

Chemical Composition and Energy Evaluation of *Abies* spp. and *Pinus* spp. Sawdust Collected as a Byproduct of the Primary Wood Sawing

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ABSTRACT

The aim of this paper is to chemically evaluate the byproducts of the primary processing of genera *Abies* and *Pinus*, to determine the possibility of using them as solid biofuel. Ash percentage, volatile matter and fixed carbon values were determined by proximate analysis. The basic chemical composition includes the determination of extractives content, lignin and holocellulose. Ash microanalysis was performed with an X-ray spectrometer and the calorific value of the samples was determined by using an AC600 calorimeter. The results of this research varied as follows: the content of inorganic substances (0.33% to 0.41%), volatile matter (88.54% to 82.57%), fixed carbon (11.13% to 17.06%), extractives content (5.37% to 17.82%), Runkel lignin (27.33% to 30.97%), holocellulose content (58.53% to 69.56%) and calorific value (19.09 MJ·kg⁻¹ to 20.42 MJ·kg⁻¹). According to the X-ray analysis, the most abundant elements were potassium, calcium and magnesium; whereas no heavy metals were found. The results of this research show that the two genera studied here are suitable for solid biofuel production.

Keywords: byproducts; sawmills; biofuel; chemical composition; calorific value

INTRODUCTION

During the last decades, atmosphere composition has been affected by the increasing emission of greenhouse gases produced by anthropogenic activities. In addition, activities such as the combustion of fossil fuels and the possible exhaustion of oil reserves, which are causing climate change, have also boosted a worldwide demand for renewable energy sources (Escorsim et al. 2018), such as biomass.

Biomass production is significant and stands in the fourth place in the global energy balance right after coal, oil and natural gas. Biofuels are considered better than fossil fuels, since their overall CO₂ emissions can be considered equal to zero. This is due to the fact that during its growth a plant can absorb the same amount of CO₂ and release oxygen through the photosynthetic process, as well as because plants have low sulfur and nitrogen contents (Di

Blassi 2009). Furthermore, thanks to its abundance, wood is the lignocellulosic raw material most widely-used, namely, as construction material, for furniture production, and for charcoal and biofuel production (Karinkanta et al. 2018).

The mechanical forestry industries use wood primarily for sawmilling, which is one of the least complex activities. It consists of a certain number of operations, starting with log handling and transportation, wood drying, selection and classification. Several types of energy are needed to perform these operations (FAO 1991). Throughout this productive process, several byproducts are generated, such as sawdust, woodchips, shavings, and slabs (FAO 1991, Zavala-Zavala and Hernández-Cortés 2000), which might be incorporated into the production of pulp, paper, boards, and fertilizers (Kollman 2001). Nonetheless, the volume of generated byproducts places biomass as a source of energy that is economically competitive with fossil fuels (García et al. 2012).

In the interest of achieving both domestic and industrial uses of these byproducts, it is necessary to determine their properties and components (Mitchual et al. 2014). Properties of combustible materials include calorific value, density, moisture content, ash, mineral content, volatile matter, and fixed carbon content (Khan et al. 2009, Mediavilla et al. 2009, Telmo et al. 2010), while their structural analysis consists of determining the cellulose, hemicellulose and lignin content (Zhang et al. 2010) and extractive content (White 1987). Thus, the aim of this paper is to evaluate the byproducts of the primary processing of genera *Abies* and *Pinus* wood to determine the possibility of using them as solid biofuel.

MATERIALS AND METHODS

Study Area

The study was carried out in the municipality of San José del Rincón, Mexico. The study area is located between parallels 19°29' and 19°47' N; meridians 100°01' and 100°16' W; with altitude of 2500-3700 meters. The municipality's climate is mild sub-humid with rains in the summer months and an average annual precipitation of 900 mm. The average temperature is 12°C (INEGI 2009). This area stands out for belonging to the Monarch Butterfly Biosphere Reserve. The byproducts of forest genera *Abies* and *Pinus* were collected from two private sawmills that process sawn lumber into commercially sized timber. The facilities are located on the periphery of San José del Rincón.

Samples Preparation

The samples were taken from the process machinery: the main saw, string trimmer and edging-saw. Then, sawdust samples were dried at room temperature and grounded in a Micron mill, model K20F, in accordance with standard T 257 om-85 (TAPPI 1985). After that, samples were sieved using stainless steel Montinox-sieves, and 40-mesh wood meal was stored in sealed plastic containers to avoid any contamination and moisture absorption. Each chemical determination was made out in triplicate.

Methods

Proximate Analysis: Ash Percentage, Volatile Substances and Fixed Carbon.

Inorganic material content was determined using the gravimetric method. One gram of the sample was first burnt over a Bunsen burner flame in nickel crucibles, and then heated in a Muffle furnace at 550±10°C during of 4 hours (UNE-EN 14775 2010).

Volatile matter was determined in accordance with ASTM E 872-82 (1985). 1 g of wood meal was heated in a capped nickel crucible, to avoid the entrance of environmental air.

The fixed carbon content of a char is the calculated value of the difference between 100 and the sum of ash and volatile matter, where all values are expressed on the same moisture reference base, according to Equation 1,

$$\% \text{ fixed carbon} = 100 - \% \text{ volatile matter} - \% \text{ ash}$$

Characterization of the Basic Chemical Composition of Byproducts

Total amount of extractives was determined using 9.5 g of wood meal in a Soxhlet extraction sequence under 6 hours reflux cycles, using solvents in the order of increasing polarity: cyclohexane, acetone, methanol, and hot water (Mejía-Díaz and Rutiaga-Quiñones 2008). The resulting solvents were retrieved by applying vacuum in a rotatory evaporator. The extractive content for each solvent was calculated dividing the weight of the anhydrous extract by the weight of the anhydrous meal, expressed as the percentage. The total extractives were calculated as the sum of the percentages of all extractives of the sample. Once the extraction sequence was over, wood meal was considered to be extractive-free and used to determine lignin (Runkel and Wilke 1951) and holocellulose (Wise et al. 1946). The results are expressed as percentages.

Ash Microanalysis

Ash microanalysis was performed using an X-ray spectrometer, connected to a Jeol JSM - 6400 scanning electron microscope, with operating conditions of 20 kV and 8.5 s (Téllez-Sánchez et al. 2010).

Calorific Value

Pellets were formed from 0.1 g samples, on a dry basis, to determine the calorific value (MJ·kg⁻¹) in an AC600 bomb calorimeter, which was calibrated with benzoic acid (UNE-EN 14918: 2011).

Statistical Analysis

An analysis of variance was performed using a general linear model, with a randomized complete block design. Tukey's test was used to compare means. Both statistical analyses were processed using Minitab V. 17 (Minitab Inc. 2017), at a significance value of P<0.05 and a 95% confidence interval. Additionally, a multiple linear regression was performed to correlate calorific value of the byproducts with their lignin and extractive content and between the ash content and volatile matter against the calorific value.

RESULTS AND DISCUSSION

Proximate Analysis: Ash Percentage, Volatile Matter and Fixed Carbon

The ash content found ranged from 0.33% to 0.41% (Table 1) and no statistical difference was observed between the two genera studied here. This result agrees with the data reported by Bernabé-Santiago et al. (2013) for some other species of the genus *Pinus* (0.3%) and it is within the parameter (0.1-1.0%) reported by Fengel and Wegener (1989). Likewise, the inorganic content found in this study is lower than the permitted values for softwood pellets and briquettes, 0.4% and 0.8% (Oberberger and Thek 2004), and it complies with the standard UNE-EN 14961-2 (2012), which sets the requirement for high quality pellets (≤0.7%).

Ash composition and properties are determined by age, tree species, part of the tree, environmental conditions, cultivation, harvesting, harvesting time and technique,

transportation, storage, pollution and processing (Vassilev et al. 2017). Ash content may cause operating problems during the combustion process, such as slag formation and reduction of the protective layer of oxide in the combustion chamber (Lehtikangas 2001), causing higher maintenance costs (Grover and Mishra 1996). Reported low ash content values are positively related to heating value, since the elements conforming to the ashes generally do not have heating value themselves (FAO 1997). Therefore, the risk of obstruction problems in the combustion equipment is also reduced.

The volatile matter content found is shown in Table 1, with a variation of $88.54\% \pm 0.7\%$ to $82.57\% \pm 0.8\%$, the genus *Abies* being the one with the highest value. This value is higher than the one found in the byproducts of the primary transformation of *Pinus patula* (80.15%), reported by González-Martínez (2013); it is also higher than the ones reported for woody biomass, which vary from 76% to 86% (Van Loo and Koppejan 2002). Our result is also high compared to the value of 83.1% reported by García et al. (2012) for wood byproducts. Biomass generally contains high levels of volatile matter (64-98%) depending on plant origin, compared to fossil coal (40%) (Vassilev et al. 2010). Volatile matter refers to the part of the biomass that is released when the biomass is heated at 400°C to 500°C. A high volatile matter content indicates that during combustion, most of the biomass will volatilize and burn as gas (Mitchal et al. 2014). This content influences the thermal behavior of solid biofuels (Van Loo and Koppejan 2002), therefore, it can be stated that the higher volatile matter content, the lower ignition temperature of the fuel (Vamvuka et al. 2011). Thus, the quality of the fuel is related to the high content of volatile material.

Finally, fixed carbon, which is the solid combustible residue that remains after a char particle is heated and the volatile matter is expelled, varied from 11.13% to 17.06% (Table 1), which can be considered a low percentage. The genus *Pinus* showed the highest fixed carbon content.

Yet, it is a value below the range of 20-25% reported in wood pellets (Stelte et al. 2013). The fixed carbon content is the percentage of carbon available for combustion, so it is desirable to get the lowest level of this parameter as it ensures a higher volatile content (Bandara and Kowshayini 2017).

Analysis of the Basic Chemical Composition

The results of the extractive substances are shown in Table 1. In this case, significant difference was presented for *Pinus* spp. when using cyclohexane as solvent ($12.93\% \pm 1.39\%$), in the edging saw, which indicates that the test samples were very heterogeneous. This result differs from Bernabé-Santiago et al. (2013), who reported the highest solubility in hot water for several species of the same genus (1.6-2.1%). The lowest value corresponded to *Abies* spp. with cyclohexane ($0.43\% \pm 0.03\%$) for both the main saw and the string trimmer. Wood contains secondary metabolites such as terpenes, fats, waxes, phenols and sugars, among other compounds (Fengel and Wegener 1989). This chemical composition depends on various factors such as age, species, tree conditions, geographic location, environmental factors, genetic factors, available nutrients, and part of the tree used to get the sample (Vázquez et al. 1987, Fengel and Wegener 1989, Ramos-Pantaleón et al. 2011), which explains the yield difference found in this research work.

Furthermore, the methods applied influence the extractives yield (Rutiaga-Quiñones et al. 2000). Extractives content, in turn, influences physical and mechanical properties of wood (Poblete et al. 1991, Ávila and Herrera 2012). It has been determined that extractive content is a parameter directly affecting the heating value of biomass (Kataki and Konwer 2001, Demirbas 2002, Demirbas and Demirbas 2009). Therefore, the calorific value increases as the extractives content does (Sadiku et al. 2016). On the contrary, no interaction was found between the machinery and the extractives content.

Table 1. Physical and chemical properties of timber byproducts.

	<i>Abies</i> spp.			<i>Pinus</i> spp.		
	MS (%)	ST (%)	ES (%)	MS (%)	ST (%)	ES (%)
Ash	0.41±0.06a	0.33±0.002a	0.37±0.07a	0.33±0.00a	0.37±0.06a	0.33±0.01a
Volatile matter	87.83±0.35ab	88.54±0.77a	86.23±0.63ab	85.79±1.57b	82.57±0.86c	86.23±0.64ab
Fixed carbon	11.77±0.41bc	11.13±0.77c	13.4±0.56bc	13.88±1.56b	17.06±0.85a	13.45±0.64bc
Cyclohexane	0.43±0.03b	0.43±0.05b	0.60±0.26b	2.35±0.04b	2.03±0.05b	12.93±1.39a
Acetone	2.45±0.13a	1.82±0.13b	2.63±0.20a	0.83±0.02cd	0.69±0.01d	1.23±0.04c
Methanol	3.22±1.25a	1.72±0.14ab	2.77±0.40ab	0.75±0.03b	0.76±0.08b	0.84±0.01b
Hot water	1.15±0.13a	1.40±0.04a	2.08±0.48a	1.85±0.30a	1.97±0.08a	2.82±1.42a
Total extractives	7.25±1.28bc	5.37±0.11c	8.08±0.02b	5.78±0.39c	5.45±0.20c	17.82±0.07a
Runkel lignin	28.39±0.50a	27.33±0.66a	29.29±0.97a	27.55±0.45a	30.97±2.01a	27.26±0.65a
Holocellulose	69.56±0.71a	66.72±0.53b	66.44±0.41bc	65.56±0.49bc	64.40±0.37c	58.53±1.76d

MS – main saw; ST – string trimmer; ES – edging saw; % – percentage on a dry basis. Mean±standard error: same letters on the line indicate that there is no statistically significant difference (Tukey, $P < 0.05$).

In woody plants, the content of lignin is about 15-40% (Gellerstedt and Henriksson 2008). The values of Runkel lignin varied from 27.26%±0.65% to 30.97%±2.01% for the genus *Pinus*, while for the genus *Abies* it ranged from 27.33%±0.66% to 29.29%±0.97%; with no statistical difference found (Table 1). The content of lignin is within the results reported by Lima-Rojas (2013) for sapwood and heartwood of *Pinus leiophylla* (29.1-29.8%), *Pinus montezumae* (28.3-28.4%) and *Pinus pseudostrobus* (26.9-29.2%). Pintor-Ibarra et al. (2017) reported similar values (29.57-32.52%) for the same species. Lignin contents found in this study are close to the Klason lignin values obtained in *Pinus patula* (31.0%) (González-Martínez 2013), and to those reported by Rowell et al. (2005) for other pine species (25-30%) and by Berrocal-Jiménez et al. (2012) for *Pinus radiata* at the age of 16-20 years (29.4%). No reports were found for the genus *Abies*. The calorific value of softwoods depends on lignin content, since lignin has a low oxygen content. Other intervening factors to determine this value are species, age, and growth conditions (Bajpai 2018). In addition to the lignin content, softwoods are considered to have a high calorific value due to their high extractives content (White 1987). Lignin is the main component that acts as a binder, essential for forming a good pellet (Stelte et al. 2011), and it has an important influence on durability (Lehtikangas 2001). The economic advantage is that binding additives costs are cut down in pellet production.

Holocellulose content is also shown in Table 1. Statistical differences were found in the two studied genera. The content ranges from 58.53%±1.76% (for *Pinus* spp.) to 69.56%±0.71% (for *Abies* spp.). This content is within the reported values for coniferous woods (65-75%) (Rowell et al. 2005). Holocellulose is made up of cellulose and hemicellulose (Fengel and Wegener 1989). It is known that the main components of wood have different calorific value. In a research, heating value values for holocellulose (15.9 to 16.3 MJ·kg⁻¹) and for lignin (22.5 to 23.5 MJ·kg⁻¹), isolated from oak wood, have been reported (Herrera-Fernández et al. 2017). It is clear that high lignin values are desired in the biomass used for making solid biofuels; however, for good pellet formation for example, the moisture content plays an important role (Samuelsson et al. 2009).

Ash Microanalysis

The byproducts' relative percentage of ash, expressed as an atomic percentage, is shown in Table 2. The most abundant elements were potassium, calcium and magnesium, which coincides with previous reports (Fengel and Wegener 1989, Martínez-Pérez et al. 2012, Correa-Méndez et al. 2014, Pintor-Ibarra et al. 2017). In the case of potassium, a significant difference was found in both genera, corresponding to the genus *Abies* a higher value (60.88%±0.83%), which coincides with the average value reported by Lima-Rojas (2013) for the *Abies religiosa* species in sapwood and heartwood (60.21%) under the same conditions of analysis. For calcium, the highest content was found in the genus *Pinus* with a value of 41.63%±0.03%, as well as in the case of magnesium (19.59%±0.72%), these values coincide with previous studies for *Pinus* woods (Bernabé-Santiago et al. 2013, Lima-Rojas 2013, Correa-Méndez et al. 2014). These elements can be found in wood as oxalates, carbonates, sulfates, silicates and phosphates (Ragland and Aerts 1991, Hon and Shiraiishi 1991),

resulting in the risk of corrosion and slag (Vassilev et al. 2014). Inorganic elements are highly variable between and within species and they can vary with soil and growth rate (Young and Guinn 1966, Ragland and Aerts 1991, Fernandez et al. 2020). Other elements are also present in smaller amounts: manganese, sulphur, silicon, aluminium, phosphorus; while iron was only detected in the genus *Pinus* (0.96%±0.03%), in concordance with the results of Ragland and Aerts (1991) for wood species. During combustion, mineral ions oxidize and volatilize, or form particles (Ragland and Aerts 1991). Knowledge of mineral composition is needed for analysis of ash deposition, erosion and corrosion in combustion systems, as well as for understanding stack gas opacity (Ragland and Aerts 1991, Jenkins et al. 1998). Moreover, potassium plays a key role in ash fusion and deposition (Díaz-Ramírez et al. 2014). During the combustion process mineral matter vaporizes and, when the temperature decreases, it condenses on heating surfaces and forms a sticky slagging layer (Wei et al. 2005). A high potassium content, by nucleation and condensation, increases ash-related issues, including alkali and silicate-induced slagging formation, as well as agglomeration (Niu et al. 2016). Silicon and phosphorus increase ash fusion (Mu et al. 2012); whereas potassium, magnesium, calcium and manganese decrease it (Liu et al. 2018). The presence of silicon and aluminium reduces fouling and increases ash fusion temperature. Also, aluminium may reduce the risk of corrosion during the combustion process (Vargas et al. 2001). As mentioned before, iron was detected only in the case of the genus *Pinus*. These two latter elements could be related to contaminants in wood machinery (Correa-Méndez et al. 2014). However, the presence of these two elements is characteristic in wood (Fengel and Wegener 1989).

Calorific Value

The results for heating value are shown in Table 3. The results for heating value ranged in from 19.09 MJ·kg⁻¹ to 20.42 MJ·kg⁻¹, with the genus *Pinus* being the one with the highest value. The two studied genera turned out to be statistically different. The values found in this study are higher than those reported for maritime pine (17-18 MJ·kg⁻¹) (Vassilev et al. 2013) and for *Pinus patula* (18.6-18.9 MJ·kg⁻¹) (González-Martínez 2013). A study of different agricultural and forestry species reported a calorific value between 16.18-21.23 MJ·kg⁻¹ (Özyuguran and Yaman 2017). The obtained values are within the ranges for wood, between 19.8 MJ·kg⁻¹ and 20.7 MJ·kg⁻¹ (Oberberger and Thek 2004). The variability of such results indicates that calorific value depends on the tree species, and therefore, on its chemical characteristics. Previous research has reported a direct and positive relationship between calorific value and lignin and extractive concentrations (White 1987, Lehtikangas 2001).

Multiple Linear Regression Analysis Between the Total Removable Content and Lignin Against Calorific Value

According to the multiple linear regression analysis between the byproducts, calorific value and lignin and extractive content, it was observed that calorific value tends to increase as the total extracts do. This is consistent with previous studies (Martínez-Pérez et al. 2012, Ngangyo-Heya et al. 2016), with a correlation coefficient R²=0.774. No correlation was shown between lignin and calorific value

Table 2. Ash microanalysis of timber byproducts.

Element	<i>Abies</i> spp.			<i>Pinus</i> spp.		
	MS (%)	ST (%)	ES (%)	MS (%)	ST (%)	ES (%)
K	57.32±0.95ab	56.60±0.62b	60.88±0.83a	29.13±1.75cd	32.74±0.8c	27.65±0.91d
Ca	31.18±0.71c	32.41±0.40c	21.56±1.78d	38.56±2.08ab	34.57±1.48bc	41.63±0.03a
Mg	8.90±0.86d	9.59±0.30d	7.42±0.74d	19.59±0.72a	12.85±0.70b	16.30±0.12c
Mn	0.64±0.08a	1.04±0.12ab	1.03±0.33ab	1.76±0.14c	1.98±0.51bc	1.77±0.15c
S	0.20±0.04a	0.36±0.09b	0.05±0.04b	1.52±0.18c	3.88±0.66c	2.21±0.14c
Si	0.61±0.20a	0.60±0.12ab	1.23±1.04ab	2.70±0.51b	5.11±0.04b	2.67±1.11b
Al	0.25±0.21a	0.19±0.04a	0.27±0.33a	2.56±0.18b	3.47±0.38b	3.51±0.48b
P	0.05±0.01a	0.31±0.07b	0.05±0.03b	2.85±0.16c	4.91±0.57c	3.30±0.47c
Fe	Nd	Nd	Nd	0.96±0.03a	Nd	Nd

MS – main saw; ST – string trimmer; ES – edging saw; % – percentage on a dry basis; Nd – not detected. Mean±standard error: same letters on the line indicate that there is no statistically significant difference (Tukey, $P < 0.05$).

Table 3. Calorific value of timber byproducts.

Element	<i>Abies</i> spp.			<i>Pinus</i> spp.		
	MS (MJ·kg ⁻¹)	ST (MJ·kg ⁻¹)	ES (MJ·kg ⁻¹)	MS (MJ·kg ⁻¹)	ST (MJ·kg ⁻¹)	ES (MJ·kg ⁻¹)
Calorific value	19.12±0.24b	19.15±0.13b	19.09±0.26b	19.54±0.27ab	19.71±0.67ab	20.42±0.24a

MS – main saw; ST – string trimmer; ES – edging saw. Mean±standard error: same letters on the line indicate that there is no statistically significant difference (Tukey, $P < 0.05$).

($R^2 = -0.142$), which differs from literature data (Tillman 1978, Jara 1989, Cunha et al. 1989), and which is consistent with previous reports (Avelin et al. 2014, Sadiku et al. 2016), which reported that a higher value is not correlated with lignin content.

A negative linear relationship was observed between the calorific value and the volatile matter, and also between the calorific value and the ash content. A correlation coefficient $R^2 = -0.450$ was obtained between the calorific value and the volatile matter, indicating that the calorific value values decreased with the increase in volatile matter. In this regard, Katakai and Konwer (2001), and Demirbas and Demirbas (2009) mention that the high calorific value may be due to the presence of extractives and lignin. White (1987) and Guadalajara (2015) suggest that coniferous species have a higher calorific value due to the presence of resins. While for the calorific value and ash content it was $R^2 = -0.147$, showing a tendency to decrease with a higher value of ash content, which is consistent with the literature (Márquez-Montesinos et al. 2001, Martínez-Pérez et al. 2012) and is an indicative factor of fuel quality (Kumar et al. 2009, Kumar et al. 2010, Meetei et al. 2015).

CONCLUSIONS

Chemical and physical properties of byproducts generated from the primary processing of wood were analyzed in order to determine whether they might be incorporated into the production chain of solid biofuels.

Both genera, *Pinus* spp. and *Abies* spp., presented low ash content values, indicating a low risk of slag production. Also, volatile matter content was high, which will require a low ignition temperature to burn in combustion. As for the genus *Pinus*, it presented the highest extractives content, as well as the lowest content of lignin, which is associated with higher calorific value. These results suggest that both genera are suitable to be used in the production of solid biofuels.

Author Contributions

OMG: formal analysis, writing, investigation. LFPI: formal analysis, validation. JGRQ: conceptualization, formal analysis, funding acquisition, project administration, resources, review and editing. JCT: formal analysis, data curation, validation, writing, review and editing.

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Conflicts of Interest

The authors declare no conflict of interest.

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