In the quality evaluation, pellets were found that pellet fabricated with *C. lusitanica* pellets is characterized by having high density areas (about 1300 kg m⁻³) and few cracks of short size on the surface. But the pellets of *T. grandis* residues present an irregular density (high and low density in the pellet) and present deeper and longer cracks. Regarding the properties evaluated, it was found that the pellets of *T. grandis* were characterized by having a lower apparent density, lower net calorific power, lower resistance to compression and lower uniformity in the density of its surface and a greater percentage of ash, moisture content and mechanical durability, if they are compared with pellets of *C. lusitanica*.

Keywords: efficiency, quality, X-rays, energetic, physical and mechanical properties.

Introduction

In the last few decades, the need to reduce emissions of greenhouse gases has increased, and with this, a variety of new energy sources have been developed worldwide (Stelte *et al.* 2011a). In this sense, a market dedicated to the manufacture of biofuels has been developed, with products such as pellets or briquettes (Lee *et al.* 2011). In the case of pellets, these greatly improve the conditions of biomass: increase energetic density, because they have low moisture content and ashes. So pellets decreases costs of transportation and storage, and allows a standardization of sizes and composition, resulting in a greater ease in feeding of domestic and industrial furnaces (Filbakk *et al.* 2011).

Moreover, residuals (shavings, sawdust and chips) produced in sawmills are the raw materials used today for the production of pellets (Stelte et al. 2011c; Stahl et al. 2011). Cupressus lusitanica and Tectona grandis are forest species widely used in commercial reforestation in Costa Rica mainly because of the quality of the wood and its rapid growth (Cornelius et al. 1996; Murillo et al. 2001). C. lusitanica is characterized by having high values of combustibility index and calorific power and low ash content (Moya and Tenorio 2013). Although T. grandis has lower values of combustibility index, its net calorific power is high (Moya and Tenorio 2013), Also this species has an important energetic potential due to high volumes of wastes or residuals obtained in different industries (Moya et al. 2014). Both species have a stages of combustion process (pyrolysis, ignition, flame end and incandescence start) clearly defined, which makes them suitable for combustion processes generate heat where needed (Tenorio and Moya 2013). Currently, there are many researches done related to the production of pellets and its features worldwide. These studies focus on various aspects: (i) manufacturing processes (Nielsen et al. 2009a; Filbakk et al. 2011), (ii) improving the conditions for adding additives or treat biomass before or during the manufacture of pellets (Nielsen et al. 2009b; Stelte et al. 2011b), such as torrefating (Stelte et al. 2011a), (iii) evaluating the quality of pellets manufacture in energetic, physical, mechanical and chemical aspects (Bergström et al. 2008; Berghel and Stahl 2009), and (iv) evaluation of the combustion process and emissions of pellets (Lee et al. 2011).

In temperate countries there is a wide variety of information about the different species used in the manufacturing process of pellets, as well as their characteristics. However, this is not the case in tropical countries, where few species have been studied and are hardly limited to eucalyptus or tropical pines (Soto and Núñez 2008). In Costa Rica, pellets have gained popularity because

some industries seek the conversion of heat production of fossil fuels from renewable sources, in order to achieve carbon neutrality.

However, these companies, so far have no knowledge about the properties of the species that could be used in the manufacture of pellets or its processing characteristics. Use of residuals obtained from the processing of sawlog of species such as *C. lusitanica* and *T. grandis* is an option for energy production through combustion processes of pellets in Costa Rica. Therefore, this research evaluates the behavior of sawlog residues of *C. lusitanica* and *T. grandis* in the production of pellets, and evaluate the characteristics of the pellets under four aspects: the quality of the pellet by X – ray radiography, energetic properties (net calorific power, ash and moisture content), physical properties (length, diameter, density, absorption and apparent density), variation in density by X-ray densitometry and mechanical properties (compressive strength and durability).

Materials and methods

Materials and provenance

Sawlog residuals of *C. lusitanica* and *T. grandis* were used. The *C. lusitanica* residuals were taken from the sawmilling process of the sawmill property of Alfredo Orozco, located in the province of Cartago (Costa Rica), while residuals of *T. grandis* were taken from the Timber Industry Abancari, located in the province of Guanacaste (Costa Rica). These residuals were formed by shavings, sawdust, chips, ribs and the leftovers of the re-sawing of the logs.

Manufacturing process of the pellets and efficiency in the process

The pellet production process was carried out in the company PELLETICS (http://pelletics.com/), located in Muelle de San Carlos, in the province of Alajuela (Costa Rica). This process is designed for manufacturing the pellets from the moment in which the material (ribs, edgings, re-sawing residues) reaches the plant until the pelletizing process. This process can be consulted at Aragón (2013). The residues were milled using a Kahl fixed ring matrix, with 15 mm diameter holes. The granulated material was then pelletized with a Kahl machine, model 38780, which consists of a fixed ring matrix with three rotating cylindrical rollers, reaching 125 °C temperature during the process.

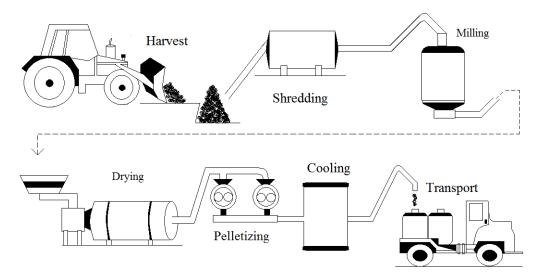


Fig. 1. Pellet manufacturing process in Agrep Forest S.A. (Aragón, 2013)

The sawlog residuals obtained in the process of sawmilling were chipped using a chipper JENZ brand, model AZ 50. Subsequently, the process of grinding the chipped material was carried out using a matrix of fixed ring with 15 mm diameter holes, KAHL brand. The granulated material was dried to a moisture content of 8% for *C. lusitanica* and 14% for *T. grandis*, through the use of a rotary drum (12 m long x 3 m) heated with hot air at 400 ° C. Finally the pelleting process is carried out in a machine KAHL brand, 35780 model, which consists of a matrix of fixed ring of 780 mm in diameter, with holes of 6 mm diameter and 30 mm in length, with three rotating cylindrical roller, which reaches a temperature of 120° C during the process.

Efficiency of the pellet production process: This efficiency refers to the amount in weight of particles that enter the pelletizing process and the amount, of pellets with size bigger than 4.75 mm. Before entering the pelletizing process, the particles were weighed. Then, the pelletized material was gathered and sieved with a 4.75 mm (mesh #4). Pellets over that size were weighed. The efficiency was determined by the ratio of the actual weight of the pellets between the weights of the particles before entering the pelletizer, expressed in percentage.

Pellets evaluation

Quality evaluation

The quality was evaluated using images obtained from exposure to X-rays, which revealed the presence of cracks in the pellets. Likewise, the quality of the pellet was analyzed using the X-ray densitometry to determine the density variation in transverse and longitudinal direction of the

pellet. These studies were conducted in 10 randomly selected pellets per crop and conditioned to a moisture content of 12%. The images of X-rays to study pellet quality were obtained by X-ray equipment (Hewlett Packard Faxitron, LX-60) at a 12 cm distance between the X-ray source and the samples. Exposure conditions were 15 seconds with 30 KV tube tension.

Determination of the energetic properties

The caloric power was determined without the presence of water (0% moisture content) and called as Net caloric Power (NCP). Ten pellets were used, selected randomly from the manufactured pellets, with a weight of about 0.60 g. NCP was determined using the norm ASTM D-5865 (ASTM 2003a). Regarding the percentage of ash, ten samples of pellets of 2 g were taken randomly from the manufactured pellets and the procedure perform is detailed in ASTM D1102-84 norm (2003b ASTM). The moisture content of the pellets was determined using a moisture balance MB45 model, OHAUS brand, which determines the moisture content of the material relative to the initial weight.

Determination of the physical properties

These characteristics of pellets was evaluated its length and diameter distribution, as well as the number of pellets in 100 grams. The bulk density, apparent density and the moisture absorption rate were too evaluated. To determine the length and diameter of the pellets, the length and diameter of 30 pellets selected at random from the total produced were measured. In the case of the bulk density of the pellets, 10 samples were taken randomly from the total produced and, using a gauge, the length and the diameter were measured, and finally the mass was evaluated (in a scale) and the equation 1 was applied. The pellet apparent density was determined using a container of known volume, then the material contained in it was weighed on a balance (0.01g precision). A 100 g of pellets were put into a recipient, and then counted to determine their number. This measurement was made 10 times with 10 different samples. For the moisture absorption test, a sample of 30 pellets was taken randomly, put simultaneously into a desiccator in a concentrated solution of potassium nitrate (equilibrium moisture content of 21%) for 14 days. The samples were weighed before and after this period. The percentage of absorption was calculated by Equation 2.

Density of the pellets
$$=\frac{weight \ of \ the \ pellets}{volume \ of \ the \ briquettes \ or \ pellets}$$
 (1)

Humidity absorption
$$=\frac{\text{initial weight-weight at }20\%}{\text{initial weight}}*100$$
 (2)

In regards to the variation of the density of x-ray densitometry in longitudinal and transverse direction, a sample of 10 pellets was used, using a x-ray scanner, brand Quintek Measurement Systems Inc., model QTRS -01X. The exposure conditions were at a voltage of 7 KV in the tube and the density readings were made during 1 second every 40 µm. The pellets were conditioned to a moisture content of 12%, then the weight, diameter and length was determined to calculate their real density. Next, the samples of cultivation were prepared horizontally on the stand included by the equipment and then these were radiographed to determine the density profile in the longitudinal direction. In determining crosswise density, they were carefully cut in cross sections of the pellet to a thickness of approximately 1.80 mm and then these were radiographed. Pellet density was measured using a X-ray scanner (Quintek Measurement Systems Inc., QTRS -01X). The exposure conditions were at a voltage of 7 KV in the tube and the density readings were made during 1 second every 40 µm.

Determination of mechanic properties

The mechanical properties of the pellets were evaluated by means of the compression test and the durability test. Compression pellets tests were evaluated following the methodology proposed by Aarseth and Prestlokken (2003). The machine has a load vs displacement record, which was used to establish the stress at the proportional limit, and the maximum force. The resistance to compression was determined by evaluating 10 pellets with a length of about 13 mm, selected randomly. This test was performed in the lateral direction of the pellet, according to the methodology proposed by Aarseth and Prestlokken (2003). The compression load is applied at a rate of 0.02 mm min⁻¹. The machine again reports the load vs. deformation of the pellet and the maximum stress (Equation 3) is calculated. The mechanical durability was performed according to the EN 15210-1 standard (CEN, 2005). Firstly, the pellets were sieved with a 3.36 mm mesh; then, they were put into a recipient until reaching 500 g weight. This weight was then put into a pellet tester (CEN, 2005), which spins at 50 revolutions per minute during 10 minutes. At the end of this period, the drum was emptied and the material was sieved again and reweighed. The mechanical durability was determined using Equation 4.

$$Compression\ stress = \frac{2 \times maximum\ load\ (Kg)}{\pi \times Diameter\ (cm) \times Length\ (cm)} \qquad (3)$$

Mechanical durability (%) =
$$\frac{\text{Pellet weight before the test}}{\text{Pellet weight after the test}} * 100 (4)$$

Statistical analysis

A descriptive analysis (mean, standard deviation, maximum and minimum values) was developed for the variables involved: length and diameter of pellets, NCV, ash content, moisture content, apparent density, absorption percentage, durability, compressive strength and longitudinal and transverse density. In addition, we checked whether the variables met the assumptions of normal distribution, homogeneity of variances, and the presence of extreme data. The method of analysis of variance (IBM SPSS Statistics) was applied to check for significant differences between the averages of the variables (P <0.05) and Tukey test was established to determine the statistical differences between means.

As for the values of X-ray density and the values provided by the equipment was corrected by the average of real density of the pellets, previously determined by the measurement their weight, length, and diameter. A correction factor was determined (Equation 3) and the density was corrected by the aid of equation 4. From the values of the corrected density, density profiles were constructed in longitudinal and transverse direction in order to determine patterns of variation.

$$Correction factor = Average density - Real density$$
 (5)
$$Corrected density = Average density - Correction factor$$
 (6)

Results

Efficiency of the pelleting process

The pelletizing efficiency (relationship of the amount of pellets produced and the weight of the particles before pelleting) for *C. lusitanica* was 25%, and in *T. grandis* the percentage was lower of 20%.

Quality evaluation

The quality evaluation using X-ray images showed that the pellets of both species have some irregularities or cracks on the surface and areas of greater clarity are observed, which appear as

white spots or stains (Fig. 2). Pellets of *C. lusitanica* (Fig. 2a and 2b) are characterized by presenting zones of greater clarity and few cracks of short size on its surface. But it was observed that in the surface of the pellets of *T. grandis* more clarity zones are presented in addition to exhibiting deeper and longer cracks (Fig. 2c and 2d).

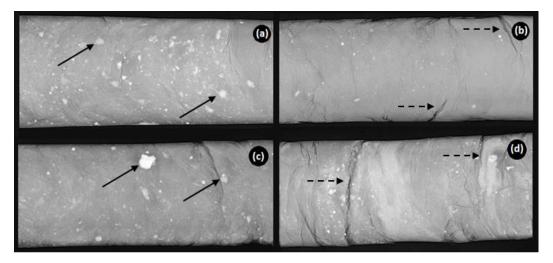


Fig. 2. X-Ray Photography of pellets of *Gmelina arborea* (a-b) and *Tectona grandis* (c-d) Note: The marker indicates the presence of cracks in the surface and the marker light area.

Energetic, physical and mechanical properties of pellets

Energetic, physical and mechanical properties are shown in Table 1 and Fig. 2, 3 and 4. It is observed that in all parameters evaluated statistical differences between species were found, with the exception of the diameter and the values of transversal and longitudinal real density (Fig. 4). Besides these differences in the mean values, a difference was also observed in the coefficient of variation (CV) of the parameters. For energetic properties, *T. grandis* presented values of CV lower than 3.67%, but in *C. lusitanica* presents a higher variation is higher especially in the moisture content (MC) and NCP (Table 1).

In the evaluation of the physical properties, pellet of *C. lusitanica* presented an average diameter of 6.16 mm and 27.27 mm in length. While *T. grandis* pellet presented an average diameter of 6.21 mm and a length of 19.78 cm (Table 1 and Fig. 3). It was not observed significant difference between both species studied. Length of pellet presented differences not only in the average of two species (Table 1), but also the distribution of the lengths and its variation were lightly

different (Fig. 3a and 3b). In *C. lusitanica* (Fig. 3a), the distribution of lengths of pellets showed a high percentage (about 76%) presents lengths between 20.5 to 32.5 cm, while a low percentage have lengths lower to 20.5 cm. The distribution of the pellets of *T. grandis* showed that a high percentage (about 90%) of the pellets have lengths of 15.5 cm to 24.5 cm (Fig. 3), while the rest were located about 24.5 to 30.5 mm. Regarding the variation of length, it is observed that pellets of *C. lusitanica* had a greater variation in their lengths of 17.5 cm to 38.5 cm, while for *T. grandis* the variation is 15.5 cm to 30.5 cm (Fig. 3a and 3b).

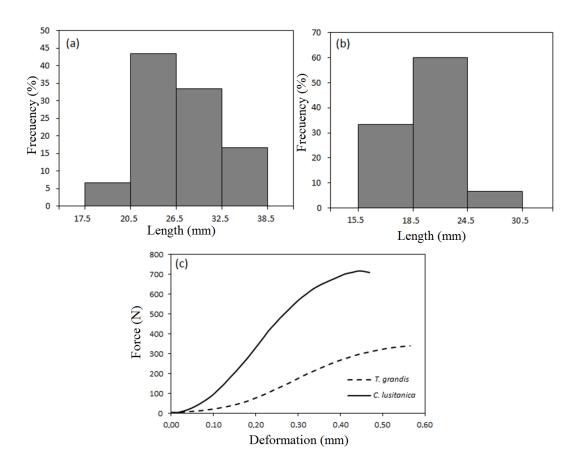


Fig. 3. Length distribution for pellets of (a) *C lusitanica* and (b) *T. grandis*, and (c) typical compressive force versus distance curves of pellets fabricated with *C. lusitanica* and *T. grandis*.

MC for *C. lusitanica* wood was statistically lower than the value obtained in *T. grandis* wood (Table 1). The absorption capacity and apparent density in the pellets of *C. lusitanica* are statistically higher than in the pellets of *T. grandis*.

Table 1. Energy, physical and mechanical properties obtained for pellets of *C. lusitanica* and *T. grandis* wood.

Properties	Parameters	Cupressus lusitanica	Tectona grandis
	Net caloric power (kJ kg ⁻¹)	16807.20 ^A (7.87)	15261.19 ^B (2.31)
Energetic	Ash percentage (%)	$1.03^{A}(0.28)$	$3.24^{\rm B}$ (0.23)
	Moisture content (%)	7.50^{A} (16.25)	12.06^{B} (3.67)
Physical	Pellet length (mm)	27.27 (18.34) ^A	19.78 (12.56) ^B
	Pellet diameter (mm)	$6.16 (1.19)^{A}$	6.21 (2.94) ^A
	Moisture absorption (%)	9.31 ^A (13.65)	3.76^{B} (21.46)
	Apparent density (kg m ⁻³)	$550.00^{A} (3.38)$	$380.00^{B} (3.66)$
	Transversal Real density (kg m ⁻³)	$1207.86^{A}(5.16)$	1135.66 ^A (7.71)
	Longitudinal Real density (kg m ⁻³)	1207.86 ^A (4.33)	1135.66 ^A (6.99)
Mechanical	Compressive stress (kg cm ⁻²)	48.93 ^A (19.01)	21.79 ^B (17.17)
	Rupture strength (N)	737.83 ^A (25.12)	393.66 ^B (16.49)
	Mechanic durability (%)	$84.74^{A}(1.98)$	$92.08^{B}(1.42)$

Note: The numbers in parentheses correspond to the coefficient of variation. Different letters mean statistical differences to $\alpha = 0.05$.

In evaluating the density by x-ray densitometry in the pellets, it was found that the average in both transversal and longitudinal direction, of *C. lusitanica* wood was statistically higher than the density value of *T. grandis* wood (Table 1). Also the variation of the density, measured by the coefficient of variation, in the pellets of *T. grandis* was higher compared with the pellets of *C. lusitanica* (Table 1). This behavior can be observed by the profiles of density variation (Fig. 4). Pellets of *C. lusitanica* showed greater uniformity than *T. grandis* pellets. This means that in *C. lusitanica* density is similar in respect of diameter and length (Fig. 4a and 4b), and instead, the pellets of *T. grandis* present greater variability in these two directions (Fig. 4c and 4d). Finally it was found that the lighter regions coincide with the highest values of density (Fig. 4c).

Regarding the mechanical properties, the compression stress and the rupture strength for pellets of *C. lusitanica* were statistically superior to pellets of *T. grandis*. This behavior can be observed in the load vs. deflection curves (Fig. 3), where the pellets of *C. lusitanica* require a higher loading to achieve the same deformation as compared with pellets of *T. grandis*. In the parameters of durability, however, *C. lusitanica* presents a lower value than the presented in *T. grandis* (Table 1).

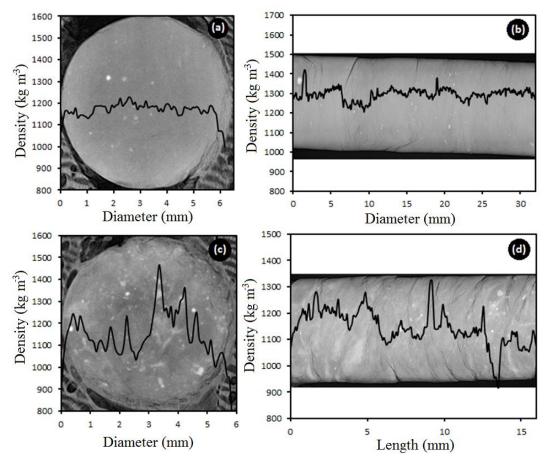


Fig. 4. Density variation (a) transversal and (b) longitudinal of pellets of *C. lusitánica*, and (c) transversal and (d) longitudinal of pellets of *T. grandis*.

Discussion

Efficiency of the pelleting process

Low percentages of efficiency found (25% in *C. lusitanica* and 20% in *T. grandis*) may be related to many factors. One of them and perhaps the most important is the use of appropriate matrices. Theerarattananoon *et al.* (2011) indicate that the size of the rolls and the diameter of the holes of the grid have an effect on the final result of the pelletizing process. These influence the pressure and the force with which the particle material enters and is compressed into the matrix and therefore, in the amount of ground material in which the pellet becomes. Other factors that can also influence the efficiency in the process of pelletizing are the size and MC of the particles, which influence the way in which particles that make up the pellets bind (Filbakk *et al.*, 2011, Nielsen *et al.* 2009a, Larsson *et al.* 2008). Low MC can cause very high temperatures in the

matrix, causing a rapid thermal decomposition on the surface of the pellets, resulting in problems of pelletized (Larsson *et al.* 2008). Conversely, if MC is too high, the pellets tend to crumble, this as a result of excessive internal pressure of the steam generated inside the pellet (Rehn *et al.* 2005; Nielsen *et al.* 2009a). This way, it is necessary that the particle possesses an optimal MC, which is considered between 9% and 12% (Bergstrom *et al.* 2008). In the case of *C. lusitanica* the MC of particle was approximately 8%, which was below the range considered optimal, and could be the cause of the low efficiency. Similarly, MC of *T. grandis* particle was 14%, value that exceeds the optimal range, which caused that many of the pellets crumble and this will affect the efficiency of the process.

Consistent with the previous results and to achieve greater efficiency in the process of pelleting is necessary to control the MC of particle before pelletizing, according to several authors this optimal value may vary according to the type of species used and the settings used in production process (Bergstrom *et al.* 2008, Lee *et al.* 2013). For the species used in this study, a MC of between 10% and 12% would be considered as optimum using the matrix of 6 mm, this MC would improve the quality of the pellets and hence its efficiency.

Evaluation of the quality of the pellets

An important parameter in the quality of the pellets, is the presence of cracks in the surface thereof, since these represent the susceptibility of the pellets to rupture (Stelte *et al.* 2011b). In this study, it was observed that both the pellets of *C. lusitanica* as the *T. grandis* showed cracks in its surface, but the cracks in the pellets of *T. grandis* were larger and deeper (Fig. 4), so it is possible that the pellets of *T. grandis* are susceptible to rupture. Nevertheless, this parameter is not correlated with the mechanical durability of pellets, this because the pellets of *T. grandis* showed a greater mechanical durability than the pellets of *C. lusitanica*. However, it appears that the pellet quality has an effect on the compression parameters, since the pellets of *C. lusitanica* had a higher compression strength than the pellets of *T. grandis* (Table 1).

The surface quality of the pellets is related to the MC of particle before pelletizing (Fasina 2008; Theerarattananoon *et al.* 2011). Fasina (2008) mentions that the humidity of the particles at the time of pelletizing strengthens the bonds between particles, but when the humidity is not appropriately decreases the capillary force between particles resulting in little bonding to the

structure of the pellets, causing cracks. As it was mentioned above, either of the species evaluated have a MC of particle within the prescribed range (9% to 12%), hence both presented cracks. Another important aspect to be pointed in the pellet quality is the presence of areas of greater clarity (Fig. 2), which are associated with high density, as is the case of pellets of *T. grandis* (Fig. 4c and 4d). These regions are the product of the structural variation and size of particles, and the characteristics of pressure and force proper of the pelletizing process (Mani *et al.* 2006b).

Evaluation of energetic, physical and mechanical properties of the pellets

One of the most important parameters for characterizing a substance as fuel is the NCP since it relates to the number of units of energy produced by the combustion of a unit mass of fuel (Elmo and Lousada 2011). Telmo and Lousada (2011) mentioned that NCP for softwood pellets as *Cedrus atlantica*, in the range of 19660 kJ kg⁻¹ to 20360 kJ kg⁻¹, and hardwood pellets as *Fagus sylvatica*, of 17632 kJ kg⁻¹ to 20809 kJ kg⁻¹. For this study both pellets of *C. lusitanica* as *T. grandis* showed values of NCP lower than those reported by these authors (Table 1). According to the norm ASTM D-5865 (ASTM 2003a) the NCP of the pellets should be about 11591 kJ kg⁻¹, value lower than that obtained for pellets of *C. lusitanica* and *T. grandis* in this study. However, the requirements of the European standards in terms of NCP are higher, these set values of NCP in the range of 17.500 to 19.500 kJ kg⁻¹ (Deutsches Institut Fur Normung, 1996), this way, the pellets manufactured in this study did not meet the requirements of the standards of the European norms.

NCP differences between *C. lusitanica* and *T. grandis* could be explained by the species-specific characteristics, about this, some studies indicate that species with high content of resins and extractives tend to have high values of NCP (Demirbas 2009, White 1987), *C. lusitanica* as a conifer is known for its high content of extractives, which translates into a caloric potential greater than *T. grandis*.

The ash content in the wood is also an important property that affects the capacity of combustion of a substance. A high quantity of ash in the wood makes it less desirable as a fuel (Kumar *et al.* 2009), also when the percentage of ash is over 4% may lead to corrosion of boilers and burners and cause equipment wear by abrasion (Mande 2009). The ash content obtained for both species (Table 1) determined by the ASTM D1102-84 norm (ASTM 2003b) do not meet the 0.5% of ash content allowed by the norm DIN 51731 (Deutsches Institut Fur Normung, 1996). In this case

the pellets of *C. lusitanica* showed a lower percentage of ash to the pellets of *T. grandis* (Table 1), which results in a higher NCP.

There are many opinions about the MC that the pellets should have to present a good performance, normally pellets contain a MC between 8% and 12% (Lehtikangas 2001, Kaliyan and Morey 2009). The MC of the pellets manufactured in this study is close to the designated range, the pellets of *C. lusitanica* obtained a MC of 7.50% and the pellets of *T. grandis* of 12.06%. The difference found between the MC of the pellets of the species is the result of the MC of the granular material before the pelletized and the pelletizing process. (Lehtikangas 2001, Rehn et al. 2005), in this case, both the pellets of C. lusitanica as the pellets of T. grandis were manufactured under the same conditions of pressure and temperature, while the particles of T. grandis showed greater MC than C. lusitanica, so that at the end of the process the high MC of particle of T grandis caused the pellets a higher MC than the MC of the pellets of C. lusitanica. Regarding the water absorption capacity, Fascina (2008), in a study of pellets, indicates that the optimum moisture absorption of the pellet is from 3% to 5%, and that an additional increase in moisture absorbed could result in a decline in quality and strength characteristics of the pellets. The absorption capacity obtained in this case for C. lusitanica was 9.31% and 3.76% for T. grandis (Table 1), which means that the pellets of T. grandis are within the indicated range, otherwise to the pellets of C. lusitanica, which have a greater capacity for absorption. Furthermore, differences in the water absorption capacity among the species can be explained by the MC that the pellets have, pellets of high MC tend to absorb a smaller amount of water, whereas low-MC pellets tend to absorb more water.

Apparent density of *C. lusitanica*, (550 kg m⁻³) can be considered normal according to the data reported by Theererattananoon *et al.* (2011) for pellets manufactured from corn stover, wheat straw and sorghum stalks, where the variation is of 479 kg m⁻³ to 649 kg m⁻³, but in *T. grandis* the value obtained (380 kg m⁻³) is lower than that mentioned by these authors. Regarding the differences between the apparent density of *C. lusitanica* and *T. grandis* can be explained by several aspects: (i) difference in specific gravity of the species and (ii) effect of MC. In the case of the specific gravity of the species, *C. lusitanica* has a specific gravity of 0.43 (Moya and Munoz 2010), which provokes having an apparent density higher than that obtained for the pellets of *T. grandis* which have a specific gravity of 0.60 (Moya et al. 2014). Regarding the MC, various authors mentioned an increase in the MC of the pellets results in a linear decrease of the apparent

density (Mani 2006b; Fascina 2008). In the case of this study *C. lusitanica* presented a MC of 7.50% while *T. grandis* has a moisture statistically higher (12.6%), so that, *C. lusitanica* by presenting a lower MC results with a higher apparent density, contrary to *T. grandis* that with a greater MC obtained a lower apparent density.

The density profiles for the two species indicate variations in both transversal and longitudinal direction (Fig. 3). In the case of *C. lusitanica* large density variations are not observed in any of the ways, which can be validated for its low coefficient of variation (Table 1). However, for *T. grandis*, it is possible to observe higher density zones produced by the presence of regions of greater clarity in the surface (Fig. 4c), which results in a greater coefficient of variation than *C. lusitanica*. As was discussed above, the presence of these light regions and its high density are produced by variations in the size and distribution of particles, some studies indicate that the smaller the particle size, the higher the density of the pellets, however, if the particle size is not uniform, it is possible that variations in density appear (Lehtikangas 2001; Rhen *et al.* 2005, Larsson *et al.* 2008, Bergstrom *et al.* 2008, Serrano *et al.* 2011).

The mechanical properties of the pellets, especially the compressive strength is influenced by the MC and by the parameters of pressure and temperature used during the pelletization (Rehn *et al.* 2005; Gilbert *et al.* 2009; Serrano *et al.* 2011). As mentioned above, the pelleting process was the same for both species, hence the only factor that could affect the compressive strength of the pellets is their MC. The differences in compression parameters found between *C. lusitanica* and *T. grandis* may be related to the MC of the pellets. According to Rhen *et al.* (2005), the compressive stress increases with decreasing of the MC of pellets, hence the pellets of *C. lusitanica* with lower MC presents a greater compressive stress and rupture force (Table 1, Fig. 3c).

The durability is defined as the ability of the pellets to withstand destructive loads and forces during transport (Tabil and Sokhansan 1996). This parameter is considered acceptable when its value is greater than 80%, medium when it is between 70% and 80%, and low when it is less than 70% (Colley *et al.* 2006). Applying these concepts to the durability of the pellets obtained in *C. lusitanica* and *T. grandis*, which was above 80% in both cases, we can say that the pellets made from these species are considered high durability. High durability values result in a decrease in the risks associated to fire explosions during transport or storage of the pellets, also

dust emissions, in the same way, prevents problems in feeding systems of pellets (Temmerman *et al.* 2006).

The differences found in the values of durability between the two species (Table 1) can be explained considering the MC again. It is reported that the durability increases as the MC increases, to reach an optimal point and that once it exceeds this point the durability and strength of the pellets decreases (Kaliyan and Morey 2009, Lee *et al.* 2013). About this, Colley *et al.* (2006) studying the effects that the MC has on the physical characteristics of the pellets of switchgrass found that pellets with a MC of 8.6% obtained a higher durability. Similar results were obtained in this study where the pellets of *T. grandis* with a MC of 12.06% obtained a percentage of mechanical durability greater than the pellets of *C. lusitancia* which MC was 7.50% (Table 1).

Conclusions

- The efficiency of the pelletizing process was 25% for *C. lusitanica* and 20% for *T. grandis*, both values are considered very low.
- In the evaluation of the quality of pellets, pellets of *C. lusitanica* are characterized by having zones of greater clarity and few cracks of short size on the surface, but the pellet of *T. grandis* has a larger number of areas of clarity and deeper and longer cracks.
- In the evaluation of physical properties, pellets of *C. lusitanica* had a diameter and an average length of 6.16 mm and 27.27 cm, respectively, and the pellets of *T. grandis* a diameter of 6.21 mm and a length of 19.78 cm. The variation of length in the pellets of *C. lusitanica* was 17.5 cm to 38.5 cm, while for *T. grandis* the variation was 15.5 cm to 30.5 cm. The MC of the pellets of *C. lusitanica* was statistically lower than the value obtained in *T. grandis*. The absorption capacity and apparent density in *C. lusitanica* is statistically greater than *T. grandis*.
- In the pellets of *C. lusitanica*, density is similar in longitudinal and transverse direction and is considered uniform density because of its low variation and in contrast, pellets of *T. grandis* have greater density variability in these two directions, making them less uniform.

• In the mechanical properties, the stress in compression and the rupture force for the pellets of *C. lusitanica* were statistically superior to pellets of *T. grandis*. In durability, however, *C. lusitanica* has a lower value than in *T. grandis*.

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2. Artículo 2. Application of the X ray densitometry in the evaluation of the quality and mechanical properties of pellets of twelve forestry and agricultural crops in Costa Rica.

Application of the X-ray densitometry in the evaluation of the quality and mechanical properties of pellets of twelve forestry and agricultural crops in Costa Rica

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Abstract

The use of the X-ray technique and the X-ray densitometry to determine pellet particle distribution and to understand the biomass compaction and its effects in pellet properties has been limited. The present work evaluates the quality of pellets manufactured with several lignocellulosic materials by using X-ray photography for studying surface cracks and irregularities, and by using X-ray densitometry to evaluate density and its variation in longitudinal and transversal directions. Density values and their variation were correlated to pellets' mechanical properties (mechanical durability and compression resistance). It was found that X-ray photography may be applied to evaluate the presence of cracks and irregularities in pellets' surface; however, these are not indicators of pellet durability or compression resistance. Moreover, density evaluation by the X-ray densitometry technique allowed determination of pellets' mechanical resistance and its durability. A negative correlation was observed between the force at break and the coefficient of variation of density. No correlation was found between the mechanical durability and the average density or its variation.

Key words: density, cracks, irregularities, clearer areas, pellet surface uniformity.

1. INTRODUCTION

Biomass is one of the most promising energy sources since it is an alternative to conventional energy sources such as petroleum, natural gas, etc. One of its main advantages is that it's a clean and renewable product, which contributes to the reduction of greenhouse gas emission effects and of dependency on fossil fuels (Luque *et al.* 2008).

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Today, wood residues are the main raw materials used in pellet production (Fasina 2008; Stelte *et al.* 2011; Stahl *et al.* 2011). Nevertheless, agricultural residues, energetic crops and other waste products from the food industry are being used, such as: corn, oil palm residues, and some potato and tapioca varieties, among others (Stelte *et al.* 2011, Stahl *et al.* 2011; Theerarattananoon *et al.* 2011; Razuan *et al.* 2011).

Pellets present some weaknesses, among which are their mechanical resistance and compaction, which result in manipulation and durability problems (Kaliyan and Morey 2009). Pellet quality shall depend, among other things, on the efficacy of particle bonds (Lee *et al.* 2013). In consequence, many studies have been carried out to examine the effects of some variables on pellet resistance and durability (Mani *et al.* 2006; Kaliyan and Morey 2010, Lee *et al.*, 2013). Some techniques have been implemented to evaluate pellet quality -compaction and durability-, of which the scanning electron microscopy (SEM) is one of the most widely used. For example, Kaliyan and Morey (2010) used this technique to determine particles' internal distribution for different materials within the pellet. Meanwhile, Stelte *et al.* (2011) found when using SEM, that the defects in pellets decrease particle cohesion in beech wood pellets. Furthermore, Reza *et al.* (2014) argue that some pellets show an apparent development of a solid bridge that maintains particles joined, which makes it impossible to observe cracks in the pellet's surfaces.

Although the SEM technique is adequate to evaluate pellet quality, it has the disadvantage that it cannot be easily applied in a process. However, other techniques such as the X-ray, allow evaluation of the material during the process instantly (Kotwaliwale *et al.* 2014). The X-ray technique is broadly used in diverse applications and material types (Stock 2008). Several X-ray application methods are used for agriculture (Kotwaliwale *et al.* 2014). One of them is the X-ray photography, which is widely used as a tool to determine the spatial distribution of different solid phases or structures, for deformations, fatigues and fractures that result from the processing, as well as corrosion and environmental interactions (Bhuiyan *et al.* 2013, Stock 2008).

This technique has been implemented for many years in solid wood and composite materials ^[15] (Polge 1973). In solid wood for example, it is applied to determine the variability in density and ring growth (Schinker *et al.* 2003). In composite materials, including wooden ones such as fiberboards or particleboards, it is applied to estimate its performance (Sanabria *et al.*, 2013). It is also applied in other materials similar to wood. For example, Zhaohui *et al.* (2004) use this same technique to determine the radial density variation profile of bamboo; while Belini *et al.* (2013) use densitometry to evaluate the quality of medium density fiberboards (MDF).

Notwithstanding, implementing X-ray photography or X-ray densitometry to determine the distribution of pellet particles, or else to understand biomass compaction and its effects on pellet properties or briquettes, has been limited. One of the few studies was led by Ferreira *et al.* (2012), who studied X-ray densitometry in briquettes made from wooden residues to determine whether their internal density had a homogeneous distribution, which would have an effect in briquette quality.

As a result, the present work evaluates pellet quality through visualizing the presence of surface cracks and surface pellet uniformity, and by using X-ray densitometry to determine density in twelve forestry and agricultural crops (*Cupressus lusitanica, Tectona grandis, Ananas cumosos, Elaeis guineensis, Arundo donax, Gynerium sagittatum, Pennisetum purpureum, Sorghum bicolor, Phyllostachys aurea, Coffea arabica and Saccharum officinarum*) in Costa Rica. Likewise, density and its variability are correlated to pellet mechanical properties (durability and compression resistance) of the above-mentioned crops.

2. MATERIALS AND METHODS

2.1.Materials and source

Twelve crops growing in Costa Rica were selected to manufacture pellets: 2 forest species and 10 agricultural crops. Table 1 presents information for the twelve crops used.

Table 1. Description and source of twelve crops used to manufacture pellets.

Crops	Scientific name	Precedence	Abbreviation
Sawlog residuals of <i>C. lusitanica</i>	Cupressus lusitanica	Agua Caliente, Cartago	CL
Sawlog residuals of <i>T. grandis</i>	Tectona grandis	Abangares, Guanacaste	TG
Pineapple leaves from the plant	Ananas cumosos	Buenos Aires, Puntarenas	PLP
Empty fruit bunch of the oil palm	Elaeis guineensis	Parrita, Puntarenas	EFB
Oil palm mesocarp fiber of the fruit	Elaeis guineensis	Parrita, Puntarenas	OPMF
Giant cane	Arundo donax	Filadelfia, Guanacaste	AD
Wild cane	Gynerium sagittatum	Río Frío, Limón	GS
King grass	Pennisetum purpureun	Paraíso, Cartago	PP
Sorghum	Sorghum bicolor	Upala, Alajuela	SB
Golden bamboo	Phyllostachys aurea	Cartago, Cartago	PA
Coffee pulp	Coffea arabica	Tarrazú, San José	CA
Sugarcane	Saccharum officinarum	San Carlos, Alajuela	SO

2.2.Pellet manufacturing process

The pellet production process was conducted in PELLETICS (http://pelletics.com/), located in San Carlos, Alajuela province (Costa Rica). Figure 1 presents the production process from the point where crops enter the processing plant and throughout the pellet production process. A detailed description of the process may be consulted in Aragón *et al.* (2014).

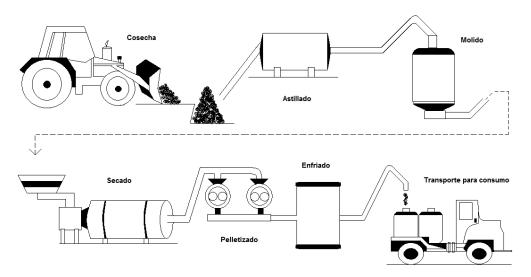


Figure 1. Pellet manufacturing process in PELLETICS S.A. (Aragón et al. 2014).

2.3. Pellet preparation for densitometry measurements

The X-ray densitometry density measurement was performed in longitudinal and transversal directions (Figure 2) on 10 randomly selected pellets for each crop. These pellets were adjusted to 12% moisture content (temperature at 22 °C and 66% relative humidity). Afterwards, weight, diameter and length of pellets were determined to calculate their actual density. For the densitometry measurement in longitudinal direction, pellet preparation was not necessary since these were placed directly in the X-ray equipment's bracket (Figure 2a). In order to determine density in transversal direction, the 10 pellets were placed longitudinally on a base with two wooden supports (Figure 2b), and then carefully cut into transversal sections of approximately 1.8 mm thick (Figure 2c).

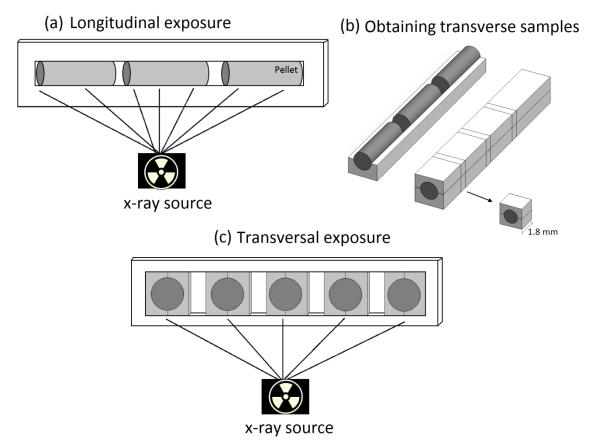


Figure 2. Densitometry in longitudinal direction (a), sample preparation for densitometry in transversal direction (b), densitometry in transversal direction (c).

2.4.Densitometrical measurements

For the longitudinal direction measurement, pellets were placed horizontally on the bracket included with the X-ray equipment, where the X-ray source runs longitudinally through the sample. Thus, the X-ray photograph is obtained and the density profile determined. For densitometry in transversal direction, the pellet samples 1.8 mm thick were once again placed on the equipment's bracket to obtain the images and density readings. The exposure of the samples in longitudinal and transversal directions were performed using an X-ray scanner, from Quintek Measurement Systems Inc., QTRS-01X model. The exposure conditions were done performed at 7 KV tension in the tube, and the density readings were carried out for 1 second every 40 μ m.

2.5. Density variation and calculation by X-ray densitometry

Firstly, the pellet's actual average density was previously determined by measuring its weight, length and diameter (Equation 1). Later, X-ray densitometry was used to determine the average pellet density (Equation 2). The density values calculated with the X-ray equipment were corrected with the correction factor (Equation 3), which is calculated by the difference between the average density of all measurements obtained by densitometry (Equation 2) and the actual average density of the pellet.

$$Actual\ density = \frac{Pellet\ weight\ (kg)}{Pellet\ radius\ (m^2)*Pellet\ length\ (m)*\pi} \qquad (1)$$

$$Average\ density\ by\ densitometry = \frac{\sum_{i=1}^n x_i}{n} \qquad (2)$$

$$Correction\ factor = Average\ density\ by\ densitometry - Actual\ density \qquad (3)$$

Where: n: number of densitometrical pellet measurements and i represents i^{th} measurements.

Once the correction factor was established, it was applied to each densitometry value evaluation (Equation 4). The correction was applied to the pellets' values of longitudinal and transversal directions. Once the corrected density was calculated, average density (Equation 2) and its standard deviation (Equation 5) were calculated, in order to calculate the coefficient of variation (Equation 6) for each of the pellets for all crops evaluated. Walker and Dodd's (1988) [22] proposed methodology, which calculates the density variation in wood by using the X-ray densitometry readings, was used to calculate the coefficient of variation by means of densitometry. The coefficient of variation was determined for variation in transversal direction (CVtrans) and for variation in longitudinal direction (CVlong).

$$Corrected\ density_{i} = Density_{i} - Correction\ factor \qquad (4)$$

$$Standard\ deviation = \sqrt{\frac{\sum (corrected\ density_{i} - average\ corrected\ density_{i})^{2}}{n-1}}$$

$$Variation\ coefficient\ (\%) = \frac{Standard\ deviation}{Average\ corrected\ density_{i}}*100 \qquad (6)$$

Where: $corrected\ density_i$ represents the corrected density value in each ith measurement and $density_i$ represents the densitometry value for each ith measurement.

2.6.Pellet quality evaluation

Two methods were used to evaluate pellet quality: the first method observed the presence of pellet surface cracks through X-ray photography; the second method evaluated pellet quality through the variation in density in longitudinal and transversal direction densitometry. 10 pellets for each crop were randomly selected for both quality evaluation techniques. X-ray photographs were taken with Hewlett Packard equipment, model Faxitron LX-60 with a 12 cm distance between the X-ray source and the samples. The exposure conditions were of 15 seconds at a tension of 30KV within the tube. To evaluate quality regarding the variation of density using densitometry, x-y graphics were created in order to show the density profiles both longitudinally and transversally.

2.7. Determination of the mechanical properties

The mechanical durability and compression resistance of pellets were determined. To calculate the mechanical durability, the DD CENT/TS 15210-1:2005 (BSI 2005) standard was applied. For this essay, 10 representative samples of 500 grams of pellets were taken, and were passed through a sieve with a 3.36 mm mesh to eliminate fine particles. The sieved samples were placed in the equipment proposed by the standard at a speed of 50 rpm for 10 min. Afterwards the sample was removed, then sieved and weighed. Durability was calculated using Equation 7.

For the compression resistance test 10 pellets of approximately 13 mm long were selected randomly. This test was performed longitudinally according to the methodology proposed by Aarseth and Prestlokken (2003), by using a universal testing machine Tinus Olsen H10KT Model of 1 ton. In this test, a compression force velocity of 0.02 mm/s is applied. It provides force at break vs. pellet deformation measurements, with which the maximum stress was calculated (Equation 8). The pellet's force at break and maximum compression stress were reported.

Mechanical durability (%) =
$$\frac{pellet\ weight\ before\ test\ (g)}{pellet\ weight\ after\ test\ (g)} \times 100$$
 (7)

$$Compression stress = \frac{2 \times maximum \ load \ (Kg)}{\pi \times Diameter \ (cm) \times Length \ (cm)}$$
(8)

2.8. Statistical analysis

A descriptive analysis was carried out (median, standard deviation, maximum and minimum values) of the average longitudinal and transversal density values for each test: densitometry, mechanical durability, force at break and compression stress. In addition, the variables were verified against the normal distribution premises, variance homogeneity, and presence of extreme data. A variance analysis was applied to verify the existence of significant differences between variable averages (P<0.05), whereas Tukey's test was done to determine the statistical differences among crops for the medians of each one of the previously mentioned values.

Pearson's correlation matrix was used to determine the correlation between density values (average, CVtrans and CVlong) and the mechanical properties studied (durability and force at break). Later, a forward stepwise multiple regression analysis was applied to determine the influence of average densities, CVtrans and CVlong, on the mechanical durability and the force at break. Then, a scatter plot graph was generated for the correlation between the force at break and density variables that were affected significantly, according to the forward stepwise regression analysis. Lastly, the variance analysis, Tukey's test and correlation analysis were performed using SAS software (SAS Institute Inc., Cary, NC).

3. RESULTS

3.1.Pellet quality

The first quality evaluation technique of X-ray photography revealed the presence of cracks, as well as clearer areas and irregularities on the surface of pellets (Figure 3).

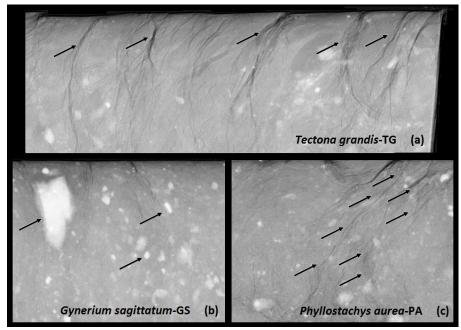


Figure 3. Cracks observed in a *Tectona grandis* pellet (a), clearer areas in a *Gynerium sagittatum* pellet (b), and irregularities in the pellet's surface of *Phyllostachys aurea* (c), observed with X-ray photographs.

The evaluation of these 3 aspects in each one of the species studied revealed that those pellets manufactured with OPMF and CA did not have surface cracks (Figures 4e, 4k). PLP, EFB, AD, PP and SB had few and not very deep cracks (Figures 4c-d, 4f, 4h-i). CL, GS and SO pellets were observed to have a higher amount of deep cracks (Figures 4a, 4g, 4l). TG and PA presented the highest amount of cracks, having a similar depth to CL, GS and SO pellets (Figures 4b, 4j). With regard to clearer areas on the pellet's surface, these are more abundant in PLP, EFB, GS, PA and SO (Figures 4c-d, 4g, 4j, 4l), followed by TG, AD, PP and CA (Figures 4b, 4f, 4h, 4k), and lastly, CL, OPMF and SB hardly present any (Figures 4a, 4e, 4i). The pellet surface irregularities evaluated were observed in: CL, TG, OPMF, AD, SB, PA and SO (Figures 4a-c. 4e, 4i, 4j and 4l) and were not observed in: PLP, EFB, GS, PP and CA (Figures 4c-d, 4g-h, 4k).

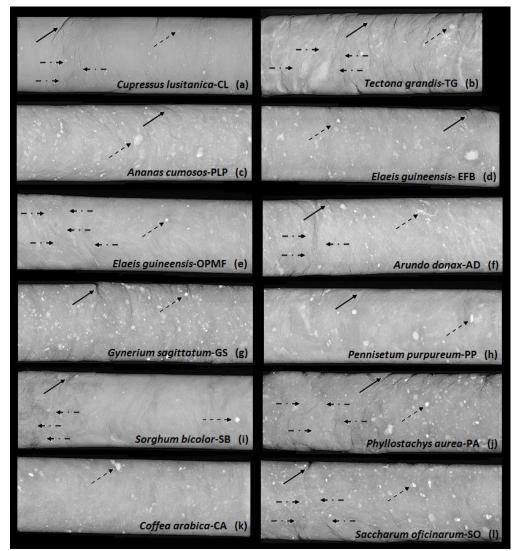


Figure 4. X-ray photograph of the surface of the pellets manufactured with twelve agricultural crops.

Note: The marker → indicates the presence of cracks in the pellet surface, the marker ---- a clear area and the marker - · - b the presence of irregularities in the pellet surface.

3.2.Density evaluation

The X-ray densitometry used to evaluate pellet density found that it varies from 1096 kg/m³ to 1294 kg/m³ (Table 2) for both longitudinal and transversal directions. However, the variation of density, reported by the coefficient of variation in longitudinal direction (CVlong) and transversal direction (CVtrans), results in differences in the average density of the same crop for both directions. Longitudinally, there are 3 groups present: (i) the group with the highest density values (1221 kg/m³ to 1294 kg/m³), composed of: PP, AD, CA, EFB, GS and PLP; (ii) a second group with intermediate density (1136 kg/m³ to 1208 kg/m³) and composed of: CL, OPMF, PA and TG; and (iii) SB and SO with the lowest densities (Table 2). Moreover, it is also possible to make 3 groupings transversally, but with a slight variation in the species that compose them: (i) the first group, which presents the highest density values (1294 kg/m³ to 1225 kg/m³), composed

of: PP, AD, CA, EFB and GS; (ii) a second group with intermediate density (1192 kg/m³ to 1221 kg/m³) composed of PLP, CL and OPMF; and lastly (iii) PA, TG, SB and SO with values between 1096 kg/m³ to 1170 kg/m³ presenting the lowest density (Table 2).

Table 2. Density and its variation obtained by X-ray densitometry in transversal and longitudinal directions for twelve pellet crops in Costa Rica.

	Length direction		Transversal direction	
Crops	Average	Coefficient of	ž	Coefficient of
	(kg/m^3)	variation (%)	(kg/m^3)	variation (%)
Cupressus lusitanica-CL	1208 ^{AF}	$4,33^{BC}$	1208^{ABCDE}	5.16 ^D
Tectona grandis-TG	1136 ^{EFI}	6,99 ^{AC}	1136 ^{DEF}	7.71 ^{CD}
Ananas cumosos-PLP	1221^{AE}	6,95 ^{AC}	1221^{ABCD}	16.35 ^A
Elaeis guineensis-EFB	1233 ^{AC}	4,98 ^{BC}	1233 ^{ABC}	11.89 ^{ABC}
Elaeis guineensis-OPMF	1192^{BCEFG}	6,45 ^{AC}	1192 ^{BCDE}	12.70^{ABC}
Arundo donax-AD	1292 ^A	8,51 ^A	1292 ^A	14.08^{AB}
Gynerium sagittatum-GS	1225^{ADG}	6,33 ^{AC}	1225 ^{ABC}	8.47 ^{BCD}
Pennisetum purpureun-PP	1294 ^A	6,24 ^{AC}	1294 ^A	12.52 ^{ABC}
Sorghum bicolor-SB	1129 ^{FJ}	5,35 ^{AC}	1129 ^{EF}	9.38 ^{BCD}
Phyllostachys aurea-PA	1170^{CDEFH}	6,38 ^{AC}	1170^{CDEF}	8.69 ^{BCD}
Coffea Arabica-CA	1279 ^{AB}	$4,50^{BC}$	1279 ^{AB}	7.06^{CD}
Saccharum officinarum-SO	1096 ^{HIJ}	5,01 ^{BC}	1096 ^F	10.25 ^{BCD}

Note: different letters for each parameter mean statistical significances at 95%.

3.3.Density in the transversal and longitudinal direction of the pellet

Density is shown by its longitudinal and transversal profiles (Figures 5 and 6). In both directions of the pellet two density variation patterns were observed: one uniform and another irregular. Density of uniform variability in longitudinal direction was observed in: CL, EFB, CA and SO (Figures 5a, 5d, 5k-l), whereas the other species (TG, PLP, OPMF, AD, GS, PP, SB and PA) presented an irregular variation pattern (Figures 5b-c, 5e-j). Transversally, the crops with a uniform pattern were: CL, TG, GS SB, PA and CA (Figures 6a-b, 6g, 6i-k), while PLP, EFM, OPMF, AD, PP and SO presented an irregular pattern (Figures 6c-f, 6h, 6l).

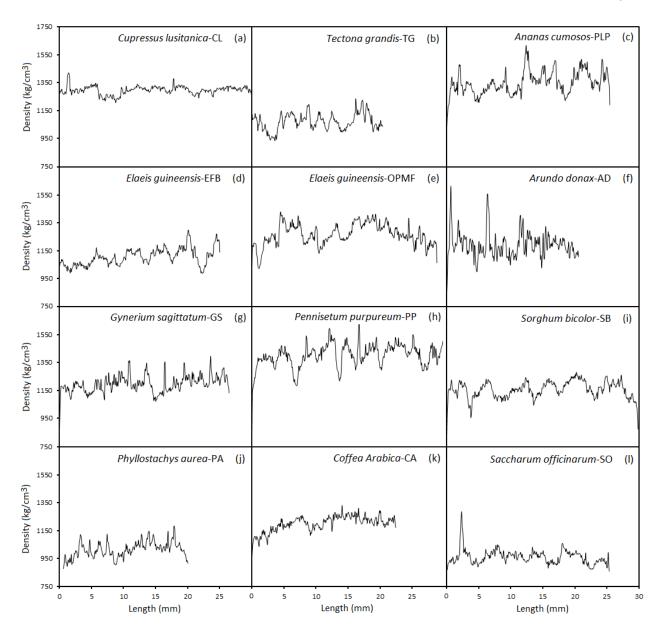


Figure 5. Longitudinal density variation in twelve pellet crops in Costa Rica.

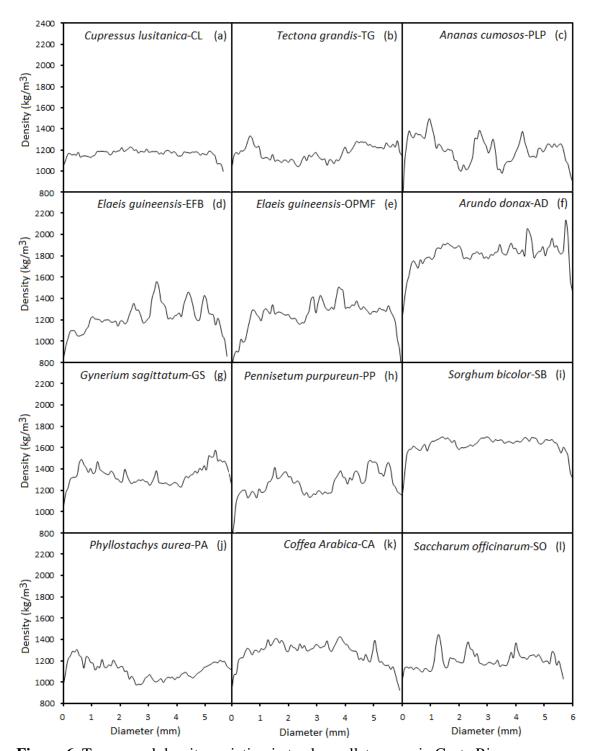


Figure 6. Transversal density variation in twelve pellets crops in Costa Rica.

3.4.Density variability of the pellet by coefficient of variation

CVlong values (from 4.33% to 8.51%) are lower compared to CVtrans (from 5.16% to 16.35%) (Table 2). The lowest CV values found both in longitudinal and transversal directions are CL and

CA, while PLP and AD present the highest. No differences were observed for CVlong among the crops, with the exception of AD, which is statistically different from CL, EFB, CA and SO. Likewise, for CVtrans, most crops do not present statistical differences among each other, with the exception of PLP, which presents differences with CL, TG, GS, PP, SB, PA, CA and SO; as well as CL, which is different from PLP, EFB, OPMF and AD (Table 2).

3.5. Mechanical properties

The force at break varied from 297 N to 875 N and the compression stress from 1.70 MPa to 4.80 MPa. The values obtained for compression stress and force at break allow to make three crop groupings: (i) crops with the highest force at break and compression stress: PP, CL and GS; followed by (ii) the crops with intermediate values: EFB, OPMF, AD and SP; and finally (iii): PA, SB, TG, PLP and CA as the lowest values (Table 3).

Figure 7 illustrates this grouping with the curve force vs. deformation. The first species group (PP, CL and GS) tend to reach high force levels (superior to 650 N) with low deformation values, and are characterized by presenting very high-sloped curves in the elastic section of the pellet. The second species group (EFB, OPMF and AD) performed differently due to the fact that the supported force is lower (in the range of 500 N) for the same deformation levels as compared with that of the first group. The third species group (PA, SB, TG, PLP and CA) shows lower forces than those of the other two species groups for the same deformation values. Furthermore, their curve slopes are lower in relation to the other species. In turn, SO is a different case, because pellets made of this species can support forces over 500N, reason for which it is included in the second species group. Nevertheless, at deformation levels of 0.2 mm or 0.4 mm it presents forces in the range of the third group species. Moreover, the curve slope for SO is similar to the one of the second group species (Figure 7).

For the mechanical durability test, values varied from 72.12% to 92.98%, and the crops with the highest durability values are: AD, EFB, OPMF, PP and TG, without statistical differences among them, while the rest of the crops (CL, PLP, GS, SB, PA, CA and SO) present lower durability values than the former group and are statistically different among them (Table 3).

Table 3. Compression effort and mechanical durability of twelve pellet crops in Costa Rica.

Crops	Force of break (N)	Compression strength (MPa)	Mechanical durability (%)
Cupressus lusitanica-CL	738.34 (25.12) ^{AB}	4.80 (19.01) ^A	84.74 (1.98) ^G
Tectona grandis-TG	393.93 (16.49) ^{DEFG}	2.14(17.17) ^{EFG}	92.08 (1.42) ^{AD}
Ananas cumosos-PLP	359.12 (50.49) ^{DEFG}	1.89 (40.89) ^{FG}	90.88 (0.63) ^{BCDE}
Elaeis guineensis-EFB	563.20 (28.37) ^{BCD}	3.13 (29.78) ^{CDE}	92.82 (0.72) ^A
Elaeis guineensis-OPMF	520.05 (34.33) ^{BCF}	2.90 (26.64) ^{DFH}	92.76 (1.38) ^{AB}
Arundo donax-AD	558.88 (9.84) ^{BCE}	$3.57 (12.64)^{BD}$	92.98 (0.48) ^A
Gynerium sagittatum-GS	675.58 (18.79) ^{AC}	4.67 (19.95) ^{AB}	88.42 (1.07) ^F
Pennisetum purpureun-PP	874.56 (19.18) ^A	4.17 (28.23) ^{ABC}	92.34 (1.06) ^{AC}
Sorghum bicolor-SB	417.67 (22.53) ^{DEFG}	2.41 (25.82) ^{EFGH}	84.50 (0.86) ^G
Phyllostachys aurea-PA	444.63 (28.63) ^{DEFG}	2.64 (30.38) ^{DG}	$72.12 (0.53)^{I}$
Coffea Arabica-CA	296.85 (24.08) ^G	1.70 (24.98) ^G	75.54 (2.14) ^H
Saccharum officinarum-SO	502.79 (49.51) ^{CG}	2.05 (31.63) ^{EFG}	90.40 (0.48) ^E

Note: different letters for each parameter mean statistical significances at 95%.

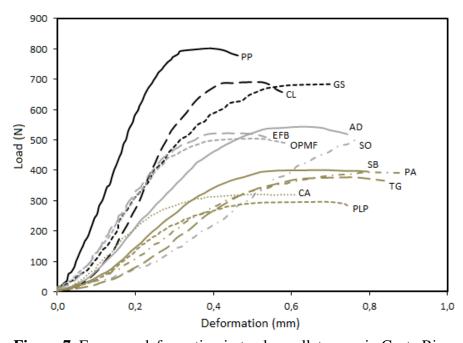


Figure 7. Force vs. deformation in twelve pellet crops in Costa Rica.

3.6.Relationship between the densitometric values and mechanical durability and compression

Correlation analysis results show that density and mechanical durability correlation values are not significant for any of the twelve crops evaluated. Pearson's correlation coefficients showed that the force at break had a stronger correlation with CVlong and CVtrans with respect to the longitudinal and transversal direction density averages (Table 4). Also, all are negative correlations. CL, TG, PP, PA and CA present a correlation between the force at break and both coefficients of variation, while OPMF and GS only have force at break and CVlong correlations; SB and SO crops were correlated to CVtrans. PLP, AD and PA were the only crops where the force at break was correlated with the average of both densities. EFB did not present correlation between the density values and the force at break.

Table 4. Correlation coefficient (r) values between density values and force at break of twelve pellet crops in Costa Rica.

	Density values			
Crops	Length	Coefficient	Transversal	Coefficient
	direction	of variation	direction	of variation
Cupressus lusitanica-CL		-0.7815*		-0.7968*
Tectona grandis-TG		-0.7420*		-0.8292**
Ananas cumosos-PLP	-0.8949*		-0.8949*	
Elaeis guineensis-EFB				
Elaeis guineensis-OPMF		-0.6828*		
Arundo donax-AD	-0.7044*		-0.7044*	
Gynerium sagittatum-GS		-0.7081*		
Pennisetum purpureun-PP		-0.9361**		-0.8230**
Sorghum bicolor-SB				-0.7035*
Phyllostachys aurea-PA	-0.8300**	-0.8583**	-0.8300**	-0.7251*
Coffea Arabica-CA		-0.7474*		-0.7371*
Saccharum officinarum-SO				-0.8623**

Note: *statistically significant at 95% and **statistically significant at 99%.

From the multiple stepwise correlation analysis it is inferred that CVlong can explain 63% of the force at break variation in CL, complemented by the longitudinal density average with 12% of the total variation. Instead, force at break variations in OPMF and AD can only be explained by CVtrans (47%) and by the transversal direction density average (50%) respectively (Table 5). Meanwhile, for SB and SO, the force at break was correlated to CVlong in 49% and 74%

respectively. CVlong explains 9% and 2% of the force at break for GS and PP, while CVtrans explains 50% and 88% respectively. CVlong explains 74% of CA's force at break variation. For the remaining crops, no significant correlation was found between the force at break and any of the density values, either in longitudinal or transversal directions (Table 5).

Table 5. Multiple stepwise correlation analysis for the correlation between density values and force at break of twelve pellet crops in Costa Rica.

	Correlation	Density values				
Crops	coefficient	Length Direction	Coefficient of variation	Transversal direction	Coefficient of variation	
Cupressus lusitanica-CL	R=0,87	0.12 (0.75)	0.63* (0.63*)	-	-	
Tectona grandis-TG	R=0,90	-	-	-	-	
Ananas cumosos-PLP	R=0,68	-	-	-	-	
Elaeis guineensis-EFB	R=0,69	-	-	-	-	
Elaeis guineensis- OPMF	R=0,68	-	-	-	0.47* (0.47*)	
Arundo donax- AD	R=0,70	-	-	0.50* (0.50*)	-	
Gynerium sagittatum-GS	R=0,77	-	0.09 (0.59)	-	0.50* (0.50*)	
Pennisetum purpureun-PP	R=0,94	-	0.02 (0.90)	-	0.88* (0.88*)	
Sorghum bicolor-SB	R=0,70	-	0.49* (0.49*)	-	-	
Phyllostachys aurea-PA	R=0,90	-	-	-	-	
Coffea Arabica- CA	R=0,81	0.09 (0.65)	-	-	0.56* (0.56*)	
Saccharum officinarum-SO	R=0,86	-	0.72* (0.72*)	-	-	

Note: * Statistically significant at 95%. Values: contribution of the parameter to the coefficient of determination; values between parenthesis: multiple coefficient of determination.

Performance of the force at break density values which showed significant correlations may be noted on Figure 6. The force at break increases when CVlong decreases for CL, SB and SO (Figure 6a-c). OPMF, GS, PP and CA present the same performance for CVtrans (Figure 6e-h). For AD, variations in transversal density cause an increase in the force at break (Figure 6d).

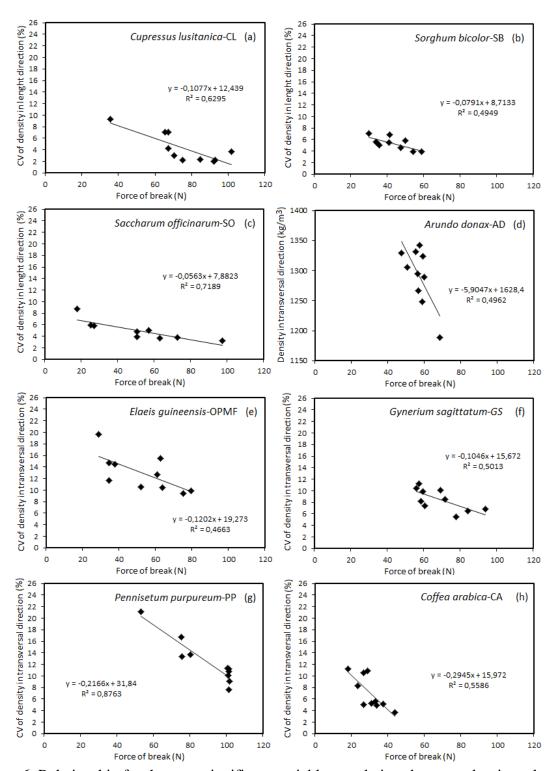


Figure 6. Relationship for the most significant variable correlations between density values and force at break.

4. DISCUSSION

4.1.Pellet quality

The surface cracks observed represent openings in between the compacted layers of the pellets. These cracks are produced by abrasion, which tends to remove the dust and fine material on the pellet surface, which are a consequence of the pellet manufacturing process (pelleting and cooling), of storage or compaction, or of movement during transportation, among others (Oveisi et al. 2013). However, two factors tend to be related with the pellet's superficial quality (cracks and irregularities): (i) moisture content of the material prior to pelleting (Mani et al. 2003, Fasina 2008, Theerarattananoon et al. 2011) and (ii) size and distribution of pellet particles (Mani et al 2003, Fasina 2008). Fasina (2008) mentions that material humidity during pelleting strengthens particle bonds, although when moisture content is not appropriate, the capillary attraction of particles decreases, resulting in a poor union in the pellet structure, which leads to surface cracks. With respect to particle size, Payne (1978) indicates that thicker particles tend to produce less durable pellets with surface cracks and irregularities; therefore, finely grounded and medium-sized particles are essential to ensure pellet quality. Likewise, Mani et al. (2003) note that wooden pellets made of fine particles achieved better quality results, when compared to pellets made with thick particles.

On the other hand, the presence of clearer areas can be associated to pellet density variations (Figure 3b). Such regions are a product of variations in the anatomical structure of each material and the size of granulated particles. For example, PA has high fiber concentration on the external layers of the stem (Pereira *et al.* 2007), which causes an increase in density from the inner part of the stem to the outer part (Zhaohui *et al.* 2004). Another example is given by AD, the anatomical structure of which varies in nodes and internodes, where the fiber wall thickness of the nodes is greater, and therefore denser (Shatalov *et al.* 2006). This variation in crops' anatomical structure and the presence of denser parts in the plants, results in some particles being placed randomly durig compaction in certain areas of the pellet; this in turn results in an increase in density in the region and can be observed as clearer areas, as was the case for the majority of crops herein studied; specifically: PLP, EFB, GS, PA, SO, TG, AD, PP and CA (Figure 4b-d, 4f-h, 4j-l).

A relevant parameter in pellet quality is the presence of surface cracks, since these characterize the susceptibility of pellets to rupture (Stelte et al. 2011). X-ray photographs of the pellets showed that cracks and irregularities present themselves in different degrees for the majority of crops, with the exception of OPMF and CA where no surface cracks were observed (Figure 4e, 4k). Therefore, as expected by Stelte et al. (2011), CL, TG, PLP, EFB, AD, GS, PP, SB, PA and SO are crops with cracks, making them susceptible to rupture (Figure 4a-d, 4f-h, 4j-l). Nevertheless, the results obtained for mechanical durability, which measures the ability of the pellet to remain intact (Temmerman et al., 2006) or resistance to compression (Table 3), did not point toward any relation with the presence of cracks. For example, pellets manufactured with CA do not present cracks (Figure 4k), but their durability and force at break during compression were one of the lowest (75.54% and 296 kg, respectively). TG and PA pellets which had the most cracks (Figures 4b, 4j), only have a low force at break and durability in PA, since TG is one of the most durable crops (92.08%). According to Temmerman et al. (2006) the durability test is a complex test where the rotation factor amply intervenes, and the factor (50 rpm) recommended by the standard does not reflect the actual pellet durability, therefore they suggest a factor of 105 rpm.

4.2.Density evaluation

The average density obtained from pellets made of the twelve different crops varied from 1096 kg/m³ to 1294 kg/m³ (Table 2). These density values are in accordance with the range established by the German DIN 51731 standard, from 1000 kg/m³ to 1400 kg/m³ (Deutsches Institut für Normung 1996). Also, Lehtikangas (2001) indicates an average density of 1234 kg/m³ for pellets manufactured with sawdust obtained from a combination of *Picea abies* and *Pinus silvestris*, which is a value found among the higher average density values obtained for PP, AD, CA, EFB, GS and PLP in this study (1221 kg/m³ to 1294 kg/m³).

Density evaluation by X-ray densitometry, measured by means of the CVlong and the CVtrans, demonstrated that both transversal and longitudinal direction variations exist for all crops (Table 2 and Figures 5-6). Crops like CL, EFB, CA and SO with the lowest CVlong, are correlated with uniform density profiles lengthwise on the pellet (Figure 5a, 5d, 5k-l). Likewise, the low variation in density profile of CL, TG, GS, SB, PA and CA in transversal direction results in lower CVtrans, producing a uniform density profile (Figure 6a-b, 6g, 6i-k).

The particle size of pellets of the crops studied is responsible for the variations observed in the density profile for both longitudinal and transversal directions (Mani *et al.* 2003). In this regard, some studies suggest that the lower the particle size, the higher the pellet density; however, if the particle size is not uniform, density variations may occur (Lehtikangas 2001; Larsson *et al.* 2008; Bergstrom *et al.* 2008; Serrano *et al.* 2011).

The internal density variations are attributed to many factors. Nevertheless, one factor that may be influencing the density variation in this study is that the pellets present clearer areas or spots in their surface (Figure 4), which tend to produce areas of greater density in some parts of the pellet, thus affecting its density pattern and resulting, in some cases, in higher CVlong and CVtrans. As mentioned earlier, some crops have cells with thicker walls or regions in which their walls become thicker (Pereira *et al.* 2007, Shalatov *et al.* 2006), this variation in its anatomical structure affects their density patterns.

In the same way, the internal structure of each crop that was analyzed, as well as the temperature and pressure applied during pelleting, may affect the density performance (Mani *et al.* 2003, Rhén *et al.* 2005). Gilbert *et al.* (2009) indicate that density increases substantially at temperatures between 14°C and 50°C, while at temperatures above 75°C and 95°C pellets have a tendency to stabilize their density. Several authors point out a relationship between pressure and pellet density, where density increases exponentially, with an increase in pressure during pellet manufacture, until reaching the maximum density value (Husain *et al.* 2002, Rhén *et al.* 2005).

4.3. Mechanical properties

Force at break values varied highly (Table 3). The compression resistance trials simulate the compression stress to which pellets are subjected during storage, or when performing. The higher the force at break and compression stress that pellets withstand, the better their final performance

(Kaliyan and Morey 2009). In this respect, García-Maraver *et al.* (2010) point to a force at break of 209.18 N for pellets manufactured with olive tree branches, value lower than the one obtained for the pellets in this study, which varied from 297 N to 875 N (Table 3). However, the compression stress values for different types of biomass have a wide range of values (Stelte *et al.* 2012) such as the ones presented in this study (Table 3).

With respect to the differences found among pellets from the different crops (Table 3), several studies suggest that pellet production stages, such as drying at high temperatures, milling and pressing, together with the moisture content, affect the final resistance properties of pellets (Rhén et al. 2005; Gilbert et al. 2009; Serrano et al. 2011), as evidenced in this study. The pelletizing process in this study was the same for all crops, although due to characteristics of each crop, different temperature and pressure variations could occur during pellet manufacture, different to those established which affected pellet resistance to compression (Stelte et al. 2011). Humidity is another factor that may also have an effect in compression resistance values. Compression resistance increases as pellet moisture content decreases (Rhén et al. 2005). In the present study, efforts were made to keep material at 10% moisture content before pelleting. However, this was not always possible due to the great capability of the drying equipment used, which did not allow for humidity control.

Durability is defined as the capability of pellets to support destructive forces and pressure during transportation (Tabil and Sokhansanj 1996). This parameter is considered acceptable when its value is greater than 80%, average when it is between 70% and 80%, and low when it is less than 70% (Colley *et al.* 2006). When applying these concepts to the durability obtained in the evaluation of pellets made from the twelve crops, it is possible to observe that the majority (CL, TG, PLP, EFB, OPMF, AD, GS, PP, SB and SO) possess an acceptable durability of 72.12% and 75.54%, considered of average durability (Table 3). Moreover, it is important to note that crops were not found to have low durability. The acceptable and average durability values obtained for the crops evaluated suggest that pellets will be of good performance. As Temmerman *et al.* (2006) mention, high durability values result in decreased risks associated to fire explosions during pellet transportation or storage as well as with dust emissions, and it avoids problems in the pellet feeding systems.

On the other hand, the differences found among durability values between crops (Table 3) may be explained by once more taking into consideration moisture content and particle size. Durability has been reported to increase as pellet moisture content increases (Colley *et al.* 2006, Filbakk *et al.* 2011), until reaching an optimum point which, once it has been surpassed, pellet durability and resistance decrease (Kaliyan and Morey 2009; Lee *et al.* 2013). Regarding pellet size, generally more durable pellets are produced when working with fine particles (Mani *et al.* 2003, Serrano *et al.* 2011). Although in the present study particle size was not determined, the methodology used for manufacturing pellets was different for some crops (PLP, SO, AD, SB, PP, OPMF and CA), which had to be adjusted through additional stages (cane sugar milling, predrying, etc.), as was mentioned earlier. The different pellet manufacturing processes used may have induced variations in particle size for the different crops, which would have affected their durability.

4.4. Relation between densitometry values and compression

The high amount of crops (CL, TG, OPMF, GS, PP, SB, PA, CA and SO) which presented a negative correlation between the force at break with CVlong and CVtrans (measured by X-ray densitometry) (Table 4), which also explain the greater variation percentage of the force at break

(Table 5), as well as the low amount of crops (PLP and AD) which presented correlations between the force at break and the average longitudinal and transversal density, (also measured by X-ray densitometry) (Table 4), suggest that the pellet compression resistance is more related to the density variation that pellets possess both in longitudinal and transversal directions, and that the higher the density variation within the pellet, the lower will be its resistance, or the lower will be the force at break that the pellet can sustain (Figure 6). This result was confirmed by Carone *et al.* (2011), who found high correlation between pellet density and compression stress in pellets of *Olea europeae* where, as density increases, the pellet compression resistance also increases.

It is important to highlight that the technique of X-ray densitometry to evaluate density is commonly utilized for studying materials and easily applied in comparison with other techniques that also evaluate pellet quality. It also determines pellet's mechanical resistance, a key parameter of pellet quality. Values reflected by this technique (average density and variation) show the variations in particle size, crop's internal structure and different factors that intervene in the pelletizing process (Mani *et al.* 2003, Rhén *et al.* 2005). This is therefore a useful technique to ensure quality and final resistance of pellets.

5. CONCLUSIONS

The use of X-ray photography for pellet quality evaluation shows that it is possible to observe cracks and irregularities, and clearer areas on the surface of the pellets. However, surface cracks and irregularities are not indicators of pellet durability or resistance to compression. Meanwhile, clearer surface areas are associated with high density values in these regions. Additionally, these regions are responsible for producing high variability in pellet's internal density.

The evaluation of mechanical properties showed that pellet durability and resistance to compression evaluated for the twelve crops can be considered as high, which warrants their adequate performance.

The X-ray densitometry evaluation showed that pellet evaluation by this technique permits the determination of pellet mechanical resistance, a very important quality parameter. The force at break and CVIong and CVtrans were correlated for a high number of the crops evaluated, suggesting that pellet compression resistance is more closely related with pellet density variations both in longitudinal and transversal directions.

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3. Artículo 3. Quality of pellets made from agricultural and forestry crops in Costa Rican tropical climates.

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Quality of Pellets Made from Agricultural and Forestry Crops in Costa Rican Tropical Climates

Carolina Tenorio, a Roger Moya, b,* Mario Tomazello Filho, c and Jorre Valaert d

Pellets may be produced with different types of agriculture or forestry crops in Costa Rica. This work evaluated the energy, physical, and mechanical properties of pellets fabricated from 12 types of agricultural and forestry crops (Ananas cumosos, Arundo donax, Coffea arabica, Cupressus lusitanica, empty fruit bunch and oil palm mesocarp fiber of the fruit of Elaeis guineensis, Gynerium sagittatum, Pennisetum purpureum, Phyllostachys aurea, Saccharum officinarum, Sorghum bicolor, and Tectona grandis), and similarities among these crops were established by multivariate principal component analysis. High variation was found in the pellet properties. The energy evaluation revealed that C. lusitanica and P. aurea are the crops with the best qualities for fuel use because of their high calorific values (from 16807 kJ/kg and 19919 kJ/kg, respectively) and low ash content (1.03% and 3.39%, respectively). As for physical properties, most crops exhibited values within the range noted by several authors and standards. All 12 pellet crops displayed high durability (from 72.12% to 92.98%) and compression force (from 295.18 N to 691.86 N). Moreover, the evaluation of crop similarities allowed the determination of four group combinations. Within these groups, C. lusitanica, P. aurea, and G. sagittatum had similar energy qualities and the best caloric characteristics.

Keywords: Biomass; Fuel; Pellet properties; Short-rotation crops; Mixture species

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INTRODUCTION

Environmental problems, increasing energy demands, and the decreasing availability of fossil fuels have stimulated the search for sustainable technologies based on renewable raw materials (Bringezu 2014). Biomass is one of the most promising energy sources, as it is an alternative to conventional energy sources such as oil and natural gas (Monteiro et al. 2012). One of its main advantages is that it is a clean and renewable product that contributes to reducing greenhouse gas emissions and dependency on fossil fuels (Monteiro et al. 2012; Daioglou et al. 2014). In this sense, the search for biomass from agricultural and forestry crops has advanced in the last few years (Hauk et al. 2014).

Nevertheless, the great amount of energy required to process biomass constitutes a limiting factor for its use, despite the fact that this energy requirement can be 70% less than what is required to process steel (Monteiro *et al.* 2012; Hauk *et al.* 2014). Its high moisture content, irregular shape and size, and low bulk density make it difficult to transport, store, and use in its original form (Hauk *et al.* 2014).

Conversion of biomass to pellets significantly reduces storage and transportation costs (Monteiro et al. 2012; Hughes et al. 2014). In addition, these pellets have higher density, are more homogeneous, and have greater energy potential (Dwivedi et al. 2014).

Currently, various raw materials are used to produce pellets in countries with temperate climates (Kuparinen *et al.* 2014). Many of these materials come from energy crops or industrial residues, mostly from the food industry. For example, some agricultural crops stand out in temperate climate regions, such as maize, sorghum, some potato varieties, and manioc, as well as short rotation forestry crops such as willow, pine, beech, and spruce (Shabani *et al.* 2013). In turn, a few agricultural and forestry crops have been used to manufacture pellets in tropical countries; some examples include coffee or forestry species like eucalyptus or tropical pines (Virmond *et al.* 2013; Lamers *et al.* 2014; Searle and Malins 2014).

There are a number of studies related to pellet characteristics and production involving species in temperate climates. These studies have focused on the following aspects: (i) the manufacturing process (Filbakk *et al.* 2011); (ii) improvement of conditions by means of additives or by treating the biomass before or after pellet manufacture, using roasting, for example; (iii) the evaluation of energy, physical, mechanical, and chemical aspects of pellet quality (Bergström *et al.* 2008); and (iv) pellet combustion and emission process evaluation (Abuelnuor *et al.* 2014).

In many small countries such as Costa Rica, pellets have gained popularity because some industries seek to switch from producing heat from fossil fuel sources to renewable sources to achieve carbon neutrality (Aragón et al. 2014). At present, for many tropical agricultural crops and forestry residues, the technological adaptations to the pellet production process (Aragón et al. 2014) and pellet quality evaluation are known (Tenorio et al. 2014); however, there is very limited information about the physical, chemical, or energy characterization of pellets produced from tropical crops in Costa Rica.

Therefore, the present work has the objective of evaluating pellet properties, taking into consideration energy (calorific value and combustibility index), physical properties (length, diameter, density, bulk density, and moisture absorption), mechanical properties (compression resistance and durability), and others parameter (ash, volatiles, and moisture content) of twelve agricultural and forestry crops (Ananas cumosos, Arundo donax, Coffea arabica, Cupressus lusitanica, empty fruit bunch and oil palm mesocarp fiber of the fruit of Elaeis guineensis, Gynerium sagittatum, Pennisetum purpureum, Phyllostachys aurea, Saccharum officinarum, Sorghum bicolor, and Tectona grandis) in Costa Rica. Finally, the similarities among these crops were established by multivariate principal component analysis.

EXPERIMENTAL

Materials

Twelve types of biomass from Costa Rican crops were selected to manufacture pellets; two were forestry species and ten were agricultural crops. The following three aspects were considered in this selection: their adaptability to the climatic and edaphic conditions of Costa Rica, an expected dry biomass production of over 20 ton/ha, and the possibility of pellet production throughout the year. Table 1 presents information regarding the 12 crops that were utilized.

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Crops	Scientific name	Origin	Abbreviation
Pineapple leaves	Ananas cumosos	Buenos Aires, Puntarenas	PLP
Giant cane	Arundo donax	Filadelfia, Guanacaste	AD
Coffee pulp	Coffea arabica	Tarrazú, San José	CA
Sawlog residuals	Cupressus Iusitanica	Agua Caliente, Cartago	CL
Empty fruit bunch of the oil palm	Elaeis guineensis	Parrita, Puntarenas	EFB
Oil palm mesocarp fiber of the fruit	Elaeis guineensis	Parrita, Puntarenas	OPMF
Wild cane	Gynerium sagittatum	Río Frío, Limón	GS
King grass	Pennisetum purpureum	Paraíso, Cartago	PP
Golden bamboo	Phyllostachys aurea	Cartago, Cartago	PA
Sugarcane	Saccharum officinarum	San Carlos, Alajuela	SO
Sorghum	Sorghum bicolor	Upala, Alajuela	SB
Sawlog residuals	Tectona grandis	Abangares, Guanacaste	TG

Table 1. Description of 12 Crops used for Pellet Manufacture

Methods

Pellet manufacturing process

The pellet manufacturing process was carried out at Pelletics S.A. (http://pelletics.com/), located in San Carlos in Alajuela province (Costa Rica). Aragón et al. (2014) detailed the pellet manufacturing process from the moment the crops enter the plant to the pelletizing process.

The manufacture of pellets from CL, GS, PA, and TG used the following equipment and procedures. The material was chipped in a JENZ chipper, model AZ 50 (Germany), and the milling was performed using a KAHL fixed ring matrix with holes 15 mm in diameter (Germany). Afterwards, the granulate material was dried to reach 8% to 14% moisture content using a rotary drum (12 m long x 3 m diameter), and air-heated to 400 °C. Finally, the pelletizing process was performed in a KAHL machine, model 35780, consisting of a fixed ring matrix that was 780 mm in diameter, containing holes 6 mm in diameter and 30 mm long, with three rotating rollers; a temperature of 120 °C was reached during the process.

The process was adjusted for the remaining species through additional stages. The adjustments were as follows: (i) For the PLP, SO, AD, and SB crops, the chipper illustrated in Fig. 1 used in the production system did not function adequately; thus, specialized machinery that can process other types of biomass, specifically sugarcane mills or chippers, were required; (ii) For the AD and PP crops, pre-drying was carried out to reach the optimum moisture for the chipping process. Pre-drying consisted of leaving the cut stems in the field for a three-day period; the semi-dry material was then taken through the chipping process; (iii) The EFB, OPMF, and CA did not need to be chipped and were taken directly to the milling stage; this material was collected from processing centers, and its moisture was similar to that of wood; and (iv) Once SB and SO were milled, a biomass

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pre-treatment was performed that consisted of extracting water using a solid separating press.

Determination of energy properties and ash content, volatiles, and moisture content

The properties determined included net caloric value (NCV), ash content, moisture content (MC), percent volatiles, and fuel value index (FVI). The NCV was determined in the absence of water (0% moisture content) using Parr's calorimetric test in accordance with the ASTM D-5865 04 (2003) standard. To determine the ash content, 10 randomly selected 2 g pellet samples were used and the ASTM 1102-84 (2013a) standard procedure was followed. The pellets' MC was determined using a moisture scale, model MB45, made by OHAUS (USA), which determines moisture with respect to initial weight. For the percent volatiles, 10 pellet samples of 3 g each were used, and the ASTM D1762-84 (2013b) standard was followed. The FVI was calculated using the NCV, density, and ash content, based on the methodology proposed by Purohit and Nautiyal (1987). Ten pellets with an approximate weight of 0.60 g each were randomly selected among the pellets manufactured for each property.

Determination of physical properties

The physical properties determined were the pellets' length, diameter, moisture absorption percentage, and bulk density. To determine length, diameter, and moisture absorption, a representative random sample of 30 pellets *per* crop was used. To determine moisture absorption, pellets were placed in a desiccator containing a saturated solution of potassium nitrate at 22 °C (21% equilibrium moisture content); pellets were weighed on a weekly basis until they reached constant weight. Samples were weighed before and after this period. The absorption percentage was calculated with Eq. 1:

Moisture absorption (%) =
$$\frac{\text{weight at 21\%(g)-initial weight(g)}}{\text{initial weight (g)}} * 100$$
 (1)

To determine the apparent density, small quantities of pellets were slowly added to a beaker, filling it up to its 500-mL capacity. Then, the weight of pellets occupying this space was determined. The apparent density was determined by the ratio between the weight and the volume occupied by the pellets. Ten pellets were randomly selected from the total crop set, and the length and diameter were measured with a calibrator. Lastly, their mass was calculated on an analytical balance. The pellets' bulk density can be obtained using Eq. 2:

Pellet bulk density
$$(\frac{g}{cm^2}) = \frac{pellet \ mass \ (g)}{pellet \ volume \ (cm^2)}$$
 (2)

Determination of mechanical properties

Pellet mechanical durability and compression resistance were determined. The DD CENT/TS 15210-1 (2005) standard was used to calculate mechanical durability. For this test, 10 representative pellet samples of 500 g each were sifted through a sieve with an aperture of 3.36 mm to eliminate fine particles. Then, the sifted samples were placed in equipment proposed by the standard, which was fabricated for this purpose, at a speed of 50 rpm for 10 min. Later, the samples were removed, sifted once more, and weighed.

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For the compression resistance test, 10 pellets with an approximate length of 13 mm were randomly selected. The test was performed longitudinally on the pellet according to the methodology proposed by Aarseth and Prestlokken (2003) using a Tinus Olsen (USA) universal test machine, model H10KT, with a capacity of 1 ton. For this test, a compression charge speed of 0.02 mm/s was applied. This test determines the pellet's force at break vs. deformation measurements. The pellet's force at break was reported.

Statistical analysis

A descriptive analysis was performed (median, standard deviation, and maximum and minimum values) for the following variables: pellet length and diameter, NCV, ash content, percent volatiles, MC, FVI, apparent density, bulk density, absorption percentage, durability, and force at break. In addition, it was determined whether variables complied with the premises of normal distribution, homogeneity of variances, as well as the presence of extreme values. A variance analysis was applied to verify the existence of significant differences among the averages of the variables (P<0.05). Tukey's test was carried out to determine the statistical differences among crops, for the mean value of each of the abovementioned values.

Finally, a multivariate principal component analysis was used among biomass crops and all evaluated energy, physical, and mechanical properties. Multivariate principal component analysis is appropriate when data have been obtained for a number of observed variables; a smaller number of artificial variables (called principal components) that will account for most of the variance in the observed variables can be obtained. The principal components may then be used as predictor or criterion variables in subsequent analyses (Johnson and Wichem 1992). Also, from the principal components, two fist components were selected and were interpreted according to properties correlated with these components. In addition, multivariate analysis provided Eigenvalues, a scale associated with a given linear transformation of a vector space provided to each property evaluated.

RESULTS

Energy, Physical, and Mechanical Properties

Table 2 shows the energy properties of the 12 different crops for pellet manufacture. The NCV varied from 11,616 kJ/kg to 19,919 kJ/kg, and four groupings were created based on the statistical differences in the energy properties: (i) PA and GS, with the highest values, (ii) followed by PP, SB, CL, AD, and, OPMF, (iii) then by TG and EFB, and lastly (iv) the group with the lowest values, composed of CA, SO, and PLP. The values obtained for ash content ranged from 1.0% to 10.5%, and five groupings were formed: (i) AD, with the highest value, (ii) followed by PP, CA, SO, and OPMF, (iii) another group formed by PLP, EFB, SB, and GS, (iv) another group formed by PA and TG, and (v) CL. The percent volatiles for the 12 crops varied between 69.2% and 78.0%, and the crops were grouped into four categories: (i) SO and CL, presenting the highest values, (ii) followed by TG, PA, and GS, (iii) PLP, SB, OPMF, and EFB, and (iv) one last group, formed by AD, CA, and PP, which represent the lowest ash percentage values. The FVI test determined that CL had the highest FVI, followed by PA; the remaining crops (PLP, EFB, OPMF, CA, GS, PP, SO, SB, and TG) had statistically similar FVI values, while AD had the lowest value. Pellet MC for the 12 crops varied from 6.7% to 12.6%, and once more four groups were established: (i) crops with the highest MC were SB, PP, TG, and AD, (ii) followed by PLP

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and CA, (iii) then by SO, OPMF, and EFB, and lastly (iv) CL and PA, with the lowest values.

Table 2. Energy Properties of 12 Pellets Crops from Costa Rica

Crops	Net caloric	Ash content	Volatile	Moisture	Fuel value
Crops	value (kJ/kg)	(%)	content (%)	content (%)	index
A. cumosos (PLP)	11,617 (3.6)E	6.1 (30.7) ^{CD}	73.1 (1.3) ^c	10.9 (3.3)B	242 (46.1)CD
A. donax (AD)	15,930 (4.0)BC	10.5 (4.6) ^A	70.5 (0.9)DE	12.0 (4.7) ^A	163 (6.1)D
C. arabica (CA)	12,249 (4.0)E	6.7 (8.8)BC	70.0 (0.8)E	10.1 (3.3)BC	232 (12.4) ^{CD}
C. lusitanica (CL)	16,807 (7.9)B	1.0 (28.0)F	76.7 (0.2) ^{AB}	7.5 (16.3)E	2803 (25.7)
E. guineensis (EFB)	14,182 (9.7)D	5.7 (12.7) ^{CD}	71.7 (0.1) ^{CD}	9.0 (6.9)D	349 (15.0)CL
E. guineensis (OPMF)	15,831 (5.7)BC	6.2 (20.9)BC	72.4 (0.8) ^c	9.2 (5.1)CD	340 (18.6) ^{Ct}
G. sagittatum (GS)	18,750 (9.9) ^A	4.9 (7.5)D	75.1 (0.3)B	9.7 (6.2)CD	491 (17.4)°
P. purpureum (PP)	16,979 (3.7)B	7.5 (4.3)B	69.2 (1.6)E	12.1 (3.8) ^A	244 (4.0) ^{CD}
P. aurea (PA)	19,919 (7.2) ^A	3.4 (13.9)E	75.3 (0.7)B	6.7 (5.9)E	1039 (10.1)
S. officinarum (SO)	12,146 (2.9)E	6.6 (17.6)BC	78.0 (0.1) ^A	9.7 (9.1) ^{CD}	212 (12.8) ^{Cl}
S. bicolor (SB)	16,906 (5.6)B	5.5 (3.0)CD	72.6 (0.9) ^c	12.6 (5.6) ^A	273 (9.7)CD
T. grandis (TG)	15,261 (2.3)CD	3.2 (24.1) ^E	75.9 (0.1)B	12.1 (3.7) ^A	463 (19.5) ^c

Values in parentheses are the variation coefficients (average*100/standard deviation)
Different letters for each parameter represent statistical differences between crops (significances at 95%)

Regarding the physical properties (Table 3), it was found that pellet length varied from 12.3 to 27.7 mm. *G. sagittatum*, CL, and CA had the longest pellets, while AD pellets had the lowest values. For the remaining crops (PLP, EFB, OPMF, PA, PP, SO, SB, and TG), pellet length varied between 15.4 and 22.9 mm. In turn, pellet diameter varied from 5.9 mm to 6.6 mm. The following five crop groupings were created based on pellet diameter: (i) SO, with the greatest diameter (6.6 mm), (ii) followed by GS, SB, PLP, and TG, (iii) then by CL, PP, and OPMF, (iv) another group formed by EFB, AD, and CA, and finally (v) PA.

Table 3. Physical Properties of 12 Pellets Crops from Costa Rica

Crops	Length (mm)	Diameter (mm)	Moisture absorption (%)	Apparent density (kg/m³)	Density (g/cm³)
A. cumosos (PLP)	15.4 (16.6)FG	6.2 (1.9)BC	8.9 (22.4) ^{AB}	2 2020 S	1.23 (2.3) ^{AE}
A. donax (AD)	12.3 (38.4) ^G	6.1 (0.6) ^E	9.7 (15.3) ^A	456 (3.0)F	1.29 (3.6) ^A
C. arabica (CA)	24.8 (14.7) ^{AB}	6.07 (2.0)E	8.1 (53.6)BC	595 (2.1)A	1.28 (3.0)AC
C. lusitanica (CL)	27.3 (18.4) ^A	6.2 (1.2) ^{CDE}	9.3 (13.7) ^{AB}	549 (3.4) ^c	1.21 (7.2) ^{AEF}
E. guineensis (EFB)	22.9 (9.6)BC	6.1 (2.0)DE	5.1 (24.9)F	575 (1.53)B	1.23 (3.7)AD
E. guineensis (OPMF)	17.3 (26.7)EF	6.1 (4.1)CDE	5.7 (14.7)DEF	596 (1.6)A	1.19 (7.1)BCDE
G. sagittatum (GS)	27.7 (9.1) ^A	6.3 (1.3)B	5.7 (15.3)DEF	542 (1.5)CD	1.23 (2.2) ^{AD}
P. purpureum (PP)	18.2 (32.5)DEF	6.1 (1.9)CDE	5.3 (7.6)EF	524 (3.0)D	1.29 (2.3) ^A
P. aurea (PA)	21.0 (12.0) ^{CD}	5.9 (4.9)F	6.9 (16.5) ^{CD}	490 (1.4)E	1.17 (4.4)BDEG
S. officinarum (SO)	18.4 (16.8)DEF	6.6 (3.3) ^A	5.7 (13.2)DEF	500 (4.2)E	1.10 (8.5) ^G
S. bicolor (SB)	16.8 (26.1)EF	6.3 (1.7)BC	6.6 (11.2)DE	386 (2.2)G	1.13 (5.1)BFG
T. grandis (TG)	19.8 (12.6)CDE	6.2 (2.9)BC	5.5 (19.2)DEF	378 (3.7)G	1.14 (4.7)BEG

Values within parentheses are the variation coefficients (average*100/standard deviation)
Different letters for each parameter represent statistical differences between crops (significance at 95%)

There was not enough material for testing the bulk density with PLP

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The percentage of moisture absorption varied from 5.1% to 9.7%, with four groupings: (i) AD, CL, PLP, and CA were the crops with the highest moisture absorption, (ii) followed by PA and SB, (iii) then by SO, GS, OPMF, TG, and PP, and finally (iv) EFB had the lowest moisture absorption percentage. The apparent density ranged from 386 to 596 kg/m³; in this case, it was not possible to make any groupings because of the large statistical differences among the crops. Therefore, OPMF and CA presented the highest values, SB had the lowest value (386 kg/m³), and the remaining crops (PLP, AD, CL, EFB, GS, PP, PA, SO, and TG) had apparent densities ranging from 456 to 575 kg/m³. The bulk density of pellets varied from 1.10 to 1.29 g/cm³ for all crops; PP, AD, CA, GS, EFB, PLP, and CL presented the highest values (between 1.21 and 1.29 g/cm³), SO had the lowest value (1.10 g/cm³), and the remaining crops (OPMF, PA, TG, and SB) had values between 1.13 and 1.19 g/cm³.

Figure 1 shows pellets mechanical durability and force at break. For mechanical durability, AD, EFB, OPMF, PP, and TG possessed the highest durability percentages (92.% to 93%), followed by PLP and SO, with 90% and 91% respectively, while the rest of the crops (CL, SB, GS, and CA) showed values between 76% and 88%. Lastly, PA had the lowest durability, with a value of 72% (Fig. 1a). The force at break varied from 295 to 692 N, and the differences found among the medians permit three groupings: (i) PP, GS, and CL were the crops with the highest values, at 571, 634, and 692 N, respectively, (ii) CA, AD, EFB, and OPMF had intermediate values, between 416 and 485 N, and (iii) SO, PA, SB, TG, and PLP had the lowest force at break, ranging from 295 to 375 N (Fig. 1b).

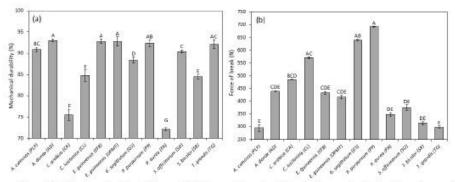


Fig. 1. (a) Mechanical durability and (b) force at break of 12 pellet crops in Costa Rica. Legend: Different letters for each crop represent statistical difference between crops (significances at 95%)

Figure 2 illustrates the behavior of one representative pellet for each crop in the force vs. deformation curve, according to three groups established in force to breakage: (i) PP, GS, and CL tended to reach high force levels (superior to 650 N) at low deformation values and present curves with steep slopes in the elastic area of the pellet; (ii) EFB, OPMF, and AD behaved differently; at the same deformation levels, the force they could withstand was lower (within the range of 500 N); and (iii) PA, SB, TG, PLP, and CA possess lower forces than those of the other groups at the same deformation values. In addition, their curve slopes were lower in relation to the other species. In contrast, SO presents a different case because its pellets were found to withhold forces superior to 500 N, which is the reason for placing it among species of the second group. Nevertheless, the deformation values of

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0.2 or 0.4 mm exhibited breaking forces within the range of crops from the third group; in any case, the crop's slope was similar to the crops of the second group (Fig. 2).

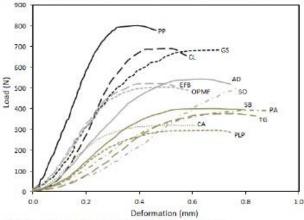


Fig. 2. Force vs. deformation for 12 pellet crops from Costa Rica

Multivariate Analysis

The multivariate principal component analysis (Table 4) of the physical, mechanical, and energy properties of the crops revealed that the first two principal

Table 4. Correlation Matrix of Multivariate Analysis for All Parameters Evaluated

Parameter	Factor 1	Factor 2
Moisture absorption	-0.26	-0.27
Length	-0.78**	0.01
Diameter	0.27	0.44
Calorific value	-0.46	-0.10
Moisture content	0.80**	-0.15
Ash content	0.76**	-0.53
Volatiles	-0.41	0.80
FVI	-0.87**	0.12
Apparent density	-0.05	-0.97**
Bulk density	-0.41	-0.50
Mechanical durability	0.47	-0.14
Force of break	-0.38	-0.72**
Compression strength	-0.53	-0.50
Eigenvalue	3.91	3.23
% Total	30.07	24.88
Eigenvalue cumulative	3.91	7.14
Total cumulative	30.07	54.95

^{**}Parameter of pellet that affect statistically at 99%.

components could explain 55% of the variations of the pellet properties, which were considered in this study. Thirty percent of the data variations could be explained by Factor 1, where FVI, MC, pellet length, and ash percentage showed statistically significant effects on this factor, for which pellet length and FVI were negatively correlated and MC and ash content were positively correlated (Table 4). Similarly, Factor 2 explained 25% of the data variations of all pellet parameters, and it was negatively affected by apparent density and force at break (Table 4).

Multivariate analysis provided Eigenvalues for each principal component. The eigenvalues are a scale associated with a given linear transformation of a vector space provided to each parameter evaluated. If a scatterplot is created for the Eigenvector of each principal component in each of the crops analyzed (Fig. 3), one can observe four groups for the physical, mechanical, and energy properties: the first group is composed of CL, PA, and GS; the second group is formed by TG, SO, and SB; the third group is composed of OPMF, EFB, and CA; and the last group is composed of PP and AD.

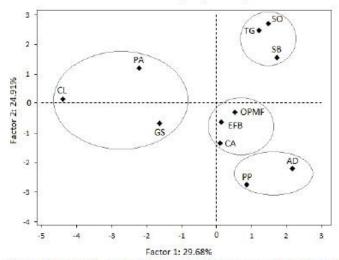


Fig. 3. Relationship of Eigenvectors of Factors 1 and 2 from the multivariate analysis of the physical, mechanical, and energy properties of species tested

ANALYSIS

Energy Properties

The NCV values varied from 11,616 to 19,919 kJ/kg (Table 2). The caloric values agree with those reported for fast-growing timber species in Costa Rica, which vary from 16,500 to 20,600 kJ/kg (Moya and Tenorio 2013). T. grandis, PLP, EFB, OPMF, AD, CA, and SO had values inferior to those for timber species, which probably makes them less recommendable as an alternative fuel compared to fast-growing timber species. Although there are many standards being used in European countries, they are irregular, but the German and Swiss standards are more developed and more effective for pellet characterization (Garcia-Maraver et al. 2011); thus they are considered in the study for comparison. The German standard (DIN 1996) recommends an NCV range for fuel

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material from 17,500 to 19,500 kJ/kg. Of the crops evaluated, only pellets from PA and GS fulfilled these recommendations. Based on the Swedish standard (SS 1998), which is less restrictive, all species can be commercialized in the Swedish marked. PA, SB, PP, and GS are grouped in Group 1 (NCV > 16, 890 kJ/kg); AD, CL, OPMF and TG are grouped in Group 2 (NCV varying from 15,100 to 16,890 kJ/kg), and other species are grouped in Group 3, because their values are lower than 15,100 to 16,890 kJ/kg. On the other hand, the European standards (CEN/TS 2004) are insufficiently restrictive because the heating value should be established by the owner manufacturer.

In relation to ash content, variation among crops was high, from 1.0% to 10.5%, and when comparing these values with those reported for timber species in Costa Rica (between 0.2% and 4.0%) by Moya and Tenorio (2013), it was observed that only CL, TG, and PA had ash content within the range of these species. Similarly, the ash content values obtained in this study according to ASTM D-1102-84 (2013a) did not comply with the permitted 0.5% limit according to the DIN 51731 (1996) standard or 1.5% permitted by Swedish standard (SS 1998). Another important aspect to consider in relation to the high ash content values (over 4%) is that a high ash percentage may lead to the corrosion of burners or boilers and abrasion of equipment (Mande 2009).

The percent volatiles variation in this study was between 69.24% and 77.96% (Table 2), which are higher values than those presented by Kataki and Konwer (2002) for timber species from Northeast India, which ranged from 13.5% to 40.1%. According to several authors, a high volatiles percentage produces more heat during combustion, which makes a material burn faster, making it less desirable as fuel (Jain 1994; Kataki and Konwer 2002).

There are different viewpoints concerning the MC that pellets should possess for good performance; normally, pellets contain a MC between 8% and 12% (Lehtikangas 2001; Kaliyan and Morey 2009). The MC of pellets manufactured in this study was between 6% and 12%, and crops such as CL and PA have a MC inferior to 8% (Table 2). However, according to several authors, high MC tends to decrease the energy potential of timber species (Jain 1994; Kataki and Konwer 2002). In the case of pellets, Telmo and Lousada (2011) point out that pellet NCV may double if pellets are completely dry. The German standard (DIN 1996) recommends an MC less than 12%, then MC of almost pellet fulfils of German standard, except PP and SB, which are in border of this range. Meanwhile, CL, EFB, OPMF, GS, PA, and SO fulfill the Swish standard (SS, 1998). On the other hand, European standards (CEN/TS 2004) establish ranges of variation for MC, such that all pellets species can be grouped in some of these ranges.

The FVI values for the 12 crops varied from 163 to 2803 (Table 2). When comparing these values to those reported by Moya and Tenorio (2013) for solid wood in Costa Rica, PLP, AD, PP, SB, CA, and SO possessed lower FVI than the range (from 337 to 6390) indicated by these authors; hence, they are less recommendable for fuel use in comparison with timber species. The FVI value is one of the best parameters for determining how desirable a species can be as fuel, mostly because it takes into account NCV and density as positive factors and ash content and MC as negative factors (Purohit and Nautiyal 1987; Jain 1992). Their FVI values suggest that CL and PA are the crops with the best properties for fuel use and therefore for pellet manufacture.

The NCV variations observed in the 12 crops (Table 2) can be explained by the variations in chemical properties for each biomass type, such as ash content, volatile substances, extractives, and fixed carbon (Gominho *et al.* 2012; Moya and Tenorio 2013). According to several authors, the ash content and volatiles directly affect the energy

conversion process and specifically decrease the NCV (Kataki and Konwar 2002; Kumar et al. 2009; Gominho et al. 2012). Even so, the correlation index indicates weak relationships between NCV and both ash content and volatiles (Figs. 4a and 4b), which could mean that NCV variations between crops could be the result of other chemical properties that were not evaluated in this study. For example, high extractives content and fixed carbon in biomass may influence the NCV (Kataki and Konwer 2001; Moya and Tenorio 2013).

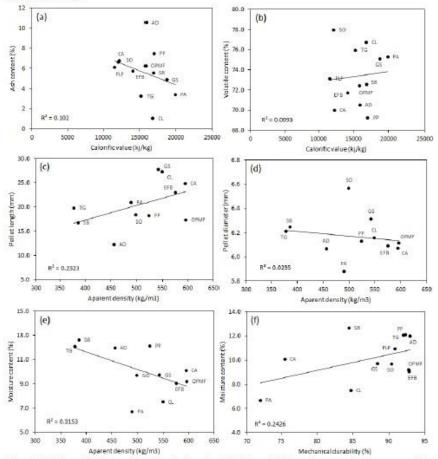


Fig. 4. Relationship between net caloric value (NCV) and different parameters measured in the pellet for 12 species tested.

Physical Properties

Faseina (2008), in a study performed on peanut hull pellets, points out that the optimum moisture absorption point for pellets is from 3% to 5% and that an additional increase in absorbed moisture could result in decreasing the quality and strength characteristics of pellets. The absorption capacity obtained in this case for the 12 crops ranged from 5.1% to 9.7%, higher values than those of the abovementioned study. Pellets

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produced with these crops could present moisture absorption problems and thus strengthrelated problems.

The values obtained for apparent density (Table 3) show that, for CA, OPMF, EFB, CL, GS, PP, SO, and PA, the apparent density values were compatible with those found in pellets manufactured from maize, wheat, and sorghum residues, where variation was from 479 to 649 kg/m³ (Theerarattananoon et al. 2011). However, values obtained for TG, SB, and AD (Table 3) were inferior to the ones mentioned by these authors. Low bulk density values have an effect on transportation costs because the energy value per unit volume is lower (Theerarattananoon et al. 2011). The average bulk density obtained for the pellets of the 12 crops varied from 1.10 to 1.29 g/cm³ (Table 3). Such density values can be found within the range suggested by DIN 51731 (1996) and Swish standard (SS 1998), which varied from 1.0 to 1.4 g/cm³. But European standards (CEN/TS 2004) are insufficiently restrictive, because the bulk density value should be established by the owner manufacturer.

Variations in the physical properties of pellets (Table 3) may be clarified from two angles: factors related to the material and factors related to the process. For water absorption capacity, variations among species can be attributed to variations in pellets' MC. Pellets with high MC tend to absorb less water, while pellets with low MC tend to absorb more water. This behavior was found in SB, PP, and TG crops, which had low absorption values and high MC (Tables 2 and 3). The differences among the bulk densities of the 12 crops may be explained by variations in size (pellet length and diameter). Figure 4c and 4d show the correlations between pellet length, diameter, and apparent density. The apparent density increases with increasing pellet length, but with respect to the diameter, no correlation with the apparent density is observed. Another aspect that influences apparent density variations is MC. Several authors have suggested that an increase in pellet MC results in a linear decrease in apparent density (Mani et al. 2006; Fascina 2008); such a correlation was found in the crops studied (Fig. 4e), but this correlation was low (R²=0.32).

The bulk density variations between crops may be caused by the internal structure of each of the analyzed crops, as well as the temperature and pressure applied during the pelletizing process (Rhén et al. 2005; Mani et al. 2006). Gilbert et al. (2009) indicated that at temperatures between 14 and 50 °C, the density increases substantially, while at higher temperatures, between 75 and 95 °C, pellets tend to maintain a stable density. Several authors indicate an existing relationship between pressure and density, where density increases exponentially with an increase in pelletizing pressure, until reaching a point of maximum density (Husain et al. 2002; Rhén et al. 2005).

Mechanical Properties

Durability is defined as the capacity of pellets to sustain destructive loads and forces during transportation (Tabil and Sokhansanj 1996), so it is of utmost importance to have adequate values for this parameter; Colley et al. (2006) suggest the following three categories: (i) acceptable, when the durability is greater than 80%, (ii) average, when durability varies between 70% and 80%, and (iii) low, when the parameter is lower than 70%. When applying these categories to the durability values obtained for the pellets of the 12 crops, one can confirm that the majority of crops (CL, TG, PLP, EFB, OPMF, AD, GS, PP, SB, and SO) exhibited acceptable durability, with values greater than 80%. Meanwhile, PA and CA showed average durability (Fig. 1a), and no crops were found to have low durability. The categories obtained for the crops evaluated (acceptable and

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average) suggest that pellets will perform well, as according to Temmerman et al. (2006). The values obtained in durability did not fulfill to DIN (DIN 1996) or Swish standard (SS 1998). But European standards (CEN/TS 2004) are not restrictive enough because the durability value is greater than 90%; thus, many species (PLP, AD, EFB, OPMF, PP, SO and TG) fulfill this standard.

The differences in durability among crops (Fig. 1a) can be focused by considering MC and particle size. Durability has been reported to increase with an increase in MC (Colley et al. 2006; Filbakk et al. 2011), and pellets from the 12 crops evaluated were observed to have a similar behavior (Fig. 4f), however a low R² value was observed, which means that the durability cannot be explained solely on the basis of variations in MC. With respect to particle size, generally more durable pellets are produced when working with fine particles (Mani et al. 2006; Serrano et al. 2011). Although in this present study particle size was not determined, as was mentioned before, the methodology used to manufacture pellets was different for some crops (PLP, SO, AD, SB, PP, OPMF, and CA). This could have caused different particle sizes among crops and low particle uniformity, which would affect their durability.

The force at break values found in this study (Fig. 1b) were superior to those obtained by García-Maraver et al. (2010) for pellets made from olive tree branches. It may be inferred from this comparison that having an adequate force at break value allows pellets to maintain their shape during transportation (Kaliyan and Morey 2009). To explain the differences found in force at break of the different crops (Fig. 1b), several studies suggest that the pellet production stages, such as drying at high temperatures, milling, and pressing, along with the MC of the materials, affect the final strength properties of pellets (Rhén et al. 2005; Gilbert et al. 2009; Serrano et al. 2011). In this study, the pelletizing process was the same for all crops; however, because of the properties of each crop, there could have been temperature and pressure variations during the pelletizing process, different to those referenced, which could affect pellet compression resistance, lowering their force at break. As for moisture, the MC of the material before pelletizing should be close to 10%. Nevertheless, it was not possible to obtain this moisture level in the present study for the crop pellets evaluated, as MC varied from 6.7% to 12.6%. Also some studies have pointed out that the compression strength increases with decreasing MC (Rhén et al. 2005). Still, for this study, it was not possible to prove this, as crops with high MC also possessed high force at break values. For example, PP had the highest force at break, of 692 N, but it also had one of the highest MC among the pellets of the crops evaluated (12.1%) (Table 2).

Multivariate Analysis

When evaluating the similarity among crops considering all pellet properties, the multivariate principal component analysis showed that a high percentage of variation was focused on two factors: the first factor is related to the energy capacity of pellets, because it is related to FVI, MC, and ash content (Table 4), while the second factor is related to their strength and presents a correlation with density and force at break during compression (Table 4). This permitted the groupings detailed in Fig. 3. Such a relationship allows these crops to be combined without altering the thermic behavior of the equipment where pellets are being used. For example, no behavioral changes are likely to be expected in a burner when CL, PA, or GS pellets are used, which form a set of species having similar thermic conditions. This means that, when pellet supplies are scarce, adding or mixing pellets of another crop could occur without altering the thermic or structural behavior.

Likewise, the groupings obtained through multivariate principal component analysis will make it possible to establish cultivating policies for these crops. For example, a company wishing to standardize the raw material utilized to manufacture pellets of similar conditions could establish a policy of cultivating species such as CL, PA, and GS, which have similarly high NCV; or AD and PP, which have the greatest densities (Tables 2 and 3).

Nevertheless, to verify this grouping, pellet behavior should be complemented with more specific studies, such as thermogravimetric analysis, which would allow more precision on energy properties (Skreiberg et al. 2011), or other indices created to evaluate the capacity of biomass to produce heat (Sommersacher et al. 2011).

CONCLUSIONS

- During the evaluation of pellet energy properties, it was found that Cupressus lusitanica (CL) and Phyllostachys aurea (PA) are the crops with the best properties to be used as fuel, and certainly to manufacture pellets, mostly due to their high net caloric value (NCV) and low ash content. There was a high variation in the pellets' physical properties, mostly as a consequence of their moisture content (MC). Nevertheless, the majority of crops had values within the ranges reported by several authors and standards. The mechanical properties of pellets from the 12 crops present overall good durability and resistance properties, ideal for their storage and transportation.
- 2. The multivariate principal component analysis determined four crop groupings having similar energy or physical properties, allowing for possible material combinations. Crops such as CL, PA, and Gynerium sagittatum (GS) present the highest NCV; Tectona grandis (TG), Sorghum bicolor (SB), and Saccharum officinarum (SO) have similar forces at break; Arundo donax (AD) and Pennisetum purpureum (PP) possess the highest densities; and Coffea arabica (CA), Elaeis guineensis empty fruit bunch (EFB), and mesocarp fiber of the fruit (OPMF) have similar FVI. The species within groups can be combined to obtain raw material for uniform pellet manufacturing. However, it is necessary to verify these groupings with a study considering other parameters, such as decomposition and ignition temperature provided by thermogravimetric analysis.

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4. Artículo 4. Evaluacion de la combustión de pellets fabricados con cultivos agrícolas y forestales de los climas tropicales de Costa Rica utilizando un quemador doméstico.

Evaluación de la combustión de pellets fabricados con cultivos agrícolas y forestales de los climas tropicales de Costa Rica utilizando un quemador doméstico

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Resumen

En este trabajo se presentan los resultados de la evaluación de la combustión de pellets fabricados de con dos cultivos forestales y ocho cultivos agrícolas en Costa Rica utilizando un quemador domestico de 50 kwh. Se evaluó el consumo de biomasa, las temperaturas en la salida del quemador y en la chimenea, las emisiones (O₂, CO₂, CO, NO, NO₂, NO_X y SO₂), las pérdidas de calor y la eficiencia. Se encontró que el consumo de pellets varía de 2,5 a 3,4 kg/hora, siendo el menor en *Tectona grandis* y el mayor en oil palm mesocarp fruit. La temperatura de salida en el quemador varío de 400 a 613 °C y la temperatura en la chimenea varío de 205 a 322 °C, la temperatura más baja corresponde a *Phyllostachys aurea* y la mayor al residuo de la palma de aceite. *Tectona grandis* presentó la mayor variación de la temperatura, mientras que *Cupressus lusitanica* la menor variación. Un resultado que resalta en la medición de las emisiones, es que los dos cultivos forestales son los que presentan la menor cantidad. En relación a la eficiencia en combustión el mejor cultivo fue *Gynerium sagitattatum*, mientras que las especies forestales presentan una eficiencia intermedia entre los cultivos analizados.

Palabras clave: biomasa, domestic stove, pellet properties, short-rotation crops.

1. INTRODUCCION

Los problemas ambientales, la creciente demanda de energía y la menor disponibilidad de combustibles fósiles, han fomentado la necesidad de desarrollar tecnologías sostenibles basadas en materias primas renovables (Bringezu 2014). La biomasa es una de las fuentes energéticas más prometedoras ya que es una alternativa a las fuentes convencionales de energía tales como petróleo y el gas natural, entre otras. Entre las ventajas que tiene el uso de la biomasa está principalmente que es un producto limpio y renovable, lo cual contribuye a la reducción de las emisiones de gases de efecto invernadero y a la dependencia de los combustibles fósiles (Monteiro *et al.* 2012; Daioglou *et al.* 2014). En este sentido la búsqueda de biomasas provenientes de los cultivos agrícolas o de residuos forestales ha tomado un importante desarrollo en los últimos años (Hauk *et al.* 2014).

Sin embargo, para manipular esta biomasa se requiere de un gran consumo energético (Monteiro *et al.* 2012; Hauk *et al.* 2014) debido a su alta humedad, sus formas y tamaños irregulares, y su baja densidad aparente, lo que hace de la biomasa un material difícil de transportar, almacenar y utilizar en su forma original (Hauk *et al.* 2014). Convertir esta biomasa en pellets reduce significativamente los costos de almacenamiento y transporte (Monteiro *et al.* 2012; Hughes *et al.* 2014). Además, la biomasa pelletizada posee una alta densidad, es más homogénea y posee un mayor potencial energético (Dwivedi *et al.* 2014).

Las materias primas utilizadas hoy en día para la producción de pellets son muy variadas en los países de clima temperado (Kuparinen *et al.* 2014). Pero en el caso de países tropicales pocos cultivos, tanto agrícolas como forestales, han sido utilizados para la fabricación de pellets (Virmond *et al.* 2013; Searle and Malins 2014).

Los estudios en especies en climas temperados están relacionados con la producción de pellets y sus características. Dichos estudios, se concentran en diversos aspectos, entre los que destaca la evaluación del proceso de combustión (Roy et al., 2011) y de emisiones de los pellets (Lee et al., 2011, Abuelnuor *et al.* 2014). Al respecto se señala que el conocimiento del potencial de combustión de las materias primas permite establecer las mejores condiciones para la generación de calor (Roy et al., 2011).

En el caso de la biomasa el alto contenido de materiales volátiles (4-8 veces del carbono fijo contenido en el material) y la presencia de hidrocarburos pesados, hacen necesaria una adecuada mezcla y reacción durante la combustión para lograr la máxima eficiencia posible

(McKendry, 2002). Una reacción poco eficiente incrementa las concentraciones de CO y una alta producción de polycyclic aromatic hydrocarbons (Johansson et al, 2004). Otro aspecto también importante en una adecuada combustión es la poca generación de material residual (cenizas, escoria y material sin combustionar) ya que este se acumula en el horno y pueden causar problemas de operación en los equipos de quemado (Ståhl and Wikström, 2009), afectando la eficiencia de la biomasa o el equipo de combustión.

La eficiencia en el proceso de combustión está relacionado con los diferentes tipos de biomasa (McKendry, 2002), los cuales producen diferentes calidades de pellet. De ahí que en la actualidad muchos estudios se concentran en la eficiencia de la combustión de los diferentes tipos de biomasa (Garcia-Maraver et al., 2014). Una baja calidad de pellet durante la combustión, además de los problemas en los equipos, puede traer consigo altos niveles de gases de combustión, entre los que destacan CO, hidrocarburos (HC) y nitratos (NOx) (Carvalho et al, 2013). Por otro lado muchos estándares desarrollados en países Europeos han establecido niveles para los diferentes tipos de gases de combustión (Verma et al., 2011), los cuales deben ser cumplidos por los distintos tipos de biomasa.

En Costa Rica, así como en otros países de América Latina, los pellets han ganado popularidad ya que algunas industrias buscan la conversión en la producción de calor de fuentes fósiles a fuentes de carácter renovable, para así alcanzar el carbono neutral (Aragón *et al.* 2014). En la actualidad se conocen las adaptaciones tecnológicas al proceso de producción de pellets para muchos cultivos agrícolas tropicales (Aragón *et al.* 2014), la evaluación de algunos aspectos de la calidad del pellet (Tenorio *et al.* 2015a) y la información sobre la caracterización física, química o energética (Tenorio *et al.* 2015b). No obstante, el comportamiento de estos cultivos en algunos equipos como quemadores domésticos de pellet, su eficiencia energética y las emisiones producidas es poco conocida.

De forma tal que el siguiente trabajo tiene como objetivo conocer el comportamiento de pellets producidos con diez diferentes tipos de cultivos agrícolas y forestales de Costa Rica (Arundo donax, Cupressus lusitanica, Elaeis guineensis, Gynerium sagittatum, Pennisetum purpureun, Phyllostachys aurea, Saccharum officinarum, Sorghum bicolor y Tectona grandis), considerando el consumo de biomasa, las temperaturas que producen, las emisiones de gases, las pérdidas de calor y su eficiencia, utilizando un quemador doméstico.

2. METODOLOGIA

2.1. Pellets utilizados

Se utilizaron pellets de diez tipos de biomasa provenientes de cultivos producidos en Costa Rica (Tabla 1). Los pellets fueron fabricados por la empresa PELLETICS S.A. (http://pelletics.com/), ubicada en San Carlos en la provincia de Alajuela (Costa Rica). El detalle de las condiciones de fabricación son mostrados en Tenorio et al. (2015a, 2015b) y Aragón et al. (2015).

Table 1. Description of teen agricultural and forest crops used for the pellets manufacture.

Crops	Scientific name	Abbreviation	
Giant cane	Arundo donax	AD	
Sawlog residuals of C. lusitanica	Cupressus lusitanica	CL	
Empty fruit bunch of the oil palm	Elaeis guineensis	EFB	
Oil palm mesocarp fiber of the fruit	Elaeis guineensis	OPMF	
Wild cane	Gynerium sagittatum	GS	
King grass	Pennisetum purpureun	PP	
Golden bamboo	Phyllostachys aurea	PA	
Sugarcane	Saccharum officinarum	SO	
Sorghum	Sorghum bicolor	SB	
Sawlog residuals of <i>T. grandis</i>	Tectona grandis	TG	

2.2. Parámetros medidos en la biomasa

Carbon fraction (C), Nitrogen content (N), high heat value (HHV), ash content, moisture content of pellet (MC_{pellet}), percent volatiles and fuel value index (FVI) were determined for each type of type. Carbon (C) and nitrogen (N) were measured in three samples and determined by means of the Elementar Analysensysteme, Vario Macro Cube model. The HHV was determined in the absence of water (0% moisture content) using Parr's calorimetric test in accordance with the ASTM D-5865 04 (2003) standard. To determine the ash content, ten randomly selected 2 g pellet samples were used and the ASTM 1102-84 (2013a) standard procedure was followed. MC_{pellet} was determined using a moisture scale; model MB45, made by OHAUS (USA). For the percent volatiles, 10 pellet samples of 3 g each were used, and the ASTM D1762-84 (2013b) standard was followed. The FVI was calculated using the HHV, density, and ash content (Equation 1), based on the methodology proposed by Purohit and Nautiyal (1987).

Fuel value index =
$$\frac{\textit{High heat value}\left(\frac{\textit{kJ}}{\textit{kg}}\right)*\textit{Density}(\frac{\textit{g}}{\textit{cm}^3})}{\textit{Ash percentage}\left(\%\right)*\textit{Moisture content}\left(\%\right)} \tag{1}$$

2.3. Quemador de pellet

El quemador utilizado para evaluar la combustión de los pellet es de marca Bmax Technology model B-Half con una capacidad de 34-50 kWth, promedio de consumo energético de 60W, tensión de 230V, potencia de encendido de 400W, nivel de ruido de 40 dB y las dimensiones generales del quemador son detallados en la figura 1a-c. La capacidad corresponde a un quemador utilizado en calefacción de casa habitación y por tanto los datos presentados son válidos para quemadores similares. Los pellets son introducidos dentro del quemador por medio de un tornillo alimentador que es colocado en el recipiente con los pellets (Figure 1d). Detrás de la región de quemado se ubica un abanico que impulsa el calor y la llama a través conducto (Figura 1c) que luego será aprovechado. El material residual en la región de quemado es desplazado hacia la salida del conducto (Figure 1c).

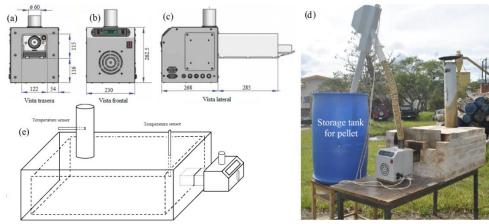


Figura 1. Esquema del sistema experimental del horno construido para la evaluación energética de los pellets.

2.4. Condiciones de uso del quemador

Fue construido un horno de ladrillo refractario de 74 cm de ancho, 73 cm de profundidad y de 30 cm de alto (Figura 1e). En una parte del extremo fue colocado el quemador de tal manera que el conducto de salida de la llama quedara centrado en la altura (Figura 1d). El recipiente con los pellets fue colocado sobre una balanza con la finalidad de establecer la masa de pellet consumido durante la prueba. Por otro lado en la parte interna del horno fue colocado un recipiente para recolectar el material residual (cenizas, escoria y el material que no fue quemado) durante la prueba. Fue colocado un sensor de temperatura a la salida de la llama (6 cm donde la

llama terminaba) y el otro sensor de temperatura fue coloca a 30 cm del inicio de la chimenea, llamadas como temperatura de llama (Tg) y temperatura de flujo de gas, respectivamente. Así mismo en este punto se colocaron los sensores para medir los gases de combustión.

Cuadro 2. Condiciones de uso del quemador de pellet Bmax Technology model B-Half.

Descripción	Unidades	Parámetros
Tiempo de operación del tornillo sin fin	Segundos	3
Potencia del ventilador	watts	300
Tiempo sinfín recarga	Segundos	3
Tiempo sinfín estabilizado	Segundos	70
Tiempo ciclo sinfín	Segundos	55
Tiempo carga inicial	Segundos	3
Tiempo sinfín arranque	Segundos	2
Tiempo sinfín limpieza	Segundos	1
Ventilador arranque	Watts	70
Ventilador carga inicial	Watts	300
Ventilador arranque	Watts	300
Ventilador limpieza	Watts	300
Ciclo carga	Segundos	70

2.5. Prueba experimental

Se realizaron tres pruebas de quemado con una duración de una hora cada una de ellas, para cada uno de los diez cultivos. Durante esa hora, la masa de pellets fue pesada en el tanque de almacenamiento de pellet (Figura 1c) antes y después de la hora de funcionamiento del quemador, así fue determinada la masa consumida por el quemador durante la prueba. Para iniciar la prueba se esperaron cinco minutos una vez que el quemador de pellet estabilizará la llama. Posteriormente, el quemador se apagó por dos horas para que todo el sistema de quemado de pellet alcanzará nuevamente la temperatura ambiente. Durante la hora de la prueba se monitoreo la temperatura de llama y temperatura de flujo de gas utilizando dos sondas tipo "J" conectadas a un data logger, marca TESTO modelo 177-T4. Se programaron los registros de ambas temperaturas cada minuto (60 mediciones en total por cada prueba de quemado).

En la determinación del material residual (contenido de cenizas, escoria y material no combustionado), el recipiente de recolección de este material se colocó al iniciar prueba de quemado y luego de finalizar se recogió y se esperó que tomará la temperatura ambiente. En esa condición fue pesado y se tomaron tres muestras para determinar la humedad que presentaba este

material utilizando la norma ASTM D-5865 04 (2003). También fueron extraídas tres muestras para determinar el porcentaje de cenizas real que contenía el material residual, utilizando la norma ASTM 1102-84 (2013a). Dichos parámetros se llamaron contenido de humedad de material residual, contenido de ceniza, escoria y material sin combustionar luego del quemado del pellet.

2.6. Medición de emisiones

En el caso de las emisiones, estás fueron determinadas utilizando un analizador de gases marca Testo, modelo 350, el cual fue colocado luego de 15 minutos de iniciado la prueba y manteniendo la medición por otros 15 minutos, durante ese periodo fue tomado 3 registros diferentes. Las emisiones que fueron determinadas fueron oxigeno (O₂), dióxido de carbono (CO₂), monóxido de carbono (CO), óxidos de nitrato (NO, NO₂ y NO_X) y dióxido de sulfuro (SO₂).

2.7. Análisis de combustión de la biomasa

En el análisis de combustión se realizó desde dos aspectos. Un primer aspecto relacionado a las características generales de la combustión de la biomasa y que contempló los siguientes parámetros: flujo de masa de pellet, el cual representa la masa consumida del pellet en el tiempo (Kg_{masa de pellet}/hours), material residual y contenido de cenizas luego del quemado del pellet, contenido de humedad de la ceniza, temperatura de la llama y temperatura de flujo de gas.

El segundo aspecto de la combustión que fue evaluado fue el análisis térmico, en el cual se evalúa las pérdidas de calor por el flujo de gas seco (Ecuación 2) y húmedo (Ecuación 3), así como las pérdidas totales de calor, para finalmente establecer la eficiencia en combustión de los pellet (Figura 4). Es importante aclarar que los datos que se obtienen en el sistema diseñado para el calor producido por los pellets no fue utilizado, si no que este fluyo directamente por la chimenea. En el caso de las pérdidas de calor, se utilizó el método ASME PTC-4 (ASME 2011), el cual es catalogado como indirecto y calcula las siguientes pérdidas:

Dry flue
$$loss \frac{=Mass_{dry} \times Cp \times (Tg-Ta)}{HHV} \times 100$$
 (2)
Wet flue $loss \frac{=Mass_{moisture} \times hg-hf \times (Hg-Tg)}{HHV} \times 100$ (3)

Wet flue
$$loss = \frac{-Mass_{moisture} x hg - hf x (Hg - Tg)}{\mu \mu \nu} x 100$$
 (3)

$$Efficiency = 100 - (dry flue loss + wet flue loss)$$
 (4)

Donde:

Mass dry: is the mass of dry flue gas in kg/kg of fuel, el cual es determinado por la suma de los gases en la masa de los pellet. Los gases presentes en la chimenea los cuales corresponde a O₂, CO, CO₂ y N₂ medidos durante las emisiones.

Moisture: is the sum of water vapor produced from hydrogen in fuel, moisture present in fuel and moisture present in air in kg/kg of fuel

Cp: is specific heat of flue gas, el cual corresponde a la capacidad calórica del aire, en kJ kg⁻¹ C⁻¹.

Tg is flue gas temperature in °C,

Ta is ambient air temperature in °C

hg is enthalpy of steam at the temperature in kJ kg⁻¹

hf is enthalpy of water at ambient temperature in kCal/kg

HHV is higher heating value in kJ/kg.

2.8. Statistical analysis:

A descriptive analysis was performed (median, standard deviation, and maximum and minimum values) for the following variables: HHV, FVI, percent volatiles, MC of pellet, C and N content, mass pellet flue, residual mass, ash content, MC of pellet, temperature of flame, temperature of flue gas, emissions parameter (O₂, CO₂, CO, NO, NO₂, NOx and SO₂), heat loss (dry and wet) and efficiency of combustion of pellet. In addition, it was determined whether variables complied with the premises of normal distribution, homogeneity of variances, as well as the presence of extreme values. A variance analysis was applied to verify the existence of significant differences among the averages of the variables (P<0.05). Tukey's test was carried out to determine the statistical differences among crops, for the mean value of each of the abovementioned values.

3. RESULTADOS

3.1. Caracterización de la biomasa

Table 3 shows biomass characterization of ten crops. The HHV varied from 12.15 MJ/kg to 19.92 MJ/kg, and four groupings were created based on the statistical differences of HHV: (i) PA and GS, with the highest values, (ii) followed by SB, CL, AD, PP and, OPMF, (iii) then by TG and EFB, and lastly (iv) the group with the lowest values, composed of SO. The FVI test

determined that CL had the highest FVI, followed by PA; the remaining crops (PLP, EFB, OPMF, CA, GS, PP, SO, SB, and TG) had statistically similar FVI values, while AD had the lowest value. The percent volatiles for the ten crops varied between 69.2% and 78.0%, and the crops were grouped into four categories: (i) SO and CL, presenting the highest values, (ii) followed by TG, PA, and GS, (iii) SB, OPMF, and EFB, and (iv) one last group, formed by AD, CA, and PP, which represent the lowest ash percentage values.

MC_{pellet} for the ten crops varied from 6.7% to 12.6%, and once more four groups were established: (i) crops with the highest MC were SB, PP, TG, and AD, (ii) followed by CA, (iii) then by SO, OPMF, and EFB, and lastly (iv) CL and PA, with the lowest values. En la evaluación del C nuevamente cuatro grupos fueron formados: un primer grupo formado por CL and TG, seguido de EFB y PA, luego un grupo con la mayoría de los cultivos (OPMF, GS, PP, SO y SB) y un último grupo formado solamente por AD. El N fue estadísticamente el más alto en OPMF, seguido de SB, luego un tercer grupo compuesto de EFB y PP. El cuarto grupo fue formado por AD, luego por GS, OS y TG y finalmente con el contenido de N más bajo fue para CL y PA (Table 3).

Table 3. Pellet characterization of ten pellets crops from Costa Rica

Crops	Higher heating value (MJ/kg)	Fuel value index	Volatile content (%)	Moisture content of pellet (%)	Carbon (% w/w)	Nitrogen content (% w/w)
Arundo donax-AD	15.93 ^{BC}	163 ^D	70.5^{DE}	12.0 ^A	40.87^{D}	0.57 ^D
Cupressus lusitanica-CL	16.81 ^B	2803 ^A	76.7^{AB}	7.5^{E}	46.40^{A}	0.26^{F}
Elaeis guineensis-EFB	14.18^{D}	349 ^{CD}	71.7^{CD}	9.0^{D}	44.49^{B}	0.68^{C}
Elaeis guineensis-OPMF	15.83 ^{BC}	340^{CD}	72.4 ^C	9.2 ^{CD}	42.79 ^C	1.69 ^A
Gynerium sagittatum-GS	18.75 ^A	491 ^C	75.1^{B}	9.7 ^{CD}	43.70°	0.29^{E}
Pennisetum purpureun-PP	16.98^{B}	244 ^{CD}	69.2^{E}	12.1 ^A	41.72 ^C	0.67 ^C
Phyllostachys aurea-PA	19.92 ^A	1039^{B}	75.3^{B}	6.7^{E}	44.27^{B}	0.24^{F}
Saccharum officinarum-SO	12.15^{E}	212^{CD}	78.0^{A}	9.7^{CD}	43.60°	0.36^{E}
Sorghum bicolor-SB	16.91 ^B	273^{CD}	72.6 ^C	12.6 ^A	42.84°	0.84^{B}
Tectona grandis-TG	15.26 ^{CD}	463 ^{CD}	75.9^{B}	12.1 ^A	45.19^{AB}	0.28^{E}

Legend: Different letters for each parameter represent statistical differences between crops (significances at 95%).

3.2. Emisiones de gases

En el caso de emisiones de gases (Tabla 4), se encontró que nuevamente hay una alta variación entre los cultivos. En el caso de O₂ este varío de 12.3 a 19.7%, siendo los valores más

altos en las especies SO, PP, OPMF y los valores más bajos en GS. En el gas CO₂, la variación fue de 8.1% a 2.3%, siendo GS el de mayor valor y SO el de menor valor. En la emisión de CO, la madera CL presentó el valor más bajo y SB el valor más alto. En tanto que OPMF, GS y SB presentaron emisiones sobre 150 ppm en NO, pero las emisiones de NO₂ y SO₂ fueron casi nulas en todos los cultivos. En las emisiones de NO_X, las especies EFB, OPMF, GS y PS presentaron emisiones sobre 120 ppm (Tabla 4).

Table 4. Emission of ten pellets crops from Costa Rica in a stove.

Crons	Oxigeno	CO_2	CO	NO	NO_2	NOx	SO_2
Crops	(%)	(%)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
Arundo donax-AD	15.1 ^C	6.0^{B}	888 ^E	84.0^{D}	0.0^{A}	84.0 ^C	0.0^{A}
Cupressus lusitanica-CL	16.4^{B}	4.4 ^C	190^{G}	55.3^{D}	0.0^{A}	55.7^{D}	2.7^{B}
Elaeis guineensis-EFB	17.0^{AB}	$4.0^{\rm C}$	622^{E}	142.0^{B}	0.3^{A}	142.3^{B}	0.0^{A}
Elaeis guineensis-OPMF	18.2^{A}	5.2^{BC}	1441 ^D	175.5 ^A	1.5 ^A	175.5^{B}	0.0^{A}
Gynerium sagittatum-GS	12.3 ^D	8.1 ^A	1702 ^C	155.0^{AB}	0.7^{A}	182.7 ^A	0.0^{A}
Pennisetum purpureun-PP	19.7 ^A	3.5 ^C	3268^{B}	121.3 ^C	0.0^{A}	121.3^{B}	0.0^{A}
Phyllostachys aurea-PA	16.7^{AB}	3.8°	205^{F}	69.0^{D}	0.0^{A}	72.3 ^C	1.0^{B}
Saccharum officinarum-SO	18.2 ^A	2.3^{D}	1312 ^D	52.3 ^D	0.0^{A}	52.3^{D}	0.0^{A}
Sorghum bicolor-SB	17.5^{AB}	3.0^{CD}	4287^{A}	155.0^{AB}	0.0^{A}	156.0^{AB}	0.0^{A}
Tectona grandis-TG	17.6^{AB}	3.3 ^C	2685^{B}	59.0^{D}	0.0^{A}	59.0^{D}	0.0^{A}

Legend: Different letters for each parameter represent statistical differences between crops (significances at 95%).

3.3. Consumo de pellet, características de las cenizas y temperaturas

En la evaluación de consumo de la masa de pellet se encontró que este varío de 2.5 a 3.4 kg/hora. Los pellets de CL, EFB y OPMF son los de mayor consumo en el quemador, seguido por los pellets de AD, GS, SO y SB, luego el grupo con los consumos más bajos, compuesto de PP, PA y TG (Tabla 5). En lo referente a la masa residual, la variación fue de 0.36% a 6.26%. El valor más alto corresponde al cultivo PP y el más bajo a SB y CL, como segundo grupo con los valores más altos se presentaron los cultivos AD, EFB, OPMF, GS, PA, SO y TG (Tabla 5).

En la masa residual se encontró que entre 46 y 80% corresponde a cenizas. La masa residual con mayor porcentaje de cenizas fueron AD, CL y SB, luego un grupo formado por EFB, OPMF, PP, PA y TG, y finalmente SO y GS son los cultivos con los porcentajes más bajos en cenizas en la masa residual. El contenido de humedad de la masa residual varío de 3.33% a 8.17%, con la especie de PP con el mayor valor y AD con la humedad más baja (Tabla 5).

Otro aspecto importante en la masa residual (ceniza y escoria) es su aspecto físico, Este fue diferente en cada tipo de cultivo (Figura 2), no obstante tres grandes grupos pueden observarse considerando el tamaño de las partículas: una masa residual compuesta de material granulado fino, como en el caso de CL y TG (Figura 2b y 2j), luego un tipo de material residual de tipo granulado de tamaño de las partículas más grandes que las anteriores especies y formado por AD, PA y SO (Figura 2a, 2g y 2h). Finalmente en EFB, OPMF, GS, PP y SB, el material residual presenta material granulado de tamaño grande y generalmente forma cúmulos, en la cual se presentan biomasa no combustionada.



Figura 2. Aspecto físico del material residual luego de una hora de quemado de pellets.

En relación con las temperaturas se encontró que en promedio, CL y OPMF son los cultivos con la mayor temperatura tanto en la salida como en el flujo de aire en la chimenea, luego AD y EFB también presentan altas temperaturas en estos dos puntos de medición. Seguidamente GS, SO, SB y TG con valores intermedios de temperaturas, mientras que los cultivos con las temperaturas más bajas fueron PP y PA (Tabla 5)

Tabla 5. Consumo de pellet, características de las cenizas y temperas en las pruebas de quemado de pellet producido de 10 cultivos agrícolas forestales en Costa Rica.

	Mass pellet	Residual	Ash content	Moisture	Temperature	Temperature
Crops	flue	mass	of residual	content of	of flame	in flue gas
<u>-</u>	(kg/hours)	(%)	mass (%)	ash (%)	stove (°C)	(°C)
Arundo donax-AD	2.8^{BC}	4.05^{B}	80 ^A	3.33^{H}	518 ^B	270^{B}
Cupressus lusitanica-CL	3.1^{AB}	1.78^{D}	71 ^A	5.30^{E}	590 ^A	310^{A}
Elaeis guineensis-EFB	3.1^{AB}	4.06^{B}	67^{B}	3.76^{G}	544^{B}	284^{B}
Elaeis guineensis-OPMF	3.4^{A}	3.71^{BC}	68^{B}	3.44^{H}	613 ^A	322^{A}
Gynerium sagittatum-GS	2.9^{ABC}	3.76^{BC}	44 ^C	4.16^{F}	473 ^C	232^{CD}
Pennisetum purpureun-PP	2.5^{CDE}	6.26^{A}	67^{B}	8.17^{A}	396 ^F	203^{E}
Phyllostachys aurea-PA	2.7^{DE}	2.21^{BC}	64^{B}	5.26^{E}	401^{EF}	205^{E}
Saccharum officinarum-SO	2.7^{BC}	2.98^{BC}	46 ^C	5.63^{D}	449 ^{CD}	245 ^C

Sorghum bicolor-SB	2.9^{ABC}	0.36^{E}	78 ^A	7.91^{B}	436^{D}	225^{D}
Tectona grandis-TG	2.1^{E}	2.27^{BC}	63^{B}	6.84 ^C	431 ^{DEF}	222^{D}

Note: values between parenthesis means the variation coefficient and different letters for each parameter means statistical significances at 95%. La masa residual corresponde a cenizas, escoria y biomasa no combustionada.

La variación de la temperatura, medido por su coeficiente de variación, en los dos puntos de medición, a la salida de la llama y el flujo en la chimenea, fue mayor en a la salida del quemador en relación a la temperatura en el flujo de aire caliente de la chimenea (Figura 3a y 3b). En el caso de la temperatura en la chimenea la mayor variación se presentó en TG, seguido de PP y SB, luego se presentó un grupo intermedio formado por SO y GS. Las especies con menor variación se presentaron en CL, OPMF y PA (Figura 3a). En el caso de flujo de aire en la chimenea, el comportamiento fue similar que la temperatura a la salida de la llama, solo que PP presenta mayor variación en el flujo de aire en la chimenea (Figura 3b).

La Figura 3c presenta el comportamiento de la temperatura en la salida de la llama en el quemador y la temperatura en el flujo de aire para TG y CL, correspondiente a la más alta y baja variación, respectivamente. En el caso de TG, se observa una mayor irregularidad de la temperatura durante una hora (tiempo de duración de la prueba) en los dos puntos de medición, pero en el caso de CL la irregularidad es menor que TG en los dos puntos de medición (Figura 3c).

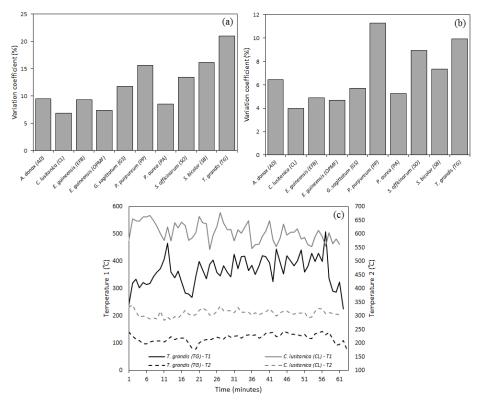


Figura 3. Coeficiente de variación de las temperaturas de la llama del quemador y temperatura de flujo de gases y ejemplo de variación presentada en una corrida de *C. lusitanica* y *T. grandis*

3.4. Eficiencia y pérdidas

La evaluación de las pérdidas de calor se encontró que varío de 26 a 69% (Figura 4a), siendo la especie GS las de menor pérdida. Las especies SO, EFB, CL, PP, SO y TG con pérdidas sobre 50% y el resto de las especies con valores intermedio a los anteriores grupos (Figura 4a). En todos los cultivos, las dry flue loss fueron menores a los wet flue loss (Figura 4a). En el caso de wet flue loss las pérdidas más altas se presentaron en EFB y SO, luego se forma un segundo grupo formado por CL, OPM, FSB y TG, en tanto que las especies AD, PP y PA forma un grupo de valores bajos, junto GS (Tabla 4). En tanto que las mayores pérdidas por flujo de gas húmedo se presentaron en OPMF y PP y las menores pérdidas se presentaron en GS, PA, SO y SB (Figura 4a). Consecuentemente la eficiencia fue mayor en GS y PA, seguido por AD, PP y SB. La menor eficiencia se presentó en EFB y SO y el resto de las especies presentan eficiencias intermedias (Figura 4b).

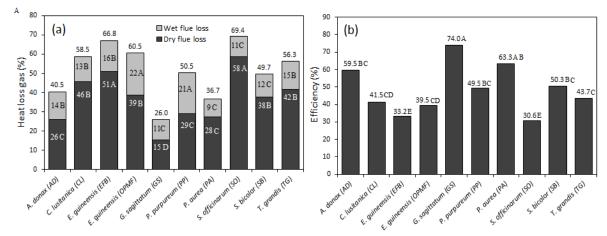


Figure 4. Heat losses dry and wet (a) and efficiency (b) for of ten pellets crops from Costa Rica

3.5.Relación entre los parámetros de combustión con las emisiones y parámetros energéticos

Los coeficientes de correlación entre los diferentes tipos parámetros medidos (Tabla 6) mostró que los dos tipos de temperaturas (de la llama a la salida del quemador y en el flujo de aire en la chimenea) presentaron los mismos coeficientes de correlación. Estas temperaturas fueron negativamente correlacionadas (α<0.01) con las emisiones de CO y el MC_{pellet} y positivamente correlacionadas (α<0.05) con HHV, SO₂, y FVI. En el caso del flujo de masa, este es positivamente correlacionado con N y los nitratos (NO, NO₂ y NO_X) y negativamente correlacionado con MC_{pellet}. La masa residual fue negativamente correlacionada con N₂ y C (Tabla 6). El flujo de calor seco fue negativamente correlacionado con CO₂, MC del pellet y HHV, en tanto que el O₂ y C afectaron positivamente con el flujo de calor seco. En el caso del flujo de calor húmedo fue negativamente correlacionado con contenido de volátiles y HHV, y positivamente correlacionado con las emisiones de CO₂ y HHV y negativamente solo por O₂ (Tabla 6).

Tabla 6. Matriz de correlación entre los parámetros mediciones durante la combustión con las emisiones, y parámetros energéticos de pellets de 10 tipos de cultivos en Costa Rica.

Parameter	Mass pellet flue	Residual mass	Temperature of flame stove	Temperature in flue gas	Heat loss dry gas	Heat loss wet gas	Efficiency
O_2	-0.19 ^{ns}	0.11 ^{ns}	-0.11 ^{ns}	-0.11 ^{ns}	0.57**	0.54**	-0.70**
CO_2	0.33^{ns}	0.22^{ns}	0.22^{ns}	0.22^{ns}	-0.72**	-0.04^{ns}	0.68**
CO	-0.23 ^{ns}	-0.05^{ns}	-0.48**	-0.48**	-0.12^{ns}	0.25^{ns}	$0.03^{\rm ns}$
NO	0.46*	0.02^{ns}	$0.07^{\rm ns}$	0.08^{ns}	-0.25 ^{ns}	0.13^{ns}	0.19^{ns}
NO_2	0.40*	0.07^{ns}	$0.27^{\rm ns}$	0.27^{ns}	-0.14 ^{ns}	0.13 ^{ns}	0.09^{ns}
NO_X	0.44*	0.03^{ns}	$0.05^{\rm ns}$	$0.05^{\rm ns}$	-0.32^{ns}	0.08^{ns}	$0.30^{\rm ns}$
SO_2	$0.17^{\rm ns}$	-0.33ns	0.36*	0.36*	0.09^{ns}	-0.19 ^{ns}	-0.02^{ns}
N	0.53**	0.20^{ns}	0.40*	0.40*	0.06^{ns}	0.62**	-0.25 ns
C	-0.02^{ns}	-0.46**	$0.23^{\rm ns}$	0.23^{ns}	0.42*	-0.30ns	-0.30ns
MC of pellet	-0.49**	-0.07^{ns}	-0.62**	-0.62**	-0.37*	0.08^{ns}	0.33^{ns}
FVI	$0.20^{\rm ns}$	-0.31 ^{ns}	0.42*	0.42*	0.11^{ns}	-0.22 ^{ns}	-0.03 ^{ns}
Volatiles content	$0.14^{\rm ns}$	-0.41 ^{ns}	0.18^{ns}	0.18 ^{ns}	0.28^{ns}	-0.62**	-0.07^{ns}
HHV	-0.32^{ns}	-0.15^{ns}	0.49**	0.49**	-0.51**	-0.49**	0.63**

Legend: MC: moisture content, FVI: Fuel value index; HHV: Higher heating value.

4. DISCUSIÓN

4.1. Caracterización de la biomasa

Los valores obtenidos de HHV en los cultivos evaluados varían de 12,15 MJ/kg a 19,92 MJ/kg (Tabla 2), rango que en las especies CL, GS, PP, PA y SB concuerda con las especies maderables de rápido crecimiento en Costa Rica, los cuales varían de 16,50 kJ/kg a 20,60 MJ/kg (Moya and Tenorio, 2013). Pero AD, TG, EFB, OPMF, SO y TG mostraron valores de HHV inferiores a los presentados para las especies maderables. Los valores del FVI para los diez cultivos variaron de 163 a 2803 (Tabla 2) y comparando estos valores con los reportados por Moya and Tenorio (2013) para especies maderables en Costa Rica se tiene que AD, PP y SO, poseen FVI menores al rango reportado para la maderas (de 337 a 6390). La variación del porcentaje de volátiles en este estudio fue de 69.24% a 77.96% (Tabla 2), valores más altos que los presentados por Kataki and Konwer (2002) para especies maderables del noreste de India, los cuales reportaron valores 13.53% a 40.08%. En relación con los valores de MC_{pellet}, se señalan varias opiniones acerca del MC_{pellet} que deben presentar los pellets para un buen desempeño, normalmente los pellets contienen un MC de entre 8% y 12% (Lehtikangas 2001; Kaliyan and Morey 2009). Para el MC_{pellet} de los pellets fabricados en este estudio se encuentra que solamente

CL y PA poseen MC_{pellet} inferiores a 8% (Tabla 3). Los valores de contenido de C, de 40.87 a 46.20%, y nitrógeno (de 0.26 a 1.69%) son comunes en las diferentes tipos de biomasa (Abuelnuor et al., 2014).

4.2.Emisiones

Las emisiones de CO₂, como es de esperar, es su baja cantidad de emisiones, menores a 8.1%, y que es una ventaja de la biomasa. Sin embargo en AD, OPMF y GS, las emisiones de CO₂ deben ser manejadas más cuidadosamente ya que se presentan valores entre 5-8%, altas para este tipo de material. En el caso de las emisiones de O₂ encontradas fueron en promedio de 17% (Tabla 4), valor considerado alto ya que este ser aproximadamente 10% (Garcia-Maraver et al., 2014). Lo cual indica que es necesario ajustar el diseño fabricado con la finalidad de disminuir las emisiones de O₂.

En tanto que en las emisiones de CO, los datos obtenidos para las diferentes especies son muy variables (Tabla 4). Algunos autores (Garcia-Maraver et al., 2014, Verma et al., 2012, Rabaçal et al., 2013) reportan valores inferiores a 2000 ppm en 17% de O₂, valores que presentan AD, CL, EFB, OPMF, GS, PA y SO, por lo que estos cultivos se encuentran dentro del rango normal. Pero PP, SB y TG, presentan valores sobre 2680 ppm, considerados como altos. Los altos valores en las emisiones de CO son producto de malos diseños de la cámara donde se realizada la combustión (tiempo que permanecen los gases en la cámara, temperatura de la cámara, turbulencia/mezcla, exceso de aire, entre otros) o bien por las características propias del pellet, como la especie o humedad del pellet (Maraver et al., 2014).

En el caso de NO and NOx, los valores encontrados son también muy variables entre especies, siendo nuevamente estos comparables con los estudios realizados por Garcia-Maraver et al. (2014), Verma et al. (2012) y Rabaçal et al. (2013). Así mismo Limousy et al. (2013) cataloga como bajos valores de emisiones inferiores a 100 ppm, por lo que en el caso de los cultivos AD, CL, PA, SO y TG estos son catalogados de bajas emisiones para este tipo de gases. Las altas emisiones de NO y NOx, como los presentados en EFB, OPMF, GS, PP, SB y TG, son atribuidas al contenido de nitrógeno en la biomasa (Limousy et al., 2013). En el caso de las biomasa analizada se presenta ligeramente esta tendencia, ya que los cultivos con bajos contenidos de N, entre 0.24 a 0.36% (Tabla 3), son los cultivos de bajas emisiones de NO y NOx

(Tabla 4). Sin embargo, AD es la única excepción, ya que presenta un alto contenido de N pero bajas emisiones de los anteriores gases.

4.3. Consumo de pellet, características de las cenizas y temperaturas

En general se observó que el flujo de masa del pellet es en promedio 3 kg/hora, pero con una variación de 2.5 a 3.4 kg/hora (Tabla 5). La variación del consumo del pellet fue altamente significante por el MC_{pellet}, el contenido de N, NO, NO₂ y NOx (Tabla 6), concordando dichos resultados con lo reportado por Dai y Grace (2011) y Obernberger y Thek (2004). Sin embargo, la variación en el consumo es afectada por otras características propias de los pellets. Aunque en este trabajo no se reportaron las características físicas y químicas de los pellets, en trabajos previos se reportaron dichas propiedades (Tenorio et al., 2015b; Aragón et al. 2015) y se demostró una variación en el largo, densidad (del pellet y densidad aparente) y en las dimensiones de las partículas que conforman el pellet. Carvalho et al. (2013) indica que estas propiedades de los pellets afectan la alimentación de los pellets en el sistema de combustión cuando se utiliza un tornillo para alimentar.

Durante el proceso de combustión de los pellets es posible observar que la masa residual es muy irregular, ya que presenta una variación muy alta, de 0.36 a 6.26%. Esta masa está compuesta por ceniza, escoria y material no combustionado (Lindström et al., 2010). Un alto porcentaje de material residual produce problemas en el sistema de alimentación de los quemadores de pellets ya que se tiende a almacenar en la salida (Öhman et al., 2004) y contribuye a aumentar las emisiones de nitratos (Carvalho et al., 2013). Al analizar los diferentes tipos de biomasa, en el caso de los cultivos maderables (CL y TG) con baja masa residual (Tabla 5) no se observó la formación de escoria (Figura 2b y 2j). El material era muy fino, lo que indica que no hubo formación de cúmulos de escoria, pero probablemente se presentó una falta de combustión, ya que del material residual la cantidad de ceniza varía de 63 a 71%, dejando entre 37 y 29% en material no combustionado. El grupo cuyo material residual fue tipo granulado de tamaño medio, formado por AD, PA y SO (Figura 2a, 2g y 2h) o de tamaño grande, compuesto por EFB, OPMF, GS, PP y SB, no se observa ninguna concordancia con el porcentaje de material residual y la cantidad de ceniza. Por ejemplo en el grupo de material residual de tamaño medio, AD presentó alto el porcentaje de residual y una alta cantidad de cenizas, pero los otros dos cultivos tienen bajo masa residual y bajo cantidad de cenizas (Tabla 5). En tanto que cuando el material

granulado es grande, la especie SB presentó bajo material granulado, pero con bajo contenido de cenizas. GS presentó un alto porcentaje de material residual, pero un bajo contenido de cenizas (Tabla 5).

Por otro lado se encontró que el material residual fue altamente correlacionado con N₂ y contenido de C de la biomasa, sin embargo esto debe ser tomado con mucho cuidado ya que en el caso de la escoria hay muchos factores que intervienen en su formación, tales como el contenido de óxidos (de Fe, Ca, Mg, Na, Si, Ti y Al), el sistema de alimentación durante el quemado del pellet, o bien el manipuleo de la materia prima antes de la formación del pellet (Rabaçal et al., 2013, James et al., 2012).

En el caso de las temperaturas, a la salida del quemador o en la chimenea, nuevamente se observa una variación entre los diferentes cultivos utilizados en la fabricación de los pellets (Tabla 5). En este caso los pellets fabricados con GS y CL estos presentan una mayor generación de temperatura. Esto presenta la ventaja que en un posible aprovechamiento se alcanzarían las temperaturas deseadas en menor tiempo. Además en CL se sumaría a esta ventaja, una temperatura más estable que en el resto de cultivos (Figura 3a y 3b). Otros cultivos como en el caso de PA o TG, con una menor generación de calor (Tabla 5) y una mayor variación de temperatura, respectivamente, (Figura 3c) se presenta como una desventaja. En estas condiciones en caso de querer aprovechar el calor generado por los pellets no sería posible alcanzar las temperaturas deseadas o estables.

Estas temperaturas fueron correlacionadas con HHV, MC_{pellet}, FVI, cantidad de N y las emisiones de CO y SO₂ (Tabla 6). Como era de esperar el MC_{pellet} y HHV afectaron negativamente y positivamente, respectivamente, las temperaturas de combustión. En el caso de CO es generalmente considerado un buen indicador de la calidad de combustión, ya que se oxida a CO₂ en presencia de oxígeno, el cual depende la temperatura de combustión, tiempo de residencia y rango de mezcla entre los gases de combustión y el aire (Carvalho et al. 2013) y cuando disminuye la emisión de CO se tiene una mayor temperatura, como sucedió en el presente trabajo (Tabla 6). Pero las relaciones con SO₂ y N son poco explicadas, sin embargo Hedman et al, (2006) afirma que estas sustancias pueden formar auxinas y furanos bajo ciertas condiciones dando como resultado una irregularidad de la combustión y por tanto en las temperaturas.

4.4.Pérdidas y eficiencia

Las pérdidas de calor por aire seco, como era de esperar, son más altas que las pérdidas de calor por aire húmedo (Figura 4a), ya que los pellets fabricados presentan baja humedad (10-12%). Dicho comportamiento coincide con los resultados reportados por Roy et al. (2011). Sin embargo, una diferencia entre las pérdidas totales del presente estudio con las presentadas por Roy et al. (2011) es que las del presente estudio con más altas. En el presente estudio las pérdidas variaron de 26 a 69% (Figura 4a), pero Roy et al. (2011) reporta 37% para pellets de madera. Las altas diferencias en las pérdidas de calor seco son atribuidas a que durante las pruebas de combustión el aire caliente no fue utilizado en ningún tipo de trabajo, por lo que gran parte de este aire va a fluir por la chimenea, y por tanto se obtienen pocas diferencias entre los dos puntos de medición que son referencia para calcular las pérdidas de calor por aire seco (Ecuación 2).

Las diferencias en las pérdidas de calor en aire seco y aire húmedo en la chimenea son atribuidas a factores atribuidos al proceso de combustión mismo y a las características del pellet. En el caso del aire seco las variaciones fueron atribuidas a las emisiones de O₂ y CO₂ durante la combustión (factor relacionado al proceso de combustión) y el MC y el HHV del pellet, como factores relacionados a las características del pellet. En el caso las emisiones de CO₂, el MC y el HHV presentan una relación negativa con las pérdidas de calor en aire seco (Tabla 6), indicando que el aumento de las emisiones de CO₂, MC y HHV disminuye las pérdidas de calor del aire seco. Pero en el caso de O₂ presenta una relación positiva (Tabla 6), por tanto el incremento de las emisiones de O₂ aumenta las pérdidas de aire seco en la chimenea.

En el caso del flujo de aire húmedo en la chimenea, la cantidad de O₂ (factor relacionado al proceso de combustión) y la cantidad de N presentes en la biomasa (característica de pellet) aumentan las pérdidas por aire húmedo ya que son positivamente correlacionados (Tabla 6). En tanto que la cantidad de volátiles y HHV (factores relacionado al pellet) afectan negativamente las pérdidas de calor (Tabla 6), por lo que un aumento en la cantidad de volátiles o HHV disminuye las pérdidas por aire húmedo.

Finalmente la combustión de los diferentes tipos de pellets es derivada de las pérdidas de aire seco y aire húmedo (Ecuación 4) en la chimenea, y como se explicó anteriormente no se presentó aprovechamiento el calor generado durante el experimento por lo que las pérdidas fueron muy altas, por lo tanto se presentan bajos valores en la eficiencia, entre 30% y 75% (Figura 4b). No obstante al considerar estos valores relativos las especies más eficientes son GS y PA,

seguido por AD, PP y SB, los de menor eficiencia son EFB y SO y el resto de las especies presentan eficiencias intermedias (Figura 4b).

Estas diferencias en los valores de la eficiencia son explicadas por las diferentes características intrínsecas de los pellets y su diferente comportamiento durante la combustión. Específicamente la matriz de correlación mostró que la eficiencia es afectada positivamente por las emisiones de CO y el HHV (Tabla 6), pero negativamente por las emisiones de O₂. Un indicador de la buena combustión son las emisiones de CO y HHV (Carvalho et al. 2013), concordante en la relación positiva con la eficiencia en este estudio (Tabla 6). Pero un bajo contenido de O₂ indica que el proceso de combustión se está realizando adecuadamente (Abuelnuor et al., 2014) y por tanto hay un incremento de la eficiencia.

5. CONCLUSIONES

Las emisiones de gases durante la combustión presentaron una alta variación entre los cultivos: el O₂ varío de 12.3 a 19.7%, el CO₂ de 8.1% a 2.3%, el CO de 190 a 4287 ppm, las emisiones de NO fueron de 84 a 175 ppm, NO_X de 55 a 182 ppm y las emisiones de NO₂ y SO₂ fueron casi nulas en los diferentes tipos de pellet. De estos valores se notó que en el caso de las emisiones de O₂ se deben mejorar para hacer más eficiente el proceso de combustión en el quemador de pellets utilizado. En tanto que las emisiones de CO, CO₂ y nitratos, para algunos cultivos como AD PP, SB, AD y TG se debe buscar la forma de disminuir estas emisiones.

El flujo de masa de pellet varío de 2.5 a 3.4 kg/hora y fue afectada por el MC_{pellet} y el contenido de N, pero también fue afectado significativamente por las emisiones de NO, NO₂ y NOx. En la masa residual, la variación fue de 0.36% a 6.26% y está compuesta por ceniza (entre 46 y 80%), escoria y material sin combustionar. Los pellets fabricados con cultivos forestales son los que dejan menor masa residual, mientras que los de mayor cantidad de escoria fueron GS y SO. La masa residual fue correlacionada con el contenido de N y C. En las temperatura evaluadas, a la salida del quemador o en la chimenea, nuevamente se observa una variación entre los diferentes tipos de pellet, siendo GS y CL los cultivos con mayor generación de temperatura y PA o TG los que poseen una generación de calor menor. Estas temperaturas fueron altamente significativas con el HHV, MC_{pellet}, FVI, cantidad de N y las emisiones de CO y SO₂.

Las pérdidas de calor por aire seco son más altas que las pérdidas de calor por aire húmedo. Las pérdidas de calor en aire seco fueron atribuidas a las emisiones de O₂ y CO₂ durante

la combustión (factor relacionado al proceso de combustión) y el MC y el HHV del pellet (factores relacionados a las características del pellet). En tanto que el aire húmedo fue positivamente correlacionado con contenido de volátiles y HHV, y positivamente correlacionado con O₂ y N. Finalmente la eficiencia varío de 30% y 75%, siendo los valores más eficientes en GS y PA, seguido por AD, CL, PP y SB, los de menor eficiencia son EFB y SO y el resto de las especies presentan eficiencias intermedias. Estas diferencias en los valores de la eficiencia entre cultivos fueron afectadas positivamente por las emisiones de CO₂ y HHV y negativamente por O₂.

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5. Artículo 5. Use of coffee (Coffea arabica) pulp for the production of briquettes and pellets for heat generation.

USE OF COFFEE (Coffea arabica) PULP FOR THE PRODUCTION OF BRIQUETTES AND PELLETS FOR HEAT GENERATION

Uso de pulpa de café (Coffea arabica) para a fabricação de briquetes e pellets para geração de calor

Robert Cubero-Abarca¹, Roger Moya², Jorre Valaret³, Mario Tomazello Filho

ABSTRACT

Coffee bean (Coffee arabica) processing generates high amount of residues that are sources of environmental pollution. Therefore, an appropriate solution is needed. The objective of this study was to determine the potential of coffee pulp to produce briquettes and pellets. The study included pulp drying (using air, solar and hot air methods); the production of briquettes and pellets; the evaluation of their energy, physical and mechanical properties; and the evaluation of pellet quality using X-ray densitometry. The results showed that the pulp presented an initial moisture content of 90%, resulting in drying times of 699, 308 and 55 hours for air, solar and hot air drying, respectively, and the calorific values of the pellets and briquettes were 12,501 kJ kg and 11,591 kJ kg1, respectively. The ash content was 8.68% for the briquettes and 6.74% for the pellets. The density of the briquettes was 1,110 kg m³, compared with 1,300 kg m³ for the pellets. The apparent densities were 1,000 kg m³ and 600 kg m³ for the briquettes and pellets, respectively, and the water absorptions by the briquettes were 7.90% and 8.10% by the pellets. The maximum horizontal compression effort was 26.86 kg cm⁻², measured in the pellets, compared with 4.52 kg cm⁻² in the briquettes. The maximum horizontal load was 93.24 kg, measured in the briquettes, compared with 33.50 kg in the pellets. The value of the pellet durability test was 75.54%. X-ray densitometry showed that the pellet was uniform and a few cracks were observed on the pellet surface.

Index terms: Coffee residues, drying, coffee processing, biomass, bio-resources.

RESUMO

No processamento dos grãos de café (Coffea arabica) ocorre o problema de geração de grandes quantidades de resíduos não utilizados pela indústria, tornando-se uma fonte de poluição ambiental. Portanto, é necessária a busca de soluções para atender a este problema de geração de residuos. O objetivo do trabalho foi determinar a possibilidade de utilização da polpa dos grãos de café processados para a fabricação de briquetes e pellets. A pesquisa compreendeu a secagem da polpa dos grãos de café (pelo meio do ar, solar e ar aquecido); a manufatura de briquetes e de pellets; a avaliação das suas propriedades como fonte de energia e propriedades físicas e mecânicas e a determinação da sua qualidade pela metodologia de densitometria de raios X. Os resultados mostraram que a umidade inicial da polpa de café foi de 90% e o tempo de secagem foi de 699, 308 e 55 horas no ar, secagem solar e por ar quente, respectivamente. O poder calórico foi de 12 501 kJ kg⁻¹ e de 11 591 kJ kg⁻² de pellets e de briquetes, respectivamente; o teor de cinzas foi de 8,68% e de 6,74% para os briquetes e os pellets, respectivamente. Em relação às propriedades físicas, a densidade dos briquetes foi de 1,110 kg m⁻³ e dos pellets de 1,300 kg m⁻³ a densidade aparente dos briquetes foi 1,000 kg m⁻³ e de 600 kg m⁻³ dos pellets e a absorção de água de 7,90% dos briquetes e 8,10% e dos pellets. Finalmente, o esforço de compressão horizontal máxima foi de 4,52 kg cm² para os briquetes e de 26,86 kg cm² para os pellets, a carga máxima horizontal foi 93,24 kg e de 33,50 kg para os briquetes e os pellets e de 75,54% para o teste de durabilidade dos pellets. A avaliação da qualidade dos pellets por densitometria de raios X mostrou que a sua homogeneidade, observando-se algumas fissuras ou fendas em sua superficie.

Termos para indexação: Residuos de café, secagem, processamento de café, biomassa, bio-recursos.

INTRODUCTION

Currently, more than 98 000 ha are dedicated to coffee production in Costa Rica (Alfaro; Rodriguez, 1994). Only 20% of the volume of ripe coffee cherries, i.e., the seed or bean, is used. As a result, 80% of the fruit becomes waste during processing (Alfaro; Rodriguez, 1994). Coffee residues comprise the husk, pulp and wastewater, all of Tenorio; Moya, 2012). Therefore, the development of

which pollute the environment (Alfaro; Rodriguez, 1994). Approximately 11,764 tons of coffee are produced per day during harvesting, which are associated with ecological damage (Alfaro; Rodriguez, 1994). In addition, the use of residues in Costa Rica and other developing countries is limited because of a lack of technology and inadequate knowledge of agricultural waste (Ulloa et al., 2004;

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an innovative alternative for biomass waste is a major challenge for developing countries that produce coffee (Moya; Tenorio; Bond, 2013).

One of the greatest constraints to using coffee pulp for biomass is the lack of a system that makes efficient use of coffee waste (Alfaro; Rodriguez, 1994) mainly because of its high moisture content (McKendry, 2002b). The moisture content of the pulp must be decreased (McKendry, 2002a), which results in increased energy expenses because of the drying process (Moya; Tenorio; Bond, 2013).

It is important to develop knowledge on drying methods for biomass that will be used in pellet and briquette production. The biomass used to manufacture these products is milled and compacted (to increase the density) to obtain low cost and environmentally friendly fuel products (McKendry, 2002b). Despite the benefits of using pellets and briquettes produced from coffee pulp, studies on their processing, from drying to the quality of the end product, are scarce. The Instituto del Café de Costa Rica, a reference that is housed at coffee processing facilities, is aimed at producers that use coffee pulp as an energy source (Alfaro; Rodriguez, 1994).

In this context, the objective of this study is to present a proposal for drying coffee pulp based on three drying systems: air, sun and high temperature chamber. In addition, the properties of the pellets and briquettes produced from dry coffee pulp are presented; their energy contents and physical and mechanical properties were analyzed, and the quality of the pellets was evaluated using X-ray exposure.

MATERIALS AND METHODS

Materials and origin: the coffee pulp was obtained from coffee processing at the Cooperativa de Caficultores y Servicios Múltiples de Tarrazú R.L. (Coopetarrazú). This cooperative is located in San Marcos de Tarrazú, San José, Costa Rica, in a particular geographic region known as Zona de los Santos, one of the main coffee growing areas at an altitude between 1,800 and 2,200 m asl.

Experimental process and types of drying systems.

The wet material was dried using three different methods that are commonly used in Costa Rica. These drying systems are: (i) Air drying: performed in Cartago, Costa Rica, from October through December (Figure 1a). This site is located at 1,380 m asl; the temperature ranges from 17.6 to 19.3 °C, the relative moisture content ranges from 87.5 to 89.5%, the precipitation ranges from 0 to 50 mm month¹ and the wind speed from 12.5 to 13.4 m h¹. (ii) Solar drying:

studies and tests were performed in a solar dryer with a 6 m³ capacity (Figure 1c). A description of this chamber is provided in Salas, Moya and Cordoba (2008) and Tenorio and Moya (2012). (iii) Drying in a hot air chamber: drying was conducted using an experimental NARDI dryer (Soave-Italy) with a 2 m³ capacity and used Leonardo Software for drying control (Figure 1b). In this dryer, the air moves through the material at a speed of 2 to 2.5 m s¹.



Figure 1 – Devices designed for samples drying: air drying (a); a 6 m³ capacity solar dryer (b); experimental (conventional) NARDI dryer (c) and stacked trays with gaps to allow air circulation (d).

Material arrangement and stacking during drying: recipients (trays) were designed to allow air circulation through the top, bottom and sides (Figure 1d). The dimensions of the trays were 50 cm deep x 67 cm wide x 7 cm high. The trays were stacked using a gap of 25.4 mm between the trays to allow air circulation.

Conditions of the drying test: solar and air drying were performed simultaneously to avoid variations in weather conditions. In addition, a hot air drying chamber was set at 80 °C and 4% equilibrium moisture content to achieve a moisture content of approximately 8%.

Humidity control: Six samples from each of the drying systems were used to control the change in moisture content (MC). Because of the small size of the material, a bag with a 0.4 x 0.4 mm mesh size was used to prevent the loss of material and allow airflow through the material. These bags were placed on trays at different heights in the drying stacks to monitor the variation in the MC in different parts of the chambers. The samples were

weighed at the start of each type of drying, and then twice a day for the solar and air drying, and every 2 hours for the drying chamber with hot air. At the end of the drying process, the samples were placed in an oven at 103 °C for 24 hours and the MC was determined.

Pelletizing and briquetting process: The pellet production process was conducted by the PELLETICS company (http://pelletics.com/). This process is designed for manufacturing pellets from waste wood (ribs, edgings, re-sawing residues) and further details are provided by Aragón et al. (2014). The residues were milled using a Kahl fixed ring matrix with 16 mm diameter holes. The granulated material was pelletized using a Kahl machine (model 38-780), which consists of a fixed ring matrix with three rotating cylindrical rollers. The machine reaches 125 °C during this process. The China system was used to fabricate the briquettes. In this system, the material is milled, dried at 70 °C and transferred to a storage bunker before been placed in the briquetting machine. The briquetting machine operates using screw extrusion: the milled material passes through a hot tube at 210 °C. The screw extrusion is located at the bottom of a hopper; the screw is driven by a 15 kW motor and the heating is performed by resistors with a combined power of 3 kW. This type of briquette system is used in the coffee mill of the Cooperativa de Caficultores y Servicios Múltiples de Tarrazú R.L. (Coopetarrazú).

Evaluation of pellets and briquettes made using coffee residues: the quality of the pellets and briquettes manufactured using coffee pulp was evaluated through the determination of various parameters related to energy, physical and mechanical properties. (i) The energy properties included the calorific value according to the ASTM D-5865 standard (ASTM, 2003a), the ash percentage according to the ASTM D-1102-84 standard (ASTM, 2003b) and the moisture content according to the ASTM D-4442 standard (ASTM, 2003c). These properties were measured on 10 randomly selected pellets and 10 randomly selected briquettes. This quantity was selected for statistical representativeness. (ii) Physical properties: These characteristics varied between the pellets and briquettes. The length and diameter distribution was determined on 30 randomly selected pellets, and the number of pellets in 100 g samples were counted. The density, apparent density and the moisture absorption rate of the pellets and briquettes were evaluated. The density of 30 briquettes and 30 pellets was determined. The briquettes were weighed using a scale, and their volume (geometric calculations) was measured to calculate their density. The bulk density of the pellets was determined using a gauge; the length and diameter were measured, the mass was measured using a scale, and the density was calculated using a mass/volume relation ship. The apparent density of the pellets was determined using a container with a known volume and the weight of the material in the container, which was measured using a balance (0.01g precision). The apparent density of the briquettes was determined by dipping these samples into paraffin to obtain an accurate result. The waxed briquette was immersed in water to determine the volume of displaced water, which was equivalent to the weight of the displaced water. The number of pellets in a 100 g sample was counted. This measurement was performed 10 times using 10 different samples. A sample of 30 pellets and 10 briquettes were randomly selected for the moisture absorption test. These samples were simultaneously placed into a desiccator in a concentrated solution of potassium nitrate (equilibrium moisture content of 21%) for 14 days. The samples were weighed before and after this period, and the percent absorption was calculated using equation 1. A friability test consisted of throwing individual pellets from a height of 1 m onto a ceramic floor and noting how many pieces each pellet broke into (i.e., 2, 3 or more parts). After throwing 100 pellets, a ratio between the initial number and the resulting number of pellets at the end of each trial was calculated using equation 2.

$$Humidity absorption = \frac{Initial weight - Weight at 20\%}{Initial weight} *100 (1)$$

Friability =
$$\frac{\text{Initial number of pellets}}{\text{Final number of pellets}}$$
 (2)

(iii) Mechanical properties: the mechanical properties were evaluated using a compression test (briquettes and pellets) and a durability test (pellets). To determine the briquette resistance to compression, 20 pieces of briquettes with a height of 5 cm were selected, of which 10 were tested using vertical axial compression of the briquette and the remaining 10 using horizontal compression perpendicular to the length of the briquette. The samples were tested in a 60 ton capacity universal Timus Olsen testing machine. Aloading speed of 1 mm minwas applied for the axial compression and speed of 3 mm min' was used for the perpendicular compression following Aarseth and Prestlokken (2003). The compression test reports the load relative to the deformation of the pellet and the maximum stress (Equation 3) is calculated. The mechanical durability was performed according to the EN

15210-1 standard (CEN, 2005). The mechanical durability was determined using equation 4:

$$Compression stress = \frac{2 \times maximum load (kg)}{\pi \times Diameter(cm) \times Length(cm)}$$
(3)

Mechanical durability (%) =
$$\frac{\text{Pellet weight before the test}}{\text{Pellet weight after the test}}$$
 (4)

Quality of the pellets: The quality of the pellets was evaluated using images obtained from X-ray exposure, which revealed the presence of cracks in the pellets. Similarly, the quality of the pellet was analyzed using X-ray densitometry to determine the density variation in the transverse and longitudinal directions of the pellet. These studies were conducted on 10 randomly selected pellets conditioned to a moisture content of 12%. The X-ray images were obtained using X-ray equipment (Hewlett Packard Faxitron, LX-60) at a 12 cm distance between the X-ray source and the samples. The exposure conditions were 15 seconds with 30-KV tube tension. To determine the density in the transverse direction, the pellets were carefully cut into sections approximately 1.80 mm thick, which were X-rayed. The pellet density was determined using an X-ray scanner (Quintek Measurement Systems Inc., QTRS-01X). The exposure conditions were 7 KV in the tube and the density readings were performed for 1 second each 40 µm.

Statistical analysis and mathematical modeling of the drying process: Descriptive analysis (i.e., the mean, standard deviation, maximum and minimum values) of all response variables was performed. In addition, the assumptions of the normal distribution, homogeneity of variance and non-presence of extreme data were tested. Subsequently, a variance analysis was performed to test differences in the measured parameters among the different types of drying. Significant

differences between the means were verified using the Tukey test ($P \le 0.01$). A descriptive analysis of the parameters measured on the briquettes and pellets was conducted. An analysis of variance was performed to test the differences in these parameters.

RESULTS AND DISCUSSION

Moisture content of coffee residues driedusing 3 drying systems

There was no difference in the initial moisture content (MC) of the coffee residues between solar and air drying, whereas the initial MC of the coffee residues in the hot air chamber was significantly lower than in the solar and air drying samples (Table 1). The hot air chamber had the shortest drying time to reach 20% MC, followed by solar drying and air drying (Table 1).

The MC variation for the three drying types is shown in figure 2. The hot air chamber had the fastest drying time; the reduction in the MC was linear and rapid. The decrease in the MC with solar drying was slow for the first 180 hours, followed by a rapid decrease in moisture until 400 hours. The MC in the air dried samples decreased slowly until 300 hours, followed by a more rapid decrease until 550 hours, after which the humidity was maintained at approximately 50% (Figure 2).

The MC of coffee pulp exceeds 85% (Shenoy et al., 2011), which is similar to the initial MC measured in this study (Table 1). High MC values are common in agricultural crops. For example, Ratnasingam, Mcnulty and Manikam (2008) reported 67% MC foran oil palm fruit bunch, and an MC of 86% was reported for pineapple leaf (Tenorio; Moya, 2012). Although the unprocessed coffee cherry is reported to have 77% MC (Murillo et al., 1977), the higher MC found in the residues is attributed to the fact that there are steps in the process in which water is used, specifically during pulping, mucilage removal and grain washing, which increases the MC of the pulp.

Table 1 - Initial and final moisture content and drying time of coffee pulp using three drying types.

Types of drying	Initial moisture content (%)	Final moisture content (%)	Total drying time (hours)	Drying time to reach 20% (hours)
Air	90.0 (1.6) ^B	38 (14) ^B	699 (0)	699 (0.0) ^c
Hot air chamber	81.0 (0.3)^	18 (3) ^A	58 (0)	55 (4.1)^
Solar	89.1 (1.9) ⁸	11 (4) ^A	424 (0)	308 (83.2) ^B

Values given in parentheses represent the standard deviation. Different letters within a column indicate significant differences at the 99% confidence level.

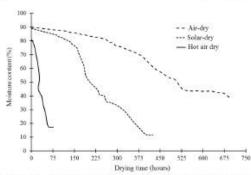


Figure 2 - Variation in the moisture content of coffee pulp relative to time for the three drying types.

The differences in the initial MC between the solar and air drying (89.1% and 90.0%, respectively) samples and the samples in the hot air chamber (81.0%) can be attributed to different levels of fruit ripening, the harvest time and the presence of rain during the harvest (Serrano et al., 2011). In addition, the drying time was related to the temperature in the 3 drying systems. The shortest drying time was measured in the hot air chamber, followed by solar drying and air drying (Figure 2). Higher temperatures result in a more rapid reduction in the MC because of increased evaporation and faster water movement inside the material, whereas low temperatures result in less evaporation and slower water movement inside the material (Tenorio; Moya, 2012). Therefore, longer drying times were recorded under air drying.

Properties of the briquettes and pellets

The pellets and briquettes are shown in figure 3. The energy characteristics of the briquettes and pellets manufactured from coffee pulp are presented in table 2. There were no differences (p-value >0.01) in the average calorific value, ash content or moisture content of the pellets and briquettes. In terms of the physical properties, (i) The bulk density of the pellets was higher than the briquette density, but this was not significant (p-value>0.01). (ii) The apparent density of the briquettes was significantly higher than the pellets. (iii) There was no difference in the moisture absorption between the two product types.

The compression test showed that the maximum stress, the force at the proportional limit, and the vertical and horizontal failure force of the briquettes and pellets were significantly different from one another (Table 2). The pellets presented statistically higher maximum stress values and stress at the proportional limit (Table 2). The greatest maximum force was measured in the briquettes in the vertical direction, followed by the briquettes in the horizontal direction and the pellets; these differences were significant. The durability test applied to the pellets indicated a value of 75%.

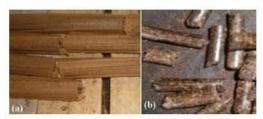


Figure 3 – General aspects of the briquettes (a) and pellets(b) manufactured using coffee pulp.

The calorific values of the briquettes (12.501 kJ kg¹) and pellets (11.591 kJ kg¹), determined using the ASTM D-5865 standard (ASTM, 2003a) are considerably lower than the European standards required for the production of commercial biomass, which range between 17.500 and 19.500 kJ kg¹ (Deutsches Institut für normung, 1996). Therefore, briquettes and pellets manufactured using coffee residues do not meet European standards.

The ash content (8.68% in briquettes and 6.74% in pellets) determined using the ASTM D-1102-84 standard (ASTM, 2003b) failed to comply with the 0.5% ash content allowed by the DIN 51731 standard (Deutsches Institut fur normung, 1996). Furthermore, a high ash content (at a level exceeding 4%) may corrode the burners or boilers and abrade equipment (Mande, 2009).

The MC values (11.4% for briquettes and 10.1% for pellets) (Table 2) measured using the ASTM D-4442 standard (ASTM, 2003c) comply with the range established for this type of product, i.e., 10 to 12%, according to the DIN 51731 standard (Deutsches Institut für normung, 1996). An adequate MC level in pellets or briquettes allows adequate heat and temperature in the chamber and reduces the amount of exhaust gas (Theerarattananoon et al., 2011).

The bulk density of the briquettes (1110 kg m³) and pellets (1300 kg m³) meets the requirements of the DIN 51731 German standard of minimum and maximum values of 1000 kg m³ and 1400 kg m³, respectively (Deutsches Institut für normung, 1996). In addition, these density values are within the range of 1000 kg m³ to 1200 kg m³ established by Torres (2008) for pellets of different crop types.

Table 2 - Energetic, physical and mechanical properties of briquettes and pellets manufactured using coffee pulp.

Properties	Parameters	Briquettes	Pelle	ets	
	Calorific value (kJ kg ⁻¹)	12501^ (490)	11591^ (580)	
Energetic	Ash (%)	8.68 ^ (2.20)	6.74^ (8.80)	
	Moisture content (%)	11.40 ^	11.40^		
	Dimensions	Hexagonal shape of 5.5 cm	Length 20.3 mm (9.63)	Diameter 6.12 mm (0.61	
	Bulk density (kg m ⁻³)	1110^ (3.34)	13004 (3.11)	
Physical	Apparent density (kg m ⁻³)	1000^ (8.72)	600 ^B (2	2.11)	
Section Control Control	Number of pellets in a 100 g	3 <u>5</u> 3	261 (10.11)		
	Moisture absorption (%)	7.90^ (12.40)	8.10^ (5	1.90)	
	Friability		0.95		
	Density in longitudinal direction	1 (kg m ⁻³)	1276.8		
X-densitometry	Density variation in longitudina	4.52			
	Density in radial direction (kg n	1478.4			
	Density variation in radial direct	7.71			

Mechanical properties		Briqu	Pellets	
		vertical	horizontal	horizontal
	Maximum stress (kg cm ⁻²)	11.20^ (54.20)	4.52 ⁸ (68.22)	26.86 ^c (39.25)
Compression test	Stress at the proportional limit (kg cm ⁻²)	9.25^(52.39)	3.77B (74.16)	19.25° (21.1)
	Failure force (kg)	479.73^ (54.96)	93.24 ^B (68.22)	33.50° (38.20)
Durability				75.54 (2.10)

Values in parentheses represent the standard deviation. Different letters within a column indicate significant difference at the 99% confidence level.

The apparent density of the briquettes is higher compared with other materials, e.g., briquettes made from wood chips had an apparent density of 721.24 kg m³ and briquettes made from barley bran had an apparent density of 687.24 kg m³ (Fascina, 2008). The apparent density of the pellets (600 kg m³) is satisfactory according to the literature range of 600 to 700 kg m³ (Larson et al., 2008).

The moisture absorption test in the briquettes and pellets (Table 2) indicated high values, given that the optimum range is 3% to 5% (Fascina, 2008). This result suggests that these products absorb moisture rapidly. However, this can be avoided through the use of appropriate packaging and storage containers or waterproof glue and resinsto reduce the damage caused by moisture (Mckendry, 2002a). High humidity absorption plays an important role in the performance of the products as it may cause a decrease in the quality

and mechanical resistance of the pellets and briquettes (Fascina, 2008).

The value obtained from the friability test was 0.95%. Values equal to or above 0.93% are satisfactory (Soto, Nuñez, 2008).

The horizontal compression test indicated that the maximum stress in the pelletswas higher than in the briquettes (Table 2). This difference can be attributed to the pellet's cylindrical shape, which is less resistant than the briquette's hexagonal shape. In addition, cracks, a common characteristic in milled materials once processed (Mani; Tabil; Sokhansanja, 2003), weaken the pellet (Serrano et al., 2011). Furthermore, the density may influence the maximum stress (Serrano et al., 2011; Mani et al., 2006). In this study, the apparent density was higher in the briquettes than in the pellets (Table 2).

The durability test values (75.54%) are not acceptable according to the values determined for

pellets made using mixtures of invasive acacia and nutshells tested using the ASAE269.4S standard. Commercial pellets should have durability values above 90%. Mechanical durability is essential in pellets to maintain their manufacturing conditions and to prevent dust during transport and storage (Lethikangas, 2001).

Pellet quality

The X-ray images showed that the pellet was uniform and there were few cracks on the surface (Figure 4a). The average density of the pellet determined using X-ray densitometry was 1276.8 kg m³ in the longitudinal direction and 1478.4 kg m³ in the transverse direction. The variation coefficient was higher in the transverse than in the longitudinal direction (Table 2). The variation of the density in the longitudinal direction was uniform (Figure 4b).

Three patterns can be distinguished in the density in the transverse direction: in the first pattern (Figure 5a), high density is observed at the ends with low density in the central part; in the second pattern (Figure 5b), there is a high density portion (shown here in the center of the pellet), and the third pattern (Figure 5c) shows a homogeneous density.

Higher variation was observed in the density of the pellet in the transverse direction compared with the longitudinal direction. This finding can be attributed to the irregularities in the density in the transverse direction (Figure 5), which were not observed in the

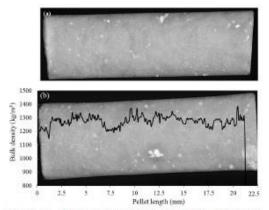


Figure 4 – a) X-ray image showing the pellet quality and (b) variation in the density in the longitudinal direction of pellets made using coffee pulp.

longitudinal direction (Figure 4). The quality of the pellet can be evaluated by the presence of cracks, which are directly related to the susceptibility to rupture (Stelte et al., 2011). The X-ray images showed small cracks (Figure 4). These cracks are common in milled material once pelletized (Kaliyan; Morey, 2009) and they are attributed to an inadequate MC for pelletizing or to inadequate particle sizes. The MC significantly affects the physical properties of biomass densification (Fasina, 2008) because water strengthens particle bonding. A decrease in the water content reduces the capillary strength (which maintains the structure of the pellet) and leads to the formation of cracks.

The density profile of the pellet in the longitudinal direction is shown in figure 4b. The variation in the density profile is small compared with pellets made from other crops that were evaluated using the same technique. For example, high irregularity was found along pellets manufactured from Pennisetum purpureum, Gynexamplerium sagittatum, Phyllostachys aurea and Arundo donax. However, Sorghum bicolor pellets have similar variation and quality as the pellets manufactured from coffee pulp.

The density of the pellet had a greater variation in the transverse compared with the longitudinal direction (Figures 4 and 5). This finding can be attributed to the heterogeneous sizes of the particles produced by the pellet manufacturing process, which favors a heterogeneous densification (Ramírez-Gómez et al., 2014). A high density profile was observed around the edge of pellet surface compared with the central section (Figure 5a). This type of pattern was observed in a wood briquette study, in which the difference in compaction was attributed to the variation in temperature and internal pressure of the briquette (Quirino et al., 2012).

The quality of the pellets, evaluated by the presence of cracks and the density profiles in this study, indicates the degree of effectiveness of the amount of compressed material and the pressure generated during the pelletizing process (Relova et al., 2009). These techniques can be used to explain the performance of the pellets, e.g., the presence of cracks and high variability affect pellet durability and fragility (Serrano et al., 2011). The results obtained in the present study indicate that the pelletizing process used for the coffee pulp was suitable.

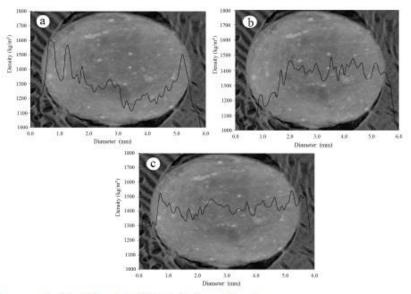


Figure 5 - Transverse density of the pellets fabricated using coffee pulp.

CONCLUSIONS

The drying times for the coffee residues were 699, 308 and 55 hours for air, solar and hot air drying, respectively. Hot air drying is therefore the best option for coffee residues because of its low drying time. The physical properties of the pellets and briquettes comply with most of the standards established for these products. The calorific value, however, is lower than the established standards. The pellets and briquettes did not differ (p-value >0.01) with respect to the calorific value, ash content and moisture content. The compression stress of the pellets was significantly (p-value >0.01) higher than the briquettes; the briquettes, however, resisted greater loads. Anegative aspect of the pellets is that their durability failed to comply with some requirements specified in the literature. The presence of cracks may affect the pellet quality. Furthermore, X-ray densitometry showed that the density variation of the pellet was more uniform in the longitudinal direction compared with the transverse direction.

ACKNOWLEDGEMENTS

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6. Artículo 6. Production and quality analysis of pellets manufactured from five potencial energy crops in the northern region of Costa Rica.

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Production and quality analysis of pellets manufactured from five potential energy crops in 1 the Northern Region of Costa Rica 3 Stephanie Aragón-Garita^a, Roger Moya^a, Brain Bond^b, Jorre Valaert^c, Mario Tomazello Filho^d 5 ^a Instituto Tecnológico de Costa Rica; Escuela de Ingeniería Forestal; P.O. Box: 159-7050 Cartago-Costa Rica; Phone: (506) 2550-2433 / Fax: (506) 591-3315; *Corresponding author: rmoya@itcr.ac.cr 7 8 Department of Sustainable Biomaterials, Virginia Tech. Blacksburg, Virginia, USA, bbond@vt.edu 10 Agrep Forestal S. A.; Phone: (506) 2474-7025; jorre@agrepforestal.com ^d Departamento de Ciências Florestais, ESALQ/Universidade de São Paulo, Av. Pádua Dias, 11-Caixa 11 12 Postal 09, CEP 13418-900-Piracicaba, São Paulo Brasil; mtomazel@usp.br 13 Abstract 14 Pellet production needs calibration and adaption depending on the raw material. This work presents the 16 production calibration for pelletizing five energy crops in a wood pellet manufacturing plant in Costa Rica. The evaluated aspects include: the caloric value, the loss of moisture content in each production 17 18 stage and the convertion ratio of particle-pellet, quality, tranversal density and surface density of the pellets through X-ray radiography. The results showed that the analyzed crops were rhizomatous plants, 19 with caloric values ranging between 17.1 and 20.3 MJ kg⁻¹. It was possible to produce pellets with 20 Gynerium sagittatum and Phyllosstachys aurea with the same production process used for wood. 21 22 However, Arundo donax and Pennisetum purpureum needed pre-air-drying and to the Sorghum bicolor was a mechanical dewatering stage applied before the drying. A. donax, P. purpureum and G. sagittatum 23 showed the highest convertion ratio of particle-pellet with 87, 76 and 68%, respectively. In the quality 24 evaluation, P. aurea and G. sagittatum were observed to have a large quantity of cracks, unlike A. donax, 25 26 P. purpureum and Sorghum bicolor. The tranversal and surface pellet density varied from 1129 to 1294 kg m³. The highest values of bulk density were obtained in A. donax and P. purpureum, followed by G. 27 28 sagittatum and P. aurea, and the lowest bulk density was obtained in S. bicolor.

29 Key words: biomass, waste, lignocellolose crops, short rotation crops.

1. Introduction

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Tropical conditions in Costa Rica allow the development of many agricultural and forestry crops, activities that generate a high amount of lignocellulosic waste [1]. The forest industry generates about 500,000 Mg of waste [2], this estimate does not consider that 43% of the volume of the tree remains in the field as waste [3]. The agricultural sector is estimated to produce approximately 1.5 million Mg of waste per year [4]. The forest industry has been concerned with finding solutions to the waste problem and one possbility is to genereate heat by direct combustion in the wet condition [5]. Manufacturing waste into pellets has captured the interest of these industries, where pellets could provide the source of heat for other processes, such as drying of fruits and vegetables, cooking or cleaning products [4].

39 Wood pellets are manufactured by a mechanical process in which pressure is applied to crush the 40 cell structure and increase its density [6]. The normal process of producing wood pellets consists of: (i) 41 using grinders and mills (discs, rollers, balls, blades or hammers) to reduce the size of the material, (ii) 42 drying the biomass with rotary drum or pneumatic type driers to a 10 % moisture content and (iii) 43 compressing the material using rotary rollers which pushes the particles from the inside of a ring or die 44 (cylindrical or annular type or flat matrix) outwardly through a series of holes [7-8], a technology 45 commonly used to produce animal feed. While pellets can be made from wood, the high demand for wood 46 waste in fiberboard and particleboard production represents direct competition for pellet industries in Costa Rica [5]. There are two important aspects regarding the use of wood waste in Costa Rica: (i) a 47 48 tendency to reduce wood consumption in industry [9] and (ii) the waste produced is in high demand to 49 keep chicken, horse and cattle breeding sites clean [5]. Pellet industries are therefore seeking alternative 50 raw material with high lignocellulosic content, such as agro-wastes, or the cultivation of energy crops that 51 can be densified [8]. Agricultural wastes are currently in high demand as a supplement for animal feed 52 concentrate [10-11] or to produce organic compost products [12].

The use of agricultural or sawmill residues for pellet production is highly developed in many countries [7]. Pellets are as efficient in boilers as other fuels [13]. For pellet production to be economically viable, pellet industries are generally designed to process raw material with moisture content less than 55%. [14]. One limiting factor to the use of energy crops for pellet manufacturing is that many have initial moisture contents above 50%, present lower hardness and smaller sizes than wood waste [15]. For the industry to be profitable manufacturing pellets from different type of biomass, such as the energy crops, with different morphology and moisture content, the process needs modification [1]. For example, air-drying in those areas with appropriate environmental conditions [16], would result in more cost-effective production [17].

With these constraints, the objective of the present work is to define the alterations in a wood

With these constraints, the objective of the present work is to define the alterations in a wood pellet production process that would allow the use of five potential energy crops (Arundo donax, Gynerium sagittatum, Pennisetum purpureum, Phyllostachys aurea, and Sorghum bicolor) in Costa Rica. The particle size prior to pelletizing, process efficiencys, moisture content in each stage of the pellet production and quality, tranversal density and surface density of the pellets by X-ray spectrometry were investigated.

2. Materials and methods

70 2.1. Selected crops

Five energy crops were selected for use in Costa Rica (Figure 1 and Table 1) based on the following constraints: adaptation to climatic and edaphic conditions of this country, dry biomass production above 20 Mg ha⁻¹ and availability throughout the year. The five selected crops are perennial, with straight stems ranging from 2 to 6 m high and diameters from 1 to 5 cm (Figure 1). *G. sagittatum, P. aurea*, and *S. bicolor* samples were harvested and transported to the plant located in San Carlos in one day and the pellets were prepared the next morning. For *A. donax*

and P. purpureum material was left in the field where it was cut, and allowed to dry to decrease moisture content for three day. (see 2.3.1).

2.2. Crop characterization

The characterization of each crop is based on the evaluation of the caloric value in the wet condition (after harvest, named Gross Caloric Value) and dry condition (0% moisture content, Net Caloric Value). For this analysis, 3 stalks were randomly selected, cut and then milled smaller than 2 mm and then sieved with #60 and #40 meshes (0.25 mm and 0.42 mm respectively). The material between #60 and #40, mesh was selected and 3, approximately 1 gram, samples were extracted. This material was then divided in two parts of 0.5 grams each. A sample with the moisture content at the time of harvesting was used to determine the Gross Caloric Value (GCV); the second sample was kiln-dried at 103 °C during 24 hours, and then the net caloric value (NCV) was determined according to the ASTM D-5865 standard [18]. Net and gross caloric values were determined using Parr calorimetric test. Carbon fraction (C), Nitrogen content (N) and the C/N relation were determined from milled dust using Elementar Analysensysteme, Vario Macro Cube model. The material was sieved through 0.25 mm and 0.42 mm meshes (40 to 60 meshes respectively), until approximately 8 g per test were obtained.

2.3. Crop evaluation in the production process

Five aspects were considered in the evaluation of potential energy crops in Costa Rica: (i) adaptation of the crop to a wood pellet production process; (ii) loss of moisture content during the pellet production; (iii) characteristics of the produced particles; (iv) efficiency in the process and (v) pellet quality.

2.3.1.	Crop adaptation	to the normal woo	d pellet pro	duction process
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The normal pellet manufacturing process from Agrep Forestal S. A. was used (Figure 2). The production consists of a wood shredder, with loose knife hammers, from which the material is directed towards a pan mill. The ground material passes to a rotary drum dryer (12 m long x 3 m) direct heated by hot air at 400°C. The particles are then pelletized (KAHL 35-780), with a 780 mm diameter flat die, orifice channel 30 mm length and 6 mm diameter, i.e., the length/diameter is a ratio of 5. However, three different approaches were used for removing moisture within of stem of crops:

- (i) The normal drying stage of the pellet manufacturing process from Agrep Forestal S. A., where the ground material passes to a rotary drum dryer was applied in G. sagittatum and P. aurea.
- (ii) A stage of pre-air-drying, in addition to normal drying stage, was added in A. donax and P.
 purpureum. The stems with leaves of these species were left in the field (Figure 3a-b) for a period of
 three days. This period was selected because it allows the stems begin drying but prevents decay.
- (iii) A milling and pressing stages were applicated in S. bicolor in addition to normal drying. For milling,
 a shredding machine was used, this machine is used commonly milling of stem of cane. Afterwards a
 press for water extraction was used and too used for sugar extraction from stem of cane.

2.3.2. Evaluation of moisture loss during the production of pellets from energy crops

MC was evaluated at each stage of the pellet production process: (1) harvesting, (2) pre-air-drying (applicated only in A. donax and P. purpureum material) (3) shredding, (4) milling, (5) pressing (applicated in S. bicolor), (6) drying and (7) MC in pellet fabrication. In the havesting stage, 30 plants per crop were randomly cut and immediately weighed and oven-dried at 103°C for 24 hours. The dry weight was then determined according to the ASTM D-3173 standard [19] and the moisture content calculated using Equation 1.

Moisture Content (%) =
$$\frac{(Green\ weight\ - Oven\ dry\ weight\)}{Green\ weight} \times 100$$
 (1)

A pre-air drying stage was added in A. donax and P. purpureum species. The stem with leaves was left in the field (Figure 3a-b) for a period of three days. The change of MC was measured during this time. Six samples of 5 plants each were taken for MC samples control. The samples were weighed each day at 10:00 am. After third day, the samples were dried at 103 °C for 24 hours, weighed and the MC determined by Equation 1. In the remaining stages for these species and others, the MC was determined by removing 10 random samples after each stage, followed by the ASTM D-3173 procedure.

2.3.3. Fineness of the particles

The fineness of particles was measured using different sieves before pelletizing. The methodology consisted of taking 10 samples of 100 grams, which were sieved on a stack of 1 mm, 2 mm, 4 mm, 6.7 mm, 8 mm, 11.2 mm and 16 mm sieves for a period of 2 to 3 minutes. Afterward, the remaining material in each of the sieves was weighed. The particle size distribution was determined by the mass of the particles retained in the sieve and between the total mass of the sample, expressed in percentage. This mass was named "mass retained". The mass that passed through the sieves was then weighed and percentage calculated and named "mass not retained". This information was used to determine the fineness index (Equation 2) used by the Brazilian NBR 7211/83 standard [20].

$$Fineness\ index = \frac{\sum mass\ retained\ in\ siefes - mass\ not\ retained\ percentage}{100} \tag{2}$$

2.3.4. Convertion ratio of particle-pellet

Convertion ratio of particle-pellet refers to the weight of particles that enter the pelletizing process compared to the amount of pellets with size bigger than 4.75 mm. This convertion rate was calculated by relation between weigh of pellets gathered and sieved with a 4.75 mm (mesh #4) and particle before pelletizing process, expressed in percentage.

2.4. Quality of the pellets

Pellet quality was evaluated using images digital obtained from exposure to X-rays, which revealed the presence cracks and area with high density in the surface of pellets. Variation of surface quality for longitudinal and transversal density was measured with this x-ray image. Pellet quality was measured in 10 randomly selected pellets per crop and conditioned to a moisture content of 12%. Cracks were areas where layers in the pellet are separated. One area with high density presents usually density values with values over 1300 kg m⁻³: X-ray images of the pellet material was obtained using X-ray equipment (Hewlett Packard Faxitron, LX-60) at a 12 cm distance between the X-ray source and the samples.

Exposure conditions were 15 seconds with 30 kilovolts tube tension.

The number of cracks present in each pellet was determined using X-ray digital images for 10 pellets and the quantity of cracks reported as frequency in relation to pellet length (cracks/mm). The images were analyzed by ImageJ software (National Institute of Mental Health, Bethesda, Maryland, USA). The digital image in TIFF fomat is filted to a threshold equal to 208. Then areas with color higher than 208, was used to represent a region with high density. These regions were compared in relation to its frecuenty (quantity/mm²) and percentage of area with high density in relation of total area of pellet. Likewise, the quality of the pellet was analyzed using the X-ray densitometry to determine the density variation in transverse and longitudinal directions. Pellet density was determined by means of an X-ray scanner (Quintek Measurement Systems Inc, QTRS-01X). Exposure conditions were of 7 kilovolts in the tube and the density readings were performed for one second each 40 µm. The first density measurement is named transveral density (TD) and the second one, surface pellet density (SPD). When measuring the transverse and longitudinal density, each pellet was weighed and its diameter and length measured. The samples by crop were fixed in horizontal position on a support and then X-rayed to determine the density in TD. To determine the SPD, the pellets were carefully cut into approximately 1.80 mm thick sections which were then X-rayed.

173 2.5. Statistical Analysis

A variance analysis (ANOVA) was applied to estimate the differences in GCV, NCV and MC in the stages of the pelletizing process, regarding the fineness index and TD, SPD and their variation coefficient determined by the X-ray densitometry. These variables were established as depend variables and type of crops as independ variable in the ANOVA. The existence of significant differences between the averages for each variable was verified by the Tukey test (P<0.05). Regarding the density values; a correction factor was created that included the actual pellet density, previously determined by measuring its weight, length and diameter, and average density (transversal and surface pellet density) measured by x-ray densitometry (Equation 3). Each value of density measured for X-ray densitometry was corrected with the aid of equation 4. With the corrected density values, density profiles were constructed in TD and SPD in order to determine patterns of variation.

Correction factor = Average density_{measured by x-ray densitometry} - Actual density (3)
$$Corrected \ density_i = Density_i - Correction \ factor \ (4)$$

Where: Corrected density, represents density values for each value measured for X-ray densitometry and density, values measured for X-ray densitometry. *Note:* density measured for X-ray densitometry was read each 40 µm in transversal or longitudinal pellet direction.

Results and discussion

191 3.1. Crop characterization

Caloric values are shown in Table 2. GCV varies from 2.5 to 10.3 MJ kg⁻¹, NCV ranging from 17.1 to

20.3 MJ kg⁻¹ and when calculated from the pellet the variation is small, 15.6 to 17.6 MJ kg⁻¹. The analysis

of variance identified three groups in relation to GCV: a first group with the highest values of GCV

included A. donax, G. sagittatum and P. aurea; a second group consisting of P. purpureum, which

converges with two species of the first group (G. sagittatum and P. aurea), as they contain no statistical differences (Table 2) and a third group, S. bicolor, with the lowest GCV. The ANOVA applied to NCV distinguished two groups, one composed of A. donax, P. purpureum and S. bicolor with higher values of NCV and a second formed by G. sagittatum and P. aurea and two species (A. donax, P. purpureum) that overlap with the first group.

The energy evaluation of the GCV shows a considerable increase of the caloric value with respect to the NCV (Table 2). This increase is supported by the decrease of the MC in the NCV. For a low MC, higher thermal efficiency is observed in the combustion process, due to lower energy investment in evaporation of the water in the biomass [21]. The influence of MC can also be observed in the values of GCV among the different crops; S. bicolor and P. purpureum, with high MC values (Table 3) resulting in the lowest values of GCV (2.4 and 7.0 MJ kg⁻¹, respectively). On the other hand, the species A. donax, G. sagittatum and P. aurea, with a MC lower than 60% (Table 3), resulted in higher GCV values than the previous species (Table 2).

NCV and GCV values obtained in pellets from five crops (Table 2) are consistent with the values reported for fast-growing timber species in Costa Rica [22], making them recommendable as an alternative agrifuel. NCV variations between the five crops can be explained by changes in nitrogen and carbon content and the carbon/nitrogen (C/N) ratio. It was found that NCV increased with nitrogen content (Figure 4a), but decressed with carbon content (Figure 4b) and C/N ratio (Figure 4c). However the variation between crops cannot be explained only by C and N content in each crop, because NCV is also affected by other chemical properties. For example, the content and characterization of the ash and the amount of volatile substances possitively affect caloric values [23]; however, the extractive content and fixed carbon contribute to an increase in the caloric value [22].

- 219 3.2. Crop evaluation in the production process
- 220 3.2.1. Crop adaptation to the normal wood pellet production process

Two species (G. sagittatum and P. aurea) among the five crops evaluated, adapted well to the normal process used for wood pellet production (harvesting, shredding, milling, drying and pelletizing), whereas for the remaining three (A. donax, P. purpureum and S. bicolor) required some modifications to the process (Table 3). For A. donax the shredding process presented no problems. An air-drying stage to reduce the MC was applied to A. donax and P. purpureum (Figure 3c). The remaining stages for these crops correspond to the normal pelletizing process. In pre-air-drying stage, the stems were left in the field for a period of three days after cutting; then the semi-dried material was taken to the shredding process. With S. bicolor it was not necessary to apply the commonly used type of shredding for woody material; instead, the harvested material was directly milled with an axial-feed mill equipped with four rotating blades. Subsequently, the material received a water extraction pretreatment by compressing it at 8.3 MPa with the press used in the extraction of juice from sugar cane.

G. sagittatum and P. aurea adapted well to the existing pelletizing process from Agrep Forestal S.

A. Although these crops differ from wood waste in its form (small cylindrical stalks less than 5 cm diameter), shredding and milling had no problems. The drum type drying system yielded a grinded particle with optimum MC (less than 10%) before pelletizing. Another advantage of the system used is that the pellet showed less than 10% MC (Table 3). According to McKendry [15], the success of the biomass conversion process into pellets, lies in having a low MC raw material (about 50%). This research has demonstrated that G. sagittatum and P. aurea are potential crops for the production of pellets using the same production system used for waste wood. Although S. bicolor, P. purpureum and A. donax are species with high MC (60-80%), the same machinery to produce pellets from wood waste can be used, although probably affecting the energy balance [24] and representing an increase in costs. High values of MC make it necessary to implement alternative processes (pre-drying or compression of the biomass) to reduce them [25]. This was evidenced by pre-air-drying applied to A. donax and P. purpureum, with which the MC diminished 28% and 13%, respectively, and a 29% MC decrease in S. bicolor by applying biomass compression.

Pre-air-drying, as used for A. donax and P. purpureum (Figure 3a-b), is a practice applied to herbaceous species in some European countries to maintain the energy balance [25]. However, for tropical countries this should be handled more carefully, as these regions have two seasons, dry and wet. The rainy season prevails for most of the year, characterized by the presence of high relative humidity, which causes the biomass MC reduction to be slow [10], while promoting the biological degradation of the biomass.

Regarding biomass compression, as applied to S. bicolor, specialized equipment for the extraction of sugar cane juice was used. The result was successful because the MC decreased from 81.9 to 52.4% (Table 3). Unlike timber, which is composed of highly bonded fibers and a hard outer surface, moisture removal in the crops studied by mechanical means is possible, since they are herbaceous plants that have more flexible bonded fibers [15], which enable the compression of the biomass to extract the water trapped in the tissues.

3.2.2. Evaluation of moisture loss during the production of pellets from energy crops

P. purpureum and S. bicolor had the highest average MC (81 and 85.5) during the harvest season, while A. donax, G. sagittatum and P. aurea had the lowest MC values (56, 57.3 and 43.4 respectively) (Table 3). For the two species that used an air-drying stage it was determined that A. donax presented MC in this stage lower than P. purpureum. In the grinding stage, the MC values were statistically different for the 5 crops, ranging from 81.9 to 25.1% for S. bicolor and A. donax respectively. The MC after drying and after the biomass had been pelletized, was statistically different for the 5 crops, ranging from 7.6 to 15.0% after drying and 6.7 to 12.6% after pelletizing. P. purpureum and S. bicolor presented the highest values in both stages, followed by A. donax, G. sagittatum and P. aurea, which showed the lowest values (Table 3).

It was demonstrated that pre-air-drying of the stems of A. donax and P. purpureum caused a 27% and 13% reduction in MC over the three days (Figure 3c). In the shredding stage of the applied crops, the MC decreaseed only 1 to 2% (Table 3), with the exception of P. aurea. In the case of biomass milling, the MC reduction in relation to the previous stage increased from 2 to 7%, the lower value being for P. urea and the highest value for G. sagittatum. In S. bicolor, were pressing before drying was applied, a 29.5%

MC reduction was observed. As expected, drying resulted in the largerst reduction of MC for all processes. Finally, in the pelletizing process, the MC diminished in the 5 crops and varied from approximately 1 to 3%.

3.2.3. Fineness of the particles

The evaluation of fineness of the particles before pelletizing showed that the crops can be divided into four groups (Figure 5a): the first, with the highest fineness index (0.34), comprising *P. aurea*; the second, with indexes between 0.27 and 0.29, including *G. sagittatum* and *P. purpureum*, a third group integrated by *S. bicolor*. *A. donax* presented the lowest fineness index. These differences arose from the variation in particle size for each crop. A high percentage of large particles (4-16 mm) decreased the fineness index value, as in *S. bicolor* and *A. donax*, which have the highest percentages (Table 4). Contrary, a high percentage of particles with size less than 4 mm, as in the case of *G. sagittatum*, *P. purpureum* and *P. aurea*, produced the lowest fineness index values (Figure 5a). The variation in particle size was influenced by two factors: (i) not applying the same manufacturing process for the 5 species, such as reducing the size of the biomass in *A. donax* (shredding) and *S. bicolor* (milling), as well as the MC reduction, where the shape and size of the particles was dependant on the milling parameters (hammers, blades and rotation speed) [26] and (ii) characteristics of the raw material. Those species with less lignified tissues tend to bend, and thus to shred into long pieces, while species with more lignified tissues tend to break rather than bend, and thus are fragmented into shorter chunks [27].

3.2.4. Convertion ratio of particle-pellet

The highest conversion ratio was obtained for *P. purpureum*, followed by *A. donax, G. sagittatum* and *S. bicolor* with values of 68 and 61%, respectively. *P. aurea* presented the lowest convertion ratio (Figure 5b). The variation in the convertion ratio for the five crops (Figure 5b), is dependent on a number of factors that affect the process of compression of the biomass. In herbaceous plants, lignin and proteins are responsible for making connections between particles, and the ratio of these compounds varies according

to the type of biomass [28]. Also, the MC of the biomass before pelleting is one of the major factors [29]. This study showed that when the MC ranged from 10-15%, as in P. purpureum, A. donax and G. sagittatum, it resulted in a higher convertion ratio, whereas when the MC of the particles before pelleting was less than 10% (P. aurea), the lowest convertion ratio was obtained. Excessive drying, as occurred in P. aurea, has a hardening effect on the tissue surface and serves as a thermal insulator, thus preventing heat transfer, which is a key element for compression [28]. The low yield of S. bicolor in relation to A. donax and P. purpureum, cannot be explained by the MC, since this crop is within the optimum range of MC (Table 3) but may be explained by the high content of large particles (16 mm), having been found that fine particles (2 mm) improve binding [30].

308 3.3. Pellet quality

The presence of cracks is one aspect of pellet quality, since they are directly related to the susceptibility of breakage [31]. The evaluation using X-ray images shows that surface cracks were the lowest in the pellets manufactured with *P. purpureum* (Table 5) and this crop presented only some irregularities (Figure 6e). For *S. bicolor* and *A. donax* the quantity of cracks was higher than *P. purpureum* (Table 5, Figures 6a and 6c). The highest quantity was observed in pellets manufactured with *P. aurea* and *G. sagittatum* (Table 5, Figures 6d and 6b), being most evident in *P. aurea*.

X-ray photography showed that cracks were present in varying degrees for all crops (Table 5, Figure 6). The presence of these cracks is common in milled materials once pelletized [32] and is attributed to inadequate MC for pelletizing or to inadequate particle size. MC significantly affects the physical properties of densifying the biomass [33], since the presence of water strengthens the bonds between particles. The decrease in water reduces the capillary force that maintains the structure of the pellet, leaving cracks. This is seen in the results, where those crops that presented MC between 12-15% (A. donax, P. purpureum, and S. bicolor) showed fewer cracks, whereas P. aurea and G. Sagittatum, species with MC values of 7% and 10%, respectively, showed a higher number of cracks in the pellet surface.

Another aspect made evident by the X-ray photography, is the presence of light colored areas in the pellet, which is more frequent, therefore high in relation to total area (Table 5), in A. donax (Figure 6a), followed by P. purpureum, G. sagittatum (Figure 6b-6c) and lastly, by P. aurea and S. bicolor where such areas are minor frequency and area in relation to total area (Table 5, Figure 6d-e). Light areas observed in the pellet are related to variations in density (Figure 7b-7e). These regions correspond to differences in size and structure of the pelletized particles, due to the heterogeneous anatomical structure of the crops. For example, the P. purpureum has a higher concentration of fibers in the outer layers of the stem, and cutinized and waxy layers [34], which results in density increasing from the inner part to the outer part [35]. Another example is the species A. donax whose anatomical structure varies between the nodes and internodes and with increased wall thickness of the fiber at the nodes [36]. This variation of the structure and the presence of regions of high density in the plant, causes these parts to remain located at certain regions of the pellet during milling, resulting in an increase in density in that region (Figure 7b-7e).

The pellet density values, for both TD and SPD, for the five crops ranged from 1129 to 1293 kg

The pellet density values, for both TD and SPD, for the five crops ranged from 1129 to 1293 kg m⁻³ and three different groups were established (Table 6): the first group consisting of high density species, A. donax and P. purpureum, with no statistical differences between the values of density; a second intermediate density group, including G. sagittatum and P. aurea; and a third group, also composed of P. aurea and S. bicolor, forming pellets of lower density. There was trendency to diminish density with the decrease in the MC of the pellet. P. purpureum and A. donax had the highest density values, with 12.11% and 11.98% MC respectively, then G. sagittatum with 9.85% MC and P. aurea with 6.68% MC. These last two species showed a large quantity of cracks (Tabla 5), which are empty spaces with the resulting density reduction. However, S. bicolor obtained the lowest density value (Table 6) with 12.64% MC, which demonstrates that, similarly, with an increase of the pellet MC to over 12%, the density tends to diminish [30]. In addition, a ratio of the density of the species with the fineness index is established, since the coarse grain size present makes the arrangement of large particles difficult compared to small particles [37].

The TD variation of the pellet was greater than SPD (Table 6). This difference is a result of the three density profiles found in the TD, the irregular profile is the one that is present in greater amounts in the pellets analyzed for each crop. This confirms that the process of pellet manufacturing generates heterogeneous particle sizes, which favors a heterogeneous densification [38]. A high density profile around the surface of the pellet in relation to the central part was also observed. This pattern was present in a study of transversal density variation with wood briquettes of Eucalyptus species, where the external high density of the briquette is explained by the variation in temperature and internal pressure of the briquette [37].

The evaluation of the TD of the pellet shows three patterns of variation: high density around the surface, uneven density and uniform density. The first pattern has a greater density variation on the surface of the pellet and a lower density in the interior (Figure 7a); the pattern of irregular density shows high values of density at any point in the pellet diameter (Figure 7b), and the uniform density pattern presents similar values along the diameter (Figure 7c). Evident examples of the first pattern are A. donax and P. purpureum (Figures 7a and 7c). The second pattern (Figure 7b) is evident A. donax, P. purpureum and P. aurea. Lastly, the uniform density pattern (Figure 7c) can be clearly observed in the five crops.

Meanwhile, two density variation patterns were determined in SPD: uniform density and irregular density. For the first pattern, there is little variation (Figure 7d). There is irregularity in the density of the pellet in the second pattern, with areas of low or high density (Figure 7e). Figure 7 shows the variation of SPD for the 5 species. This figure shows also that the uniform density variation pattern was found in S. bicolor (Figure 8a), and later this uniformity begins to diminish in P. purpureum and G. sagittatum (Figures 8b-c) until reaching the irregular pattern of the P. aurea and A. donax (Figures 8d-e).

The analysis of the density variation by means of the coefficient of variation (CV) showed a lower density value in SPD than in TD (Table 6). The ANOVA showed that A. donax is the species with the highest CV, whereas the lowest is for S. bicolor. However, both crops present no statistically different values from the other three species. The differences of CV found in the TD divide the crops in two groups: the first with statistically higher values including P. purpureum and A. donax, and the second group

composed of G. sagittatum and P. aurea. S. bicolor shows no statistical difference with respect to both groups.

The density profiles in SPD (Figure 8) show the varying CV found in the five species (Table 5).

The irregularity of the density throughout the pellet and the presence of sharp peaks in P. aurea and A. donax (Figures 8d and 8e) are consistent with the highest CV values (Table 6). Also in these species, the high variability in the density may also be related to the high abundance of clear zones that are areas of higher density (Figures 6a and 6d). By contrast, a uniform density profile (Figure 8a) as in S. bicolor, show low CV (Table 6) and few zones of high density or lightareas (Figure 7e).

The quality evaluation of pellets by means of the presence of cracks and the density profiles, show the degree of efficiency of the amount of compressed material, and the pressure generated during the pelletizing process [29]. The use of these techniques can have effects on or explain the subsequent performance in pellet quality. The presence of cracks and high variability of density have effects on pellet durability and fragility [28]. Considering this and the results obtained, the pelleting process applied to P. aurea produces a lower quality pellet in relation to the species P. purpureum, A. donax, G. sagittatum and S. bicolor, due to their high amount of cracks and low density. However, G. sagittatum also contained high quantities of cracks and S. bicolor had the lowest density value.

4. Conclusions

The selected energy crops are of interest in relation to net dry caloric and their adaptation to pelletizing process used by Agrep Forestal S. A. Gynerium sagittatum and Phyllosstachys aurea did not show any problems with this process. Sorghum bicolor, Pennisetum purpureum and Arundo donax, species with high MC required other techniques to reduce the moisture content. The adaption of this pelletizing process for the five crops generated two major differences: a variation in the distribution of particle size, with the consequent change in the fineness index; and the convertion ratio of particle-pellet was different in each crop.

400 401 As to the quality of the pellet, it was found that in P. purpureum, A. donax and S. bicolor, the moisture content varied from 11 to 13%, whereas in G. sagittatum and P. aurea, the moisture content was less 402 403 than 10%. Finally, the results indicate that the pelletizing process applied to P. aurea produces a lower 404 quality of the pellet in relation to the species P. purpureum, A. donax, G. sagittatum and S. bicolor, due to the high amount of cracks and low density of P. aurea. However, G. sagittatum also contained high 405 quantities of cracks and S. bicolor had the lowest density value, resulting in lower quality pellets. 406 407 408 Acknowledgement The authors wish to thank the Vicerrectoría de Investigación y Extensión at the Instituto Tecnológico de 409 410 Costa Rica (ITCR), Ingenio Taboga and Ingenio el Viejo, with special thanks to Dr. Fermin Subiros for 411 the raw materials and facilities for the study. References 412 413 [1] Ulloa JB, Weerd JH, Huisman EA, Verreth JA. Tropical agricultural residues and their potential uses 414 in fish feeds: The Costa Rica situation. Waste Manag 2004; 24(1): 87-97. 415 [2] Serrano R, Moya, R. Procesamiento, uso y mercado de la madera en Costa Rica: aspectos históricos y análisis crítico. Revista Forestal Mesoamericana Kurú 2011; 8(21): 1-12. 416 [3] Espinoza J, Moya R. Aprovechamiento e industrialización de dos plantaciones de Gmelina arborea de 417 15 años en diferentes condiciones de pendiente. Rev Chapingo Ser Cie 2011; 19(2): 238-248. 418 419 [4] Ramírez F, Carazo E, Roldán C, Villegas G. Encuesta de oferta y consumo energético nacional a partir 420 de la biomasa en Costa Rica año 2006. San José, Costa Rica, 2007. 421 [5] Chacón LR. Diagnóstico de las existencias de los residuos forestales en la región Huetar Norte de 422 Costa Rica. San José, 2012.

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Table 1. Geographic location and information on the state of the five energy crops evaluated.

Crop	Description of the crop site		
Arundo donax	It is a rhizomatous plants belonging to the sigmoidal group (Figures 1a) and was		
	planted in 1.6 m spaced rows (Figure 1a). The plantation yielded 25 ton/ha-year of dry		
	biomass. This is a 6 month old experimental trial in Filadelfia, Guanacaste (10°23'35"		
	latitude N and 85°27'49" longitude W) and it was harvested on January of 2013 and it		
	was sampled all stem and leaves. Each plant has 5-6 stems.		
Gynerium	It is a rhizomatous plants belonging to the monopodial group (Figures 1b).		
sagittatum	Commercial plantation for extracting poles for agriculture and construction (Figure		
	1b), about 15,000 plants ha ⁻¹ are produced, which yield about 22 Mg/ha-year of dry		
	biomass. 1-year old, located in Río Frío, Limón (10°18'38" latitude N and 83°52'58"		
	longitude W). It was harvested on February of 2013 and it was sampled all stem and		
	leaves. Each plant has only one stem.		
Pennisetum	It is a rhizomatous plants belonging to the sigmoidal group (Figures 1c). Naturally		
purpureum	growing trial approximately 7 months old (Figure 1c) in Paraiso, Cartago (9°49'44"		
	latitude N and 83°51'6" longitude W). It yielded 31 Mg ha ⁻¹ year dry biomass. It was		
	harvested on February of 2013 and it was sampled all stem and leaves. Each plant has		
	5-6 stems.		
Phyllostachys	It is a rhizomatous plants belonging to the monopodial group (Figures 1d). Crop		
aurea	planted in a small area of the Central Campus of the Costa Rica Institute of		
	Technology in Cartago (9°51'08" Latitude N and 83°54'36" longitude W), about 1 year		
	old (Figure 1d), with 50 individuals/ha and a production of 27.2 Mg ha-1 year of dry		
	biomass. It was harvested on January of 2013 and it was sampled only the stem, but		
	leaves and branch were not sampled. Each plant has only one stem.		
Sorghum bicolor	It is a rhizomatous plants belonging to the monopodial group (Figures 1e). Plantation		
	for experimental purposes and energy production, 151 days old, in Upala, Alajuela		
	(10°53'53" latitude N and 85°0'57" longitude W). This crop is planted at 0.10 m x 0.75		
	m, with a capacity to generate 26.4 Mg ha ⁻¹ year of dry biomass. It was harvested on		
	February of 2013 and it was sampled all stem and leaves. Each plant has only one		
	stem.		

Table 2. Calorific values in five agricultural crops studied in Costa Rica.

	Gross calorific		Net calorific -	Calculated calorific value of pellet	
Agriculture crops	Value Moisture conte (MJ kg ⁻¹) (%)		value (MJ kg ⁻¹)	Value (MJ kg ⁻¹)	Moisture content (%)
Arundo donax	10.3 ^A	60.0	19.2 ^{AB}	17,4	12.0
Gynerium sagittatum	8.6 ^{AB}	57.3	17.1 ^B	15,6	9.9
Pennisetum purpureum	7.0 ^B	81.0	18.5 ^{AB}	16,8	12.1
Phyllostachys aurea	7.3 ^{AB}	43.4	17.9 ^B	16,3	6.7
Sorghum bicolor	2.4 ^c	83.5	20.3 ^A	17,6	12.6

Note: different letters for each calorific value means statistical significances at 95%. And "Gross calorific value" (GCV) is based on the harvest moisture and "Net calorific value" (NCV) is defined based on zero percent in moisture content (MC). Calculate calorific value at moisture content of pellet was calculated by

$${\it Calculated \ caloric \ value=NCG-(\frac{(\textit{GCV-NCV})}{\textit{MC \ of \ harvesting}}*MC \ \ of \ pellet) }$$

Table 3. Moisture content of five agricultural cops at different stages of the pellet production process.

Step	Arundo donax	Gynerium sagittatum	Pennisetum purpureum	Phyllostachys aurea	Sorghum bicolor
Harvesting	56.0 ^B (6.87)	57.3 ^B (11.50)	81.0 ^A (2.93)	43.4° (7.85)	83.5 ^A (0.51)
Air-dry	32.4 ^B (22.41)	-	68.3 ^A (6.82)	75	(a)
Chipped	31.4° (22.41)	57.0 ^B (2.50)	67.9 ^A (1.35)	36.6 ^c (6.71)	
Milled	25.1 ^E (6.96)	49.8° (3.30)	64.1 ^B (1.18)	34.9 ^D (3.81)	81.9 ^A (0.51)
Pressed	-		Z.	87 J	52.4 (2.64)
Dry	12.5 ^B (3.28)	9.7 ^c (6.17)	15.0 ^A (2.22)	7.6 ^D (3.20)	14.6 ^A (10.74)
Pelletized	12.0 ^B (4.70)	9.8° (2.14)	12.1 ^{AB} (3.80)	6.7 ^D (5.85)	12.6 ^A (5.56)

Note: Superscript letters indicate significant differences at at 95% between moisture contents

Table 4. Particle size distribution (percentage) in relation to weight before pelletizing for five agricultural crops studied in Costa Rica.

Particle size in length (mm)	Arundo donax	Gynerium sagittatum	Pennisetum purpureum	Phyllostachys aurea	Sorghum bicolor
> 16.00	0.33	0.68	1.57	0.00	10.29
16.00-11.20	0.98	0.09	3.08	0.09	2.24
11.20-8.00	5.12	1.81	3.28	0.88	3.71
8.00-6.70	3.49	3.10	1.46	0.93	2.38
6.70-4.00	34.28	21.17	22.62	12.40	18.34
4.00-2.00	24.81	33.49	21.90	32.93	26.12
2.00-1.00	15.17	12.82	16.75	18.51	15.85
< 1.00	15.81	26.83	29.34	34.26	21.06

Table 5. Parameters characteristics of light zones in surface pellet in five agricultural crops in Costa Rica.

Agricultural crops	Craks/mm	Light areas/mm ²	Area of light (%)
Arundo donax	3.0 ^B	12.97 ^A	3.43 ^A
Phyllostachys aurea	6.1 ^A	1.43 ^D	1.03 ^c
Gynerium sagittatum	5.5 ^A	2.77 ^c	1.34 ^B
Pennisetum purpureum	0.9°	5.28 ^B	1.46 ^B
Sorghum bicolor	2.5 ^B	1.30 ^D	0.27 ^D

Note: different letters for each parameter means statistical differences significant at 95%.

Table 6. Transversal and surface pellet density obtained by X-ray densitometry in transversal and length direction in five agricultural crops in Costa Rica.

	Surface	pellet density	Transversal density		
Agricultural crops	Average (kg m ⁻³)	Coefficient of variation (%)	Average (kg m ⁻³)	Coefficient of variation (%)	
Arundo donax	1291.9 ^A	9.7 ^A	1292.0 ^A	15.3 ^A	
Gynerium sagittatum	1225.1 ^B	6.3 ^{AB}	1225.1 ^B	10.4 ^B	
Pennisetum purpureum	1293.9 ^A	6.2 ^{AB}	1293.9 ^A	16.1 ^A	
Phyllostachys aurea	1169.6 ^{BC}	7.4 ^{AB}	1169.6 ^{BC}	9.9 ^B	
Sorghum bicolor	1129.1 ^c	5.3 ^B	1129.1 ^c	13.4 ^{AB}	

Note: different letters for each parameter means statistical differences, significant at 95%.

Figure



Figure 1 Agricultural crops with energetic potential in Costa Rica. a) Arundo donax b) Gynerium sagittatum c) Pennisetum purpureum d) Phyllostachys aurea e) Sorghum bicolor

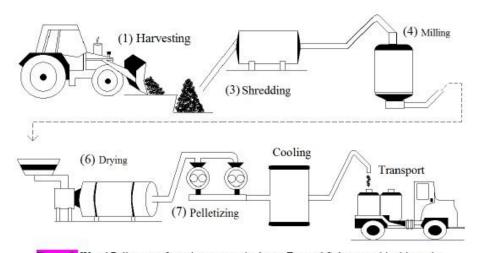


Figure 2: Wood Pellet manufacturing process in Agrep Forestal S.A. as used in this study.

Note: the number in parenthesis represents the stage of process according to table 3.

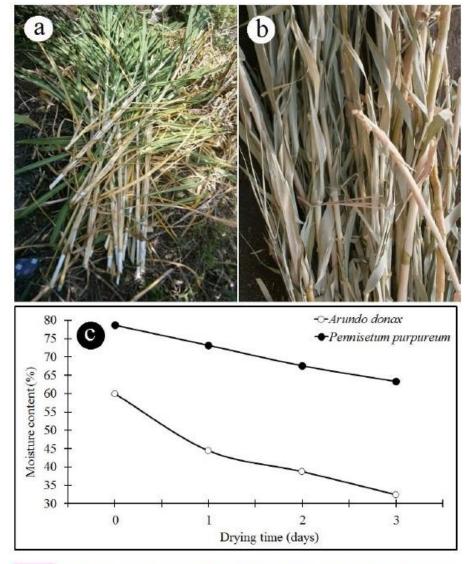


Figure 3 Stem of Pennisetum purpureum left in the field after harvest (a), Arundo donax air-dried after three day (b) and moisture content reduction in P. purpureum and A. donax in air-drying for three day.

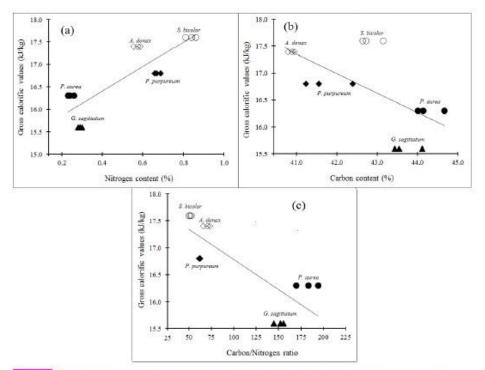


Figure 4. Relationships between carbon, nitrogen and carbon/nitrogen ratio with gross caloric values for pellets manufactured with five energy crops.

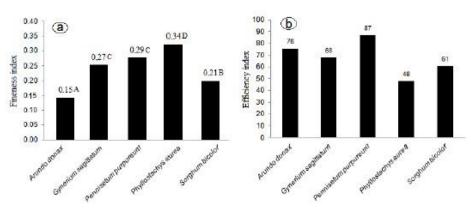


Figure 5. Values obtained for five energy crops in (a) fineness index and (b) efficiency index. Note: different letters for each value means statistical significances at 95%.

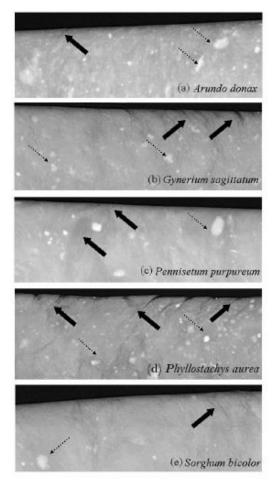


Figure 6. X-ray photograph of the surface of the pellets manufactured with five energy crops.

Note: The mark indicates the presence of cracks in the surface and the mark indicates a light colored area.

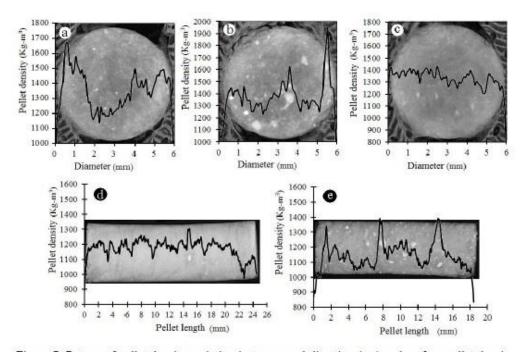


Figure 7. Pattern of pellet density variation in transversal direction (a-c) and surface pellet density variation (d-e). Samples of pellets of (a) Arundo donax (b) Arundo donax (c) Pennisetum purpureum (d) Sorghum bicolor (e) Phyllostachys aurea

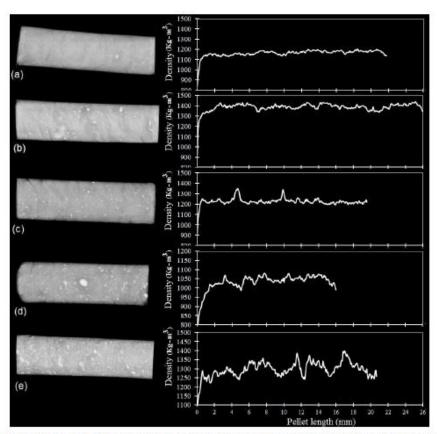


Figure 8. Surface pellet density variation for (a) Sorghum bicolor (b) Pennisetum purpureum (c) Gynerium sagittatum (d) Phyllostachys aurea (e) Arundo donax

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Abstract: Pellet production needs calibration and adaption depending on the raw material. This work presents the production calibration for pelletizing five energy crops in a wood pellet manufacturing plant in Costa Rica. The evaluated aspects include: the caloric value, the loss of moisture content in each production stage and the convertion ratio of particle-pellet, quality, tranversal density and surface density of the pellets through X-ray radiography. The results showed that the analyzed crops were rhizomatous plants, with caloric values ranging between 17.1 and 20.3 MJ kg-1. It was possible to produce pellets with Gynerium sagittatum and Phyllosstachys aurea with the same production process used for wood. However, Arundo donax and Pennisetum purpureum needed pre-air-drying and to the Sorghum bicolor was a mechanical dewatering stage applied before the drying, A. donax, P. purpureum and G. sagittatum showed the highest convertion ratio of particle-pellet with 87, 76 and 68%, respectively. In the quality evaluation, P. aurea and G. sagittatum were observed to have a large quantity of cracks, unlike A. donax, P. purpureum and Sorghum bicolor. The tranversal and surface pellet density varied from 1129 to 1294 kg m-3. The highest values of bulk density were obtained in A. donax and P. purpureum, followed by G. sagittatum and P. aurea, and the lowest bulk density was obtained in S. bicolor

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