

Article

Chemical and Energetic Evaluation of Densified Biomass of *Quercus laurina* and *Quercus rugosa* for Bioenergy Production

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Abstract: Fuels obtained from woody forest resources such as oaks have been traditionally used in various regions due to their availability and energy properties. In the search for sustainable bioenergy sources and the transition towards cleaner alternatives, biomass-derived fuels, such as charcoal and pellets, represent a relevant option for rural and urban communities. This study determines the chemical composition, physical and mechanical properties, and energy quality of pellets from two oak species (*Quercus laurina* and *Q. rugosa*) in San Sebastián Coatlán, Miahuatlán, Oaxaca. The chemical composition was determined in an Ankom fiber analyzer; the energetic, physical, and mechanical analysis was carried out with UNE-EN ISO and ASTM standards. On average, 56.18% and 54.63% cellulose, 17.81% and 17.87% lignin, and 13.96% and 13.78% hemicelluloses were obtained for *Quercus laurina* and *Q. rugosa*, respectively. Mechanical durability ranged from 87% to 95% for *Q. laurina* stump and *Q. rugosa* stem, respectively; for calorific value, values from 19.79 MJ Kg⁻¹ to 20.31 MJ Kg⁻¹ were recorded for *Q. laurina* stem and *Q. rugosa* stump, respectively. The forest biomass of both oak species is viable for pellet production.

Keywords: densified fuels; wood chemistry; calorific value; proximate analysis; pellets



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1. Introduction

The use of petroleum-based fuels poses several environmental challenges, such as global warming, a term that refers to the impact of human activities on the climate, particularly the burning of fossil fuels such as coal, oil and gas, together with deforestation, which emit large amounts of greenhouse gases such as carbon dioxide into the atmosphere. Therefore, the need to substitute fossil fuels with clean and renewable energy sources, such as forest biomass for bioenergy purposes, has increased [1–3].

Oaks are an example of forest biomass, and several species of oaks have been underutilized due to the lack of biological information regarding their distribution and abundance; in addition, there is a shortage of technical data for this genus, including market aspects, productivity and appropriate technology for processing; one of the ways to efficiently

exploit residual forest biomass is the production of pellets or briquettes; however, not all species are suitable for pelletization given their anatomical, chemical, and physical characteristics [4–6].

In terms of species diversity, ecological dominance and economic value, the *Quercus* genus is one of the most important in the northern hemisphere, dominating a wide variety of habitats [7]. Recent studies indicate that the diversity of oak trees in the world is around 435 species [8], of which 241 species are distributed in the Americas, with Mexico being the country with the largest distribution with 174 species [9]. The state of Oaxaca has the highest diversity of oaks in Mexico, with 48 species [10].

Locally, *Quercus laurina* and *Q. rugosa* are used as firewood, so they play an important role as a source of energy. Oak wood has high volumetric shrinkage, and this is an indicator of lower dimensional stability, so greater care is needed in the drying process of the wood, as there is a greater tendency to the presence of cracking and splitting in the material; however, since they have high hardness, they can also be used in the manufacture of heavy traffic flooring, moldings, boards, and furniture. In addition, oak wood has shown good performance in the transformation of firewood to charcoal [11–13].

Worldwide, annual cellulosic biomass production is estimated at 181.5 billion tons [14]. Particularly in Mexico, the annual wood production was 8.3 million m³ in 2018, of which 14.5% corresponds to oak wood [15].

Cellulosic biomass is an alternative for use as biofuel in the form of bioethanol, biodiesel, densified fuels, and chemicals. Different thermal routes are followed for the conversion of biomass into biofuels, for example, thermal conversion by combustion, thermochemical conversion by gasification and pyrolysis, and biological conversion using biocatalysts [16–18].

The chemical composition of wood is important to optimize its use, such as in biofuel, and is directly related to the calorific value, that is, the amount of energy it can release; in addition, for the production of pellets, this chemical composition plays a fundamental role in deciding whether or not to use additives, since lignin acts as a binder at high temperatures, which causes the lignin to soften and can bind particles [19–21].

Biomass pelletization can become a low-carbon renewable energy source that can challenge the use of fossil fuels and replace them in specific economic sectors, offering several advantages such as allowing year-round storage which ensures constant product availability, offering the opportunity to add value to biomass that is not used in the forest industry, and helping to reduce forest fires and pests in forests, as well as increasing the calorific value of biomass [22].

The current market demands high-quality pellets, which are classified into two main categories: residential and industrial pellets. The former are intended for small-scale combustion systems, while the latter are used in larger applications. To ensure their quality, there are specific standards, including European standards, which divide pellets into three classes: A1, A2 and B. Classes A1 and A2 correspond to residential pellets, which are characterized by stricter standards. Class B pellets, on the other hand, are intended for industrial use and have wider tolerances in terms of diameter, ash content, nitrogen, sulfur, chlorine and net calorific value [23].

Pelletization offers multiple benefits, including facilitating year-round storage, ensuring the continuous availability of the resource, adding value to biomass residues not used by the forest industry, promoting employment, diversifying income, improving the quality of life, especially in rural communities, contributing to the prevention of forest fires and pests, and improving the calorific value per unit volume of biomass. To ensure its sustainability, it is essential to provide training in the production, use and maintenance of

pellets, as well as to implement community policies and projects that include incentives, subsidies and government support [22,24,25].

Several studies have focused specifically on the manufacturing process of densified fuels and their physicochemical characterization: Ruiz-García et al. [26] describe the manufacture and characterization of pellets obtained from the pruning of guava trees, indicating good potential for their use as fuel; Soria-González [27] determined the potential energy use of avocado pruning by-products as densified solid biofuels and suggested their use in the residential, commercial and industrial sectors at the regional level; Ramírez-Ramírez et al. [28] made briquettes from hardwood sawdust without using adhesive, and indicate that this lignocellulosic residue can be used for densification at the industrial level.

In this context, the objective of the present study was to determine the chemical composition of the biomass of the stump, stem, and branches of two oak species and evaluate the energy properties of the pellets for bioenergy production.

2. Materials and Methods

2.1. Tree Selection and Sample Preparation

In the municipality of San Sebastián Coatlán, Miahuatlán, Oaxaca, Mexico (15°57' and 16°15' N., 96°48' and 96°58' W.) [29], samples were collected from two oak species: *Quercus laurina* Humb & Bonpl and *Quercus rugosa* Née, which according to Jiménez-Mendoza et al. [12], are two of the main species used as fuel in this area. Two trees of each species were felled and sectioned into three parts: stump, stem, and branches, from which 2 cm long slices were extracted. The slices were chipped using a band saw (5C-14, CELA, Puebla, México) and a hand tool. The chips were then ground in a conventional Micron equipment (K20F, Micron Mixer, México City, México) to obtain ground wood. In a vibratory sifter (RX-29, ROTAP, Mentor, OH, USA), the wood was sieved in mesh sizes from 6 (3.35 mm) to 60 (0.250 mm). For chemical analysis, wood from the 40-mesh sieve (425 µm) was used, according to ASTM D 1105-96 [30], and the rest of the material was used for pelletizing.

2.2. Chemical Analysis of Wood

The chemical analysis of the samples was carried out on a fiber analyzer (A200, ANKOM, Macedon, NY, USA); this equipment automates the chemical digestion process of the samples under controlled conditions, including agitation, temperature, and time, replacing the traditional Gooch crucible method, reducing measurement errors. This analysis was performed once [26,31], and to determine the content of inorganic substances, ground wood was used in a 50 mL crucible in a muffle at 580 °C for 6 h [32].

2.3. Pellets Processing and Analysis

Pellets were made from each of the three parts of the oak (stump, stem, and branches); the biomass was dried under shade until it reached a constant weight, and the moisture content of the biomass was determined on a thermobalance (PMB53, Adam Equipment, Oxford, CT, USA). The material was introduced into a ZSLP-R300 industrial flat disk pelletizer without using adhesive; the pelletizer consisted of a flat disk with dies of 8 mm length and 6 mm diameter [33], and it produces pellets with average lengths of 20 mm and 6 mm diameters. The particle density was determined using EN 18847 [34] and the bulk density of the pellets with EN 17828 [35]; the length and diameter were measured with a electronic vernier (HER-414, STEREN, CDMX, México City, México) [36] and the mass of each pellet was obtained by weighing 3 pellets on an analytical balance (PW 254, Adam Equipment, Oxford, CT, USA). The mechanical durability test was performed under EN 17831-1 [37]. The moisture content, volatile matter, ash content, fixed carbon content, and

upper and lower calorific value were calculated according to ASTM standards [32,38–41], respectively. An outline of the methodology is shown in Figure 1.



Figure 1. Pellet processing and evaluation.

2.4. Statistical Analysis

Data analysis was performed in triplicate for each experimental unit, evaluating the physical, mechanical, and energetic variables of the pellets. A completely randomized design was used to evaluate if significant differences exist, and Tukey's multiple comparisons test was used with a significance level of $\alpha = 0.05$ in the statistical package SAS[®] version 9.0 [42,43].

3. Results and Discussion

3.1. Chemical Analysis

The results of the chemical composition are shown in Table 1. A value of 12.90% hemicelluloses was recorded for the *Q. laurina* stump, of 14.27% for the stem, and of 14.72% for the branches, values lower than those reported by Herrera-Fernández et al. [44], who reported 33.27% for the heartwood and 23.51% for the sapwood of the same species. As mentioned by Honorato-Salazar et al. [45], these differences can be attributed to the solvent mixture used in the extraction.

Table 1. Chemical composition of two *Quercus* species (%).

Species	Tree Parts	Hemicellulose	Cellulose	Lignin	Extractable Substances
<i>Q. laurina</i>	Stump	12.90	55.74	18.97	10.29
	Stem	14.27	56.29	18.66	8.45
	Branches	14.72	56.52	15.79	10.76
<i>Q. rugosa</i>	Stump	13.67	56.39	14.98	13.00
	Stem	13.84	52.27	21.58	10.31
	Branches	13.84	55.24	17.05	11.84

Quercus rugosa had a value of 13% for the three parts of the tree; however, Ruiz [46] mentions that there are different proportions of hemicelluloses in the trunk, branches, roots and bark of the same tree, and hemicelluloses play a fundamental role in the production of pulp for paper making, as it increases its yield and resistance.

Regarding cellulose, a value of 56% was recorded for the stem and branches of *Quercus laurina* (Table 1). Honorato and Hernández [4] obtained the same value (56%) for the

sapwood and heartwood of this species. *Q. rugosa* had values of 52.7%, 55.24%, and 56.39% for the stem, branches, and stump, respectively, while Bautista and Honorato [47] reported a value of 52.9% for the heartwood and sapwood mixture of this species. In this work, the results indicate that the cellulose content exceeded 50%, which suggests that it can be used for paper production; however, pulping and paper physics tests are necessary, particularly since oak wood has short fibers and could negatively influence certain physical and mechanical characteristics of the paper [4].

For lignin, *Q. laurina* presented a value of 18% for both the stump and the trunk (Table 1). These data are within the ranges previously reported by various authors for this species, which range from 14.67% to 25.5% [4,44,48]. *Q. rugosa* presented values of 14.98%, 17.05%, and 21.58% for the stump, branches and stem, respectively, and similar values were found for the sapwood of the same species, with 18.84%, for the heartwood with 16.16% and the bark with 22.83%. It should be noted that the bark has a higher concentration of lignin than the wood [44,49].

The lignin content is positively related to the calorific value; additionally, this chemical component has a high compressive strength and allows the wood to change shape at high temperatures due to its thermoplastic nature; therefore, a high lignin content in biomass is a key factor for the production of densified biofuels. On the other hand, biomass with low amounts of lignin is more suitable for the production of cellulosic pulp [49–51].

For extractable substances, *Q. rugosa* presented the maximum value with 13.00% in the stump part (Table 1), a value higher than the 10.2% reported by Rutiaga-Quiñones et al. [50] for *Q. candicans* stem. The extractable substances have a positive influence on the calorific value since biomass with a high content of extracts has a higher calorific value, in addition to having an indirect impact on the mechanical properties of the wood since it affects the basic density [48,52].

Quercus laurina presented the highest value of ash with 1.94% for the stem part, and *Quercus rugosa* in the stump part presented the lowest value with 1.59%. Vega-Nieva et al. [53] mention that ash can cause problems during combustion in boilers, while Solla-Gullón et al. [54] argue that, depending on its composition, it can act as a nutrient for the soil, helping to reduce the degree of acidity. With ash and nitrogen fertilization, the level of nutrients is improved.

3.2. Physical Characteristics of Pellets

Before the pelletizing process, *Quercus rugosa* stump presented the highest initial moisture content with 10.40%, and *Q. laurina* branches presented the lowest initial moisture content with 9.06% (Table 2). These results fall within the range of 8% to 15% biomass moisture content, ideal for pelletizing [55]. Statistically, *Q. laurina* branches and *Q. rugosa* stem are as different as *Q. rugosa* stump and *Q. laurina* branches (Table 3). The moisture content of forest biomass is a key property regarding its potential as an energy source, specifically in the production of pellets, as it is directly linked to calorific value, transport, and storage [56,57].

Table 2. Initial moisture content, bulk density, and particle density.

Species	Tree Parts	Moisture Content (%)	Bulk Density (Kg m ^{−3})	Particle Density (g cm ^{−3})	n
<i>Q. laurina</i>	Stump	9.88 (0.45) a, b	616.61 (2.78) a	1.27 (0.01) a	3
	Stem	9.78 (0.13) a, b	595.61 (8.37) b	1.26 (0.01) a	3
	Branches	9.06 (0.06) b	558.33 (8.02) c	1.18 (0.10) a	3

Table 2. Cont.

Species	Tree Parts	Moisture Content (%)	Bulk Density (Kg m ⁻³)	Particle Density (g cm ⁻³)	n
<i>Q. rugosa</i>	Stump	10.40 (0.28) a	593.61 (3.31) b	1.22 (0.05) a	3
	Stem	10.34 (0.43) a	566.94 (8.99) c	1.23 (0.03) a	3
	Branches	9.93 (0.46) a, b	584.50 (1.30) b	1.35 (0.21) a	3

n: number of replicates; values in parentheses represent standard deviation. Equal letters in column directions indicate statistical equality ($p \geq 0.05$). Moisture content (%) refers to the material before pelletizing.

Table 3. Mechanical durability of pellets from two Quercus species.

Species	Tree Parts	Durability (%)	n
<i>Q. laurina</i>	Stump	87.88 (1.50) d	3
	Stem	90.07 (0.56) c, d	3
	Branches	91.71 (2.57) b, c	3
<i>Q. rugosa</i>	Stump	95.26 (0.21) a, b	3
	Stem	95.96 (0.60) a	3
	Branches	91.55 (1.10) c, d	3

n: number of replicates; values in parentheses represent standard deviation. Equal letters in column directions indicate statistical equality ($p \geq 0.05$).

For bulk density, statistical differences were found among tree parts (Table 2), except for *Q. rugosa* branches and stump. *Q. rugosa* presented a bulk density of 593 kg m³, 566 kg m³, and 584 kg m³ for the stump, stem, and branches, respectively, and these values are within the bulk density range (557–703 kg m⁻³) reported by Núñez-Retana et al. for four species of oaks [33]. Pellets made from *Q. laurina* stump are within category A1 of the UNE-EN 14961-2 standard [58], as they have a bulk density greater than 600 kg m³. This variable influences the commercial value of the pellets, since at high densities, there is a reduction in the transportation, handling, and processing costs of the fuel, in addition to reducing the space required for storage [59].

Regarding the particle density of the pellets, statistically, there is no difference between tree parts and species (Table 2); this varied from 1.18 to 1.35 g cm³ for *Q. laurina* branches and *Q. rugosa* branches, respectively, data similar to those found by Núñez-Retana et al. [6] for the particle density of four Quercus species. Particle density influences the bulk density and combustion process of densified fuels [60].

3.3. Mechanical Durability of Pellets

The mechanical durability ranged from 87.88% to 95.96% (Table 3) for the *Q. laurina* stump and the *Q. rugosa* stem, respectively. According to the durability classification performed by Adapa et al. [61], the pellets of these two species present high durability because they exceed 80% hardness; however, according to the UNE-EN 14961-2 standard [58], pellets with a durability greater than 96% are considered good quality; under this criterion, only the stump and trunk pellets of *Q. rugosa* are considered good quality, as they showed values slightly lower than 96%. An alternative to improve the durability of oak pellets is to add coniferous sawdust and use high-performance pelletizers [6,62]. Statistically, there is a significant difference between the pellets of both species (Table 3).

3.4. Energy Properties of Pellets

3.4.1. Moisture Content

For moisture content, statistically *Q. rugosa* is the species that presents equality in the three parts of the tree, unlike *Q. laurina* (Table 4). According to UNE-EN 14961-2 [58], the two pellet species with their respective tree parts fall into category A1, since they have a moisture content of less than 10%. This variable is important as it influences the storage

of the pellets. The pellets have a low moisture content, unlike the unprocessed biomass, which limits the growth of microorganisms [63].

Table 4. Energy properties of pellets from two species of *Quercus*.

	Tree Parts	Moisture Content (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)	High Heating Value (MJ Kg ⁻¹)	Low Heating Value (MJ Kg ⁻¹)	n
<i>Q. laurina</i>	Stump	5.02 (0.02) b	83.30 (0.63) a	1.76 (0.08) a	14.92 (0.58) a	19.90 (0.13) a	18.77 (0.12) a	3
	Stem	5.32 (0.08) b	81.19 (0.08) a	1.94 (0.05) a	16.85 (0.12) a	19.79 (0.33) a	18.60 (0.30) a	3
	Branches	6.09 (0.14) a	82.48 (2.68) a	1.79 (0.26) a	15.72 (2.64) a	20.28 (0.54) a	18.88 (0.50) a	3
<i>Q. rugosa</i>	Stump	5.00 (0.09) b	82.50 (1.46) a	1.59 (0.06) a	15.90 (1.40) a	20.31 (0.31) a	19.16 (0.30) a	3
	Stem	5.34 (0.14) b	83.68 (0.33) a	1.79 (0.02) a	14.51 (0.34) a	20.09 (0.05) a	18.87 (0.08) a	3
	Branches	5.22 (0.19) b	83.21 (1.17) a	1.62 (0.12) a	15.15 (1.18) a	19.98 (0.09) a	18.79 (0.09) a	3

n: number of replicates; values in parentheses represent standard deviation. Equal letters in column directions indicate statistical equality ($p \geq 0.05$).

3.4.2. Volatile Matter

The species with the highest volatile content was *Q. rugosa* in the stem with 83.68%, while the species with the lowest value was *Q. laurina* in the stem with 81.19% (Table 4). Statistically, there is equality in the two species with their respective compartments (Table 4). Volatile matter is the loss of mass that occurs when heating biomass; the combustion of these volatiles produces a bright flame, whose color and temperature are determined by the chemical composition of the wood [64].

3.4.3. Ash Content

The ash content ranged from 1.59% to 1.94% (Table 4) for the stump of *Q. rugosa* and the stem of *Q. laurina*, respectively; values reported by Gutiérrez-Acosta et al. [17] range within percentages of 1% to 3.1% for the biomass of two species of *Quercus* spp. The statistical analysis indicates that the ash content within the tree parts and species is equal (Table 4). According to the UNE-EN 14961-2 standard [58], the three parts of the *Q. laurina* tree as well as the trunk and branches of *Q. rugosa* fall into category B, since they have an ash content of less than 3.5%; only the stump of *Quercus rugosa* falls into category A2 since it has an ash content equal to 1.5%. Vega-Nieva et al. [53] mention that ash can cause problems during combustion in boilers, while Solla-Gullón et al. [54] argue that, depending on its composition, it can act as a nutrient for the soil, helping to reduce the degree of acidity, and that the level of nutrients is improved with ash and nitrogen fertilization.

3.4.4. Fixed Carbon

The fixed carbon of pellets ranged from 14.51% to 16.85% (Table 4) for *Q. rugosa* stem and *Q. laurina* stem, respectively, with higher values than for four oak species ranging from 6.65% to 8.88% [6] but values like those reported by Rutiaga-Quiñones et al. [16] for the biomass of two species of *Quercus* spp. According to the statistical analysis, this indicates that there is no significant difference between the means by parts of the tree or by species (Table 4). The fixed carbon content is positively related to the density and calorific value, making biomass with a high carbon content ideal for combustion processes [64,65].

3.4.5. High Heating Value

For the calorific value, the *Q. laurina* stem had the lowest value with 19.79 MJ kg⁻¹, and the *Q. rugosa* stump had the highest value with 20.31 MJ kg⁻¹ (Table 4), like that found by Ramírez-Ramírez et al. [28] for briquettes of the genus *Quercus* spp. with values from 19.91 MJ kg⁻¹ to 20.10 MJ kg⁻¹. Statistically, there is no difference in tree parts or species (Table 4). The calorific value is the amount of energy per unit weighed; therefore, *Q. laurina* and *Q. rugosa* in the stump, stem, and branches are suitable for use as densified biofuels [64].

3.4.6. Low Heating Value

The lower calorific value for the two species was 18 MJ kg^{-1} , except for the *Q. rugosa* stump, which was 19 MJ kg^{-1} . The three tree parts and the two species have statistical equality in the means (Table 4). The lower calorific value is the net energy released per unit mass after complete combustion [65]. According to UNE-EN 14961-2 [58], the three tree parts of the two species fall into category A1 since they are within the range of 16.5 to 19 MJ kg^{-1} .

4. Conclusions

The initial moisture content of the three parts of the two oak tree species is within the range of 8% to 15%, so the biomass is suitable for pellet production.

The *Q. rugosa* stump pellets showed the highest mechanical durability with 95.26%, the highest fixed carbon with 15.90%, and the highest calorific value with 20.31 MJ kg^{-1} ; these parameters are important to consider when prospecting stump biomass as a raw material to produce pellets; however, it is possible to improve the pelletizing process to increase mechanical durability.

According to the UNE-EN 14961-2 standard, considering the lower calorific value and moisture content of the pellets, the two species, with their respective compartments, fall into category A1, i.e., high-quality pellets. It should be noted that to improve the durability of pellets with this species, it is possible to mix them with coniferous wood or use binding additives to obtain a higher percentage of this parameter.

The biomass of the stem of *Q. rugosa* presented a higher lignin content, and the pellets presented higher durability, higher upper and lower calorific values, and lower moisture and volatile matter contents; therefore, the stem part of *Q. rugosa* is the optimum to produce densified biofuels.

It is important to mention that, despite the available information on densified biofuels from the *Quercus* genus, a significant energy transition has not yet been consolidated in Mexico. Therefore, it is essential to analyze the social, political, regulatory, and ecological challenges involved in replacing fossil fuels with forest biomass-based energy sources.

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