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Analysis of chemical composition and heating value of five oak species for their use as biofuels

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ABSTRACT

Objective: To determine the chemical composition and calorific value of biomass from the stump, stem and branches of *Quercus calophylla*, *Q. glaucoides*, *Q. laurina*, *Q. magnoliifolia* and *Q. rugosa*.

Design/methodology/approach: The amount of hemicellulose, cellulose, lignin, and extractable substances was determined in an Ankom fiber analyzer. Pearson's correlation analysis was conducted between the chemical components, and the higher heating value was also calculated.

Results: The highest values obtained were hemicellulose 14.72% in the branches of *Q. laurina*; cellulose 67.19% in the stem of *Q. calophylla*; lignin 21.58% in the stem of *Q. rugosa*; extractable substances 13.00% in the stump of *Q. rugosa*. In particular, the correlation between cellulose and hemicellulose was negative ($r = -0.80$). The results for calorific value varied from 19.32 MJ kg^{-1} in the stump of *Q. glaucoides* to 20.19 MJ kg^{-1} in the stem of *Q. rugosa*.

Limitations on study/implications: The shortage of studies about the chemical composition of wood affects the selection of species for their application in the area of biofuels, and a poor selection of raw material translates into inefficient combustion and greater environmental impact.

Findings/conclusions: The species studied are apt for their use as thickened biofuel, given their chemical characteristics. *Q. rugosa* presents a higher percentage of lignin content, extractable substances in the stem and the stump, and high heating value, so it is considered to have greater potential for the elaboration of quality pellets.

Keywords: Dendroenergy, lignin, extractable substances, *Quercus*, higher heating value .

INTRODUCTION

The *Quercus* genus, with approximately 500 species, belongs to the Fagaceae family (Manos *et al.*, 1999); because of the number of species of this genus, Mexico is considered a diversity center (Valencia, 2004). It is a source of raw material for fuels in timber form and

to produce carbon (Flores and García, 2021); it is also used in permanent constructions where its mechanical resistance is evident. Due to its natural durability, it is used in applications for exteriors, tool handles, fence posts, mine piles, among others (de la Paz-Pérez and Dávalos Sotelo, 2008), as well as for pulp and paper, tannin extraction, adhesive elaboration, thickened biofuel generation, and various additional applications (Honorato and Hernández, 1998).

The applications towards which wood is destined are in function of its chemical composition, because wood is an organic material composed of carbon, hydrogen, oxygen, nitrogen, and other mineral elements; it is heterogeneous because it has cells with different functions; it is hygroscopic from its ability to exchange moisture with the environment; it is porous, and in addition it is an anisotropic material (Suirezs and Berger, 2010). Chemically, it is divided into substances, macromolecular and low molecular weight. In the macromolecular components, there are polysaccharides which in turn are classified into cellulose and hemicellulose, in addition to lignin being present; on the other hand, the substances of low molecular weight are classified into organic and inorganic matter (Fengel and Wegner, 2003).

The parts of a tree such as stem, bark, roots, crown, branches and foliage differ significantly in their chemical composition and amount of water. Consequently, it is important to study the different properties and characteristics of wood to compare them and determine their potential use, as well as their different attributes, to give place to new applications, carrying out a better use, and obtaining added value (Howard, 1973; Suirezs and Berger, 2010).

In the elaboration of thickened biofuels such as pellets and briquettes, the chemical composition of the biomass is an important factor, since it has a close relationship with the amount of energy that these can liberate. Therefore, the chemical composition of the biomass is an important parameter to consider decision making to use some type of additive. For example, lignin present in the biomass acts as an agglutinant at high temperatures, which causes lignin to soften and helps the union of particles (Kaliyan and Vance, 2009; Fearon *et al.*, 2020); therefore, no additional additive is generally used for the thickening process. In communities of the state of Oaxaca, Mexico, wood from species of the *Quercus* genus is widely used as fuel. However, there is a shortage of information related to the chemical composition of wood and its higher heating value, so the objective of this study was to analyze the chemical composition of the stump, stem and branches of *Quercus calophylla*, *Q. glaucoides*, *Q. laurina*, *Q. magnoliifolia* and *Q. rugosa*, with the aim of assessing the wood's potential in its use as biofuel.

MATERIALS AND METHODS

Study area

The study was carried out in the municipality of San Sebastián Coatlán, Miahuatlán, Oaxaca, Mexico, located on the South Sierra of the state (15° 57' and 16° 15' LN, 96° 48' and 96° 58' LW) (Figure 1) with a total of 19,030 ha, at an altitude between 100 and 2300 masl. Regarding the vegetation, 74.73% corresponds to wooded surface, 11.88% is grassland, and 6.47% rain forest (Monjaraz, 2013; INEGI, 2015; INEGI, 2020).

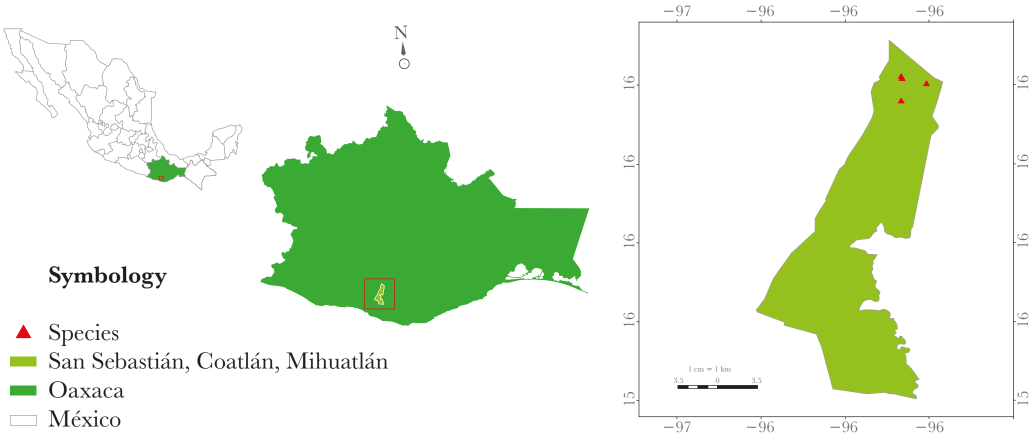

Figure 1. Study area (prepared by the authors).

Tree selection

Samples from five tree species were collected: *Quercus calophylla* Schltdl. & Cham (encino de raja), *Q. glaucoides* M. Martens & Galeotti (encino negro), *Q. laurina* Humb & Bonpl (encino blanco), *Q. magnoliifolia* Née (encino yegareche), and *Q. rugosa* Née (encino cucharilla), which, according to Jiménez-Mendoza *et al.* (2023), are the main species that are used for fuel in the study region.

Sample preparation

The trees selected were sectioned into three parts: stump, stem and branches, from which 5 cm-long slices were extracted. The slices were splintered with a manual tool. Then, the splinters were ground in a conventional Micron Mixer®, K20F mill, and finally sieved (vibratory sieve RO-TAP® Model RX-29), collecting samples with particle size 425 μm (Figure 2).

Chemical analysis of the samples

The samples were evaluated in an Ankom® Model A200 fiber analyzer (Van Soest *et al.*, 1991). To determine the content of hemicellulose, cellulose, lignin and extractables, wood

Figure 2. Classification and sampling (prepared by the authors).

samples were encapsuled in a filter bag, and the capsules were placed in a suspension tray and put into the fiber analyzer recipient. The fiber analyzer began the process and filtered the samples. When this phase finished, the samples were removed from the analyzer to be dried and weighed, with the purpose of determining the percentage of components of the wood. The content of inorganic substances was determined based on the norm ASTM D 1102-84 (ASTM, 2007).

Higher heating value The higher heating value of the samples was determined according to White (1987), taking as independent variables the content of lignin and the content of extractable substances.

Statistical analysis

Pearson's correlation was conducted with the following variables: hemicellulose, cellulose, lignin, extractable substances, and ashes; for this purpose, the statistical software SAS® version 9.0 was used (SAS Institute Inc., 2014).

RESULTS AND DISCUSSION

Hemicellulose content

Quercus laurina presented the highest value of hemicelluloses in its branches (14.72%) and stem (14.27%) (Table 1). These values are lower than what was reported by Herrera-Fernández *et al.* (2017) for this species, with 33.27% for heartwood, 23.51% for sapwood, and 22.72% for bark. Next, the percentage of hemicelluloses in the stem and branches of *Q. rugosa* was 13.84%, similar datum to the value found by Ruiz-Aquino *et al.* (2019) for three broad-leaved species from the forest in Ixtlán de Juárez, Oaxaca, Mexico.

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

Species	Component	Hemicellulose	Cellulose	Lignina	Extractable substances	Ash
Q. calophylla	Stump	10.46	63.54	17.60	6.68	1.38
	Stem	9.52	67.19	15.05	6.64	1.28
	Branches	11.34	62.86	15.75	8.38	1.40
Q. glaucoides	Stump	12.26	64.86	10.95	9.15	2.61
	Stem	10.92	62.69	14.02	9.57	2.49
	Branches	10.39	59.39	16.73	10.75	2.41
Q. laurina	Stump	12.90	55.74	18.97	10.29	1.76
	Stem	14.27	56.29	18.66	8.45	1.94
	Branches	14.72	56.52	15.79	10.76	1.79
Q. magnoliifolia	Stump	11.16	64.02	12.52	10.60	1.38
	Stem	12.36	62.89	13.26	9.89	1.33
	Branches	12.54	60.73	14.80	9.46	2.17
Q. rugosa	Stump	13.67	56.39	14.98	13.00	1.59
	Stem	13.84	52.27	21.58	10.31	1.79
	Branches	13.84	55.24	17.05	11.84	1.62

The species that presented the lowest percentage of hemicellulose was *Q. calophylla* in stem, with a value of 9.52%, lower value than what was reported for *Q. candicans* in heartwood with 27.90%, in sapwood with 21.90%, and in bark with 22.04% (Herrera-Fernández *et al.*, 2017; Valencia *et al.*, 2018). *Q. glaucoides* had a hemicellulose content in stump of 12.26%, in stem of 10.92%, and in the branches of 10.39%, lower values for three broad-leaved (34.79-41.67%) reported by Honorato-Salazar *et al.* (2015).

The hemicelluloses of broadleaf species exhibit a different complexity compared to conifers; different proportions of hemicelluloses present in the different compartments of the same tree, performing an intermediate role between the cellulose and the lignin, facilitating the insertion of microfibrils (Ruiz, 2018). Agricultural and lignocellulose forest residues can serve as raw materials to produce bioenergy and chemical products from fermentation (Saha, 2003).

Hemicellulose (also known as polyose) is the second most abundant chemical component in woody and herbaceous biomass which, along with cellulose, is found in nearly all plant cell walls (Li *et al.*, 2013). Hemicellulose has a heteropolymer structure (with molecular weight lower than cellulose) made up of various sugar monomers, such as glucose, galactose, mannose, xylose, arabinose, 4-O-methyl glucuronic acid, and galacturonic acid residues (Figure 3). The exact proportion of each of these monomeric units varies considerably depending on the exact nature or origin of the biomass (Garrote *et al.*, 1999).

Cellulose content

Cellulose was found from 52.27% to 67.19% for stem of *Q. rugosa* and stem of *Q. calophylla*, respectively (Table 1); these values are higher than those obtained for various oak species: *Quercus sebifera* with 45.71%, *Q. tinkhami* with 48.37%, and *Q. rubor* with 41.3% (Bárcenas-Pazos *et al.*, 2008; Laskowska *et al.*, 2018). *Q. laurina* presented a cellulose content in stem and branches of 56.29% and 56.52%, respectively, while for stump it was 55.74%. These values were similar to those recorded by Honorato and Hernández (1998) for stem sapwood and heartwood of *Q. laurina* with 56.20%. For stem of *Q. rugosa*, there was a value of 52.27%, for branch a value of 55.24%, and for stump 56.39%; in these two components of the tree (branches and stump), these values are higher than those mentioned by Bautista

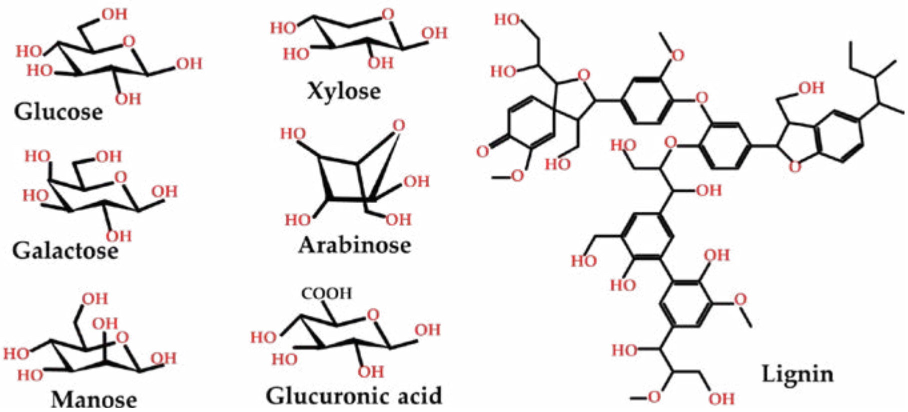

Figure 3. Main monomers of hemicellulose and molecular structure of lignin (Li *et al.*, 2013)

and Honorato (2005) for the mixture of stump heartwood and sapwood of *Q. rugosa* with 52.4%.

The cellulose content in stump of *Q. glaucoides* was 64.86%, followed by *Q. resinosa* with 64.02%, *Q. calophylla* in third place with a value of 63.54%, and *Q. laurina* in fourth place with 55.74%, which were higher values than those for stump of *Quercus oleoides* with 51.68%, *Q. coccolobifolia* with 48.97%, and *Q. durifolia* with 52.82% reported by Bautista and Honorato (2005).

Cellulose is the structural component of the primary cell walls of all the woody and herbaceous biomass and provides resistance and rigidity. Cellulose is of polymeric nature and made up of monomeric units of glucopyranose (Bridgwater, 1994), which at the same time derives from two residues of glucose anhydride united by glycosidic bonds β ($C_1 \rightarrow C_4$) (Figure 4).

Cellulose shows a strong tendency to form intra and inter molecular hydrogen bonds, which induce the formation of microfibrils that are added in highly ordered (crystalline) and less ordered (amorphous) regions. Due to its structure, cellulose is insoluble in most dissolvents and resistant to attack from acids and enzymes, which makes its treatment difficult through non-pyrolytic biomass improvement processes (Sinha *et al.*, 2015).

The variation in results can be related to the use of diverse analytical methods. The values found in this study exceed 50% of cellulose, and therefore, they can be used for paper manufacturing; however, it is necessary to conduct pulping and physical tests of the paper, especially from oak, since its fiber is shorter and could have an adverse effect on some physical and mechanical properties of the paper (Honorato and Hernández, 1998). Cellulose-rich biomass offers many advantages over agricultural raw materials, because of cell elongation, exposure of the secondary cell wall, and programmed cellular death, and with that the large amounts of biomass rich in cellulose can be used to produce bioenergy and biopolymers (Mizrachi *et al.*, 2012).

Lignin content

The lignin content ranged from 10.95% to 21.58% for stump of *Q. glaucoides* and stem of *Q. rugosa*, respectively (Table 1). *Q. laurina* presented 18% of lignin both for stump and stem, the same value (18%) that Gutiérrez-Acosta *et al.* (2021) reported for sawdust from two species of *Quercus* spp., in the state of Durango, and it is also within the range reported by various authors for this species, between 14.67 and 25.5% (Honorato and Hernández, 1998; Ruiz-Aquino *et al.*, 2015; Herrera-Fernández *et al.*, 2017). *Q. calophylla* presented

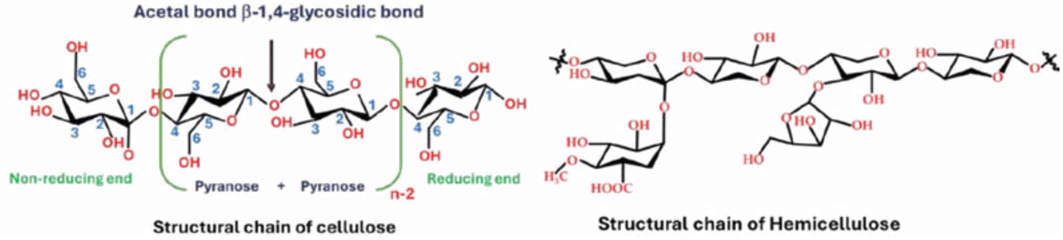

Figure 4. Chemical structure of cellulose and hemicellulose (Bridgwater, 1994).

17.60% of lignin for stump, 15.05% for stem, and 15.75% for branches, values lower than the ones reported by Rutiaga-Quiñones *et al.* (2000) in heartwood of *Q. candicans* with 21.4%. However, Herrera-Fernández *et al.* (2017) reported similar data for *Q. candicans* in heartwood of 16.45%, sapwood of 18.93%, and bark of 29.01%.

The species *Q. rugosa* presented a value of 14.98%, 17.05% and 21.58% for stump, branches and stem, respectively, a similar value to what was found for sapwood of the same species with 14.67%, for heartwood with 16.16% and for bark with 34.67%; it should be highlighted that in bark there is greater concentration of lignin than in wood (Herrera-Fernández *et al.*, 2017; Pintor-Ibarra *et al.*, 2017). *Quercus magnoliifolia* had values of lignin of 12.52% in stump, 13.26% in stem, and 14.80% in branches, lower data for *Q. palustris* with a value of 23.7% (Jaucida *et al.*, 2002).

The lignin content is correlated positively with the calorific value, that is, a high lignin content produces higher calorific value. In addition, this chemical compound has a high resistance to compression, but at the same time it is thermoplastic, which gives wood the ability to change its shape when it is subjected to high temperatures. Therefore, high amounts of lignin in the biomass are an important factor to elaborate quality thickened biofuels; on the other hand, when wood has low amounts of lignin, it can be used to obtain cellulose pulp (Rutiaga-Quiñones *et al.*, 2000; García *et al.*, 2003; Pintor-Ibarra *et al.*, 2017).

Content of extractable substances

The species that presented highest percentage of extractable substances was *Quercus rugosa* in stump with 13.00%, and the species that presented the lowest value was *Quercus calophylla* in stem with 6.64% (Table 1). *Quercus calophylla* was the species that presented the lowest values for the three parts of the tree with values in an interval of 6.64-8.38%; these data are lower than what was obtained by Rutiaga-Quiñones *et al.* (2000), with 10.2 % for stem of *Quercus candicans*.

The stump of *Quercus rugosa* had a value of extractables of 13% (Table 1), which is a similar value to what was reported by Ferreira *et al.* (2018) with 13.20% for bark of *Quercus faginea*. The stump and stem of *Q. glaucoides*, and the stem and branches of *Q. magnoliifolia* presented a value of 9% of extractables, datum higher than what was found by Ruiz-Aquino *et al.* (2020) for *Q. macdougallii*, with total extractables for heartwood of 8.35% and for sapwood of 7.24%. *Q. laurina* had values in stump of 10.29%, in stem of 8.45%, and in branches of 10.76%, similar value to what Jaucida *et al.* (2002) found for *Quercus palustris* with an average of extractables of 7.72%. The content of extractables has an indirect impact on the mechanical properties of wood when influencing their basic density (Ruiz-Aquino *et al.*, 2015). The extracts also have a positive influence on calorific value, since biomass with high content of extractable substances have a higher calorific value (Mauladdini *et al.*, 2022).

Content of inorganic substances

The content of ash varied in an interval of 1.28 to 2.61% for stem of *Q. calophylla* and stump of *Q. glaucoides*, respectively (Table 1). *Q. glaucoides* is the species that showed percentages higher than 2% in the three parts of the tree, that is, stump, stem and branches.

These were similar values to those found by Martínez-Pérez *et al.* (2015) for two broadleaf species with 2.28 and 2.16%. *Q. laurina* presented 1.76% in stump, 1.94% in stem, and 1.79% in branches; these values are lower than the ones reported by Bárcenas-Pazos *et al.* (2008) for *Quercus thinkami* with 2.67%, and for *Q. sebifera* with 3.22%. Vega-Nieva *et al.* (2015) have mentioned that ashes can produce problems during combustion in heaters. In turn, Solla-Gullón *et al.* (2001) argue that depending on the composition of ash, it can function as a nutrient for the soil, decreasing the degree of acidity of some soils, and improving the level of nutrients with ash and nitrogenated fertilization.

Pearson's correlation

Pearson's correlation analysis was conducted between the chemical components, and it was seen that cellulose and hemicelluloses are negatively correlated ($r=-0.80$); that is, as the cellulose decreases, the hemicellulose increases (Table 2). When cellulose is produced in wood, lignin is obtained as byproduct, and it is used mainly as fuel (Ruiz, 2018). For cellulose and lignin, there is a negative correlation ($r=-0.72$), which means that when the cellulose is high, the lignin tends to be low. The presence of lignin has an important role in the response from wood to fluctuations in the moisture content, significantly influencing its behavior in face of dimensional changes (Bárcenas and Dávalos, 1999).

Lignin and extractable substances are not correlated; extractable substances have diverse properties in the biomass such as color, odor, durability, adhesion, drying and discoloration (Álvarez *et al.*, 2012). It should be mentioned that these two variables are essential to select species for their aptitude as biofuels. Cellulose constitutes the fundamental structure, hemicellulose performs a role of connection, lignin confers resistance, and extractable substances influence many specific properties of the wood (Álvarez *et al.*, 2012).

Higher heating value

The higher heating value for stump of *Q. glaucoides* and stem of *Q. rugosa* varied from 19.32 to 20.19 MJ kg^{-1}, respectively. Likewise, the species that presented the highest average was *Q. rugosa* with 20.01 MJ kg^{-1} (Table 2). The values obtained in this study are

Table 2. Pearson's correlation between the chemical composition of five species of *Quercus*.

		Hemicellulose	**Cellulose**	**Lignin**	**Extractable substances**	**Ash**
Hemicellulose	Correlation	1.00	−0.80	0.33	0.56	0.06
	Significance		0.00	0.22	0.02	0.81
Cellulose	Correlation	−0.80	1.00	−0.72	−0.61	-0.08
	Significance	0.00		0.00	0.01	0.75
Lignin	Correlation	0.33	−0.72	1.00	−0.00	−0.15
	Significance	0.22	0.00		0.97	0.58
Extractable substances	Correlation	0.56	−0.61	−0.00	1.00	0.15
	Significance	0.02	0.01	0.97		0.58
Ash	Correlation	0.06	−0.08	−0.15	0.15	1.00
	Significance	0.81	0.75	0.58	0.58	

Table 3. Higher heating value of wood from five species of *Quercus* (MJ kg^{-1}).

Species	Stump	Stem	Branches	Average
Q. calophylla	19.65	19.46	19.63	19.58
Q. glaucoides	19.32	19.58	19.86	19.59
Q. laurina	19.99	19.85	19.79	19.88
Q. magnoliifolia	19.53	19.54	19.63	19.57
Q. rugosa	19.88	20.19	19.95	20.01

slightly lower than those calculated for the same species by Ruiz-Aquino *et al.* (2022), using a flat-jacket calorimeter; the authors mention that the species of the genus *Quercus* are among the most frequently used as fuel, due to their calorific capacity and their high basic density, important characteristics in the selection of biomass for bioenergetic uses. Also, the calorific value can vary depending on biomass, climate and soil where the trees are cultivated (Ciolkosz *et al.*, 2010). Based on calorific value, the five species from this study are considered to have aptitude for their bioenergetic use because they exceed 18 MJ kg^{-1} (Koppejan and Van Loo, 2012; Ngangyo-Heya *et al.*, 2022).

CONCLUSIONS

Quercus rugosa was the species that presented highest value of lignin content and extractable substances in stem and stump, respectively; it also presented the lowest content of cellulose. Four of the species studied have the highest value of hemicellulose in branches, except for *Quercus glaucoides* which has higher content of hemicellulose in stump. Regarding lignin, *Q. calophylla* and *Q. laurina* presented higher content in stump, *Q. glaucoides* and *Q. magnoliigolia* presented higher value in branches, and only *Q. rugosa* showed higher percentage of lignin in stem. As a result of the lignin content of the species, they can have potential for their use as fuels. For the content of extractable substances, *Q. glaucoides*, *Q. laurina* and *Q. rugosa* presented the highest percentage in branches, and *Q. laurina*, *Q. magnoliigolia* and *Q. rugosa* in stump. *Q. glaucoides* is the species that presented highest percentage of ash in the three parts of the tree, in contrast with *Q. calophylla*, which presented lower value of ash in stump, stem and branches. The cellulose and hemicellulose are negatively correlated, as are cellulose and lignin. Lignin and extractable substances were not correlated, and neither was ash with any other chemical component. Based on their chemical composition, the five species studied are apt for their use as biofuels, although *Q. rugosa* is the species that presents higher percentage of lignin content, extractable substances in stem and stump, and high heating value, so it is considered with greater potential for the elaboration of thickened fuels.

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