# Basic Chemical Composition of *Pinus* spp. Sawdust from Five Regions of Mexico, for Bioenergetic Purposes

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The basic chemical composition and calorific value of 19 samples of pine sawdust from different forest industries located in five states of the Mexican Republic (Chihuahua, Michoacán, Durango, Oaxaca, and Nuevo León) were determined. The results obtained ranged as follows: total extractives (6.1% to 23.4%), holocellulose (60.1% to 70.4%), lignin (20.5% to 25.8%), ash (0.27% to 0.95%), pH (4.1 to 5.3), and calorific value (20.1 MJ/kg to 21.0 MJ/kg). Except for the ash content, significant statistical differences were found according to the origin of the pine sawdust samples. Based on the results obtained, the sawdust biomass has the potential to obtain densified solid biofuels.

*Keywords: Extractives; Holocellulose; Lignin; Ash; High heating value; Biomass*

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## INTRODUCTION

Within the conifers that grow in Mexico, the richest is the *Pinus* genus, with 40 species, of the 120 pine species that vegetate in the world (Gernandt and Pérez-de la Rosa 2014). The number of pine species that grow in the states of the Mexican Republic where this study was conducted is as follows: 14 species and 2 varieties in Chihuahua (Lebgue-Keleng *et al*. 2015), 15 species in Michoacán (Cué-Bär *et al*. 2006), 20 species in Durango (García and González 2003), 14 species and 2 varieties in Oaxaca (Del Castillo *et al*. 2006), and 15 species in Nuevo León (Estrada-Castillón *et al*. 2014).

In Mexico, the national timber forest production fluctuated during the 2008 to 2017 period, starting in 2008 with 6.3 million cubic meters log ($m^3$l) and ending with 9.0 million $m^3$l in 2017. The main wood-producing states in 2017 were: Durango (28.4%), Chihuahua (18.6%), Oaxaca (8.1%), Tabasco (6.7%), and Michoacán (5.4%). By genus or group of woods, timber forest production had the following distribution: *Pinus* spp. (70.9%), Mexican pine "Oyamel" (2.5%), other conifers (0.4%), *Quercus* spp. (9.8%), other hardwoods (2.8%), precious woods (0.2%), and tropical commons woods (13.4%). On the production of coniferous wood, the states of Durango, Chihuahua, and Oaxaca stand out, representing 65.6% of national production. The total forest production of *Pinus* spp. wood was 6,386,758 $m^3$l for that same 2017 year and had the following distribution: squared timber (74.6%), cellulosic material (10.8%), veneering and plywood (7.1%), posts, piling and fencing (0.4%), firewood (3.5%), charcoal ($9.3 \times 10^{-3}$%), and tramway sleepers (cross-

ties) (4.3%). The *Pinus* spp. wood sawmill represented 84.2% of national production in 2017 (SEMARNAT 2020). This data reflects the important role of pine species in national timber production.

It is known that the wood transformation process generates lignocellulosic residues, which generally have no use (Zavala and Cortés 2000), but they are occasionally used locally (Correa-Méndez *et al*. 2014). It is important to focus efforts to use these wastes for energy purposes (Karinkanta *et al*. 2018) but given the variability in the chemical composition of the different biomasses (Werkelin *et al*. 2005; Spinelli *et al*. 2011), it is important to know their characteristics and properties to visualize domestic or industrial energy applications (Mitchual *et al*. 2014). Recently, data on granulometry, proximate analysis, and ultimate analysis have been reported for the biomass samples studied here (Rutiaga-Quiñones *et al*. 2020). Now, the goal of this work is to know the basic chemical composition of pine wood sawdust, derived from the primary processing of wood, to learn its potential for its possible use in the production of densified biofuels and also to generate reference data.

## EXPERIMENTAL

### Lignocellulosic Material

The collection of the lignocellulosic waste samples was carried out in different companies and forest ejidos (communal lands) in five states of the Mexican republic: Chihuahua (El largo, Agua azul, Grupo Gazo, and Multimaderas), Michoacán (Maderería Zamora, Maderas Preciosas Don Jesús, and Lázaro Cárdenas), Durango (Grupo Sezarik, Maderas y Tarimas Alba, Vencedores, La Cañita, and Pueblo Nuevo), Oaxaca (Unidad Comunal Forestal, Agropecuaria y de Servicios, Productora Comunal de Muebles Ixtlán, and Aserradero Mapsi), and Nuevo León (Maderas Los Pérez), and are the product of primary wood transformation of pine trees. It was not possible to separate the biomass samples by pine species, because in the forest exploitation the timber species are mixed and then transported to the sawmills. The collected sawdust was allowed to air dry (under shadow) and was subsequently ground and sieved to use the 40-mesh fraction for analysis (Mejía-Díaz and Rutiaga-Quiñones 2008).

### Chemical Analysis

To determine the total extracts content a Soxhlet extraction was applied using solvents of increasing polarity (cyclohexane, acetone, and methanol), and finally hot water under reflux, 6 h in each case (Mejía-Díaz and Rutiaga-Quiñones 2008). Holocellulose (Wise *et al*. 1946), and lignin (Runkel and Wilke 1951) were determined in the material after successive extraction. In the original biomass, the ash content as per UNE-EN 14775 (2010) and the pH by the methodology of Sandermann and Rothkamm (1959) were determined.

### High Heating Value

The high heating value of the lignocellulosic samples was calculated using the mathematical model based on the chemical composition (White 1987).

### Statistical Analysis

The chemical analyzes were performed in triplicate, and the mean value and standard deviation are reported.

Because only one sample was collected from the state of Nuevo León, the results obtained for this origin must be taken with reserve. However, to compare the results obtained by origin of the pine sawdust samples, an analysis of variance was applied at 95% statistical confidence, and the mean values were compared by means of the multiple range test with the least significant difference (LSD) method (Gutiérrez and de la Vara 2004). The results were analyzed using the Statgraphics Plus version 4.0 program (Statgraphics Technologies, Inc., The Plains, VA, USA).

## RESULTS AND DISCUSSION

### Chemical Analysis

Table 1 shows the list of samples in order of collection, and the results of the successive extraction processes applied. It was not within the scope of this work to carry out an analysis to know the chemical compounds of the extracts obtained. In general, the yield of solvent extraction had the following behavior: acetone > cyclohexane > hot water > methanol. This could indicate that most of the extractable substances present in these lignocellulosic samples correspond to nonpolar compounds (Fengel and Wegener 1983; Hillis 1987), and to a lesser extent water soluble substances, whose components can be carbohydrates, proteins, and some inorganic salts (Fengel and Wegener 1983).

**Table 1.** Extractives Content in Biomass Samples by Successive Extraction (%)

| Sample | Origin | Cyclo-hexane | Acetone | Methanol | Hot water | Total |
|---|---|---|---|---|---|---|
| 1 | Chihua-hua | 3.8 ± 0.10 | 5.3 ± 0.03 | 0.7 ± 0.13 | 2.0 ± 0.06 | 11.9 ± 0.21 |
| 2 | | 2.1 ± 0.15 | 5.8 ± 0.41 | 0.9 ± 0.19 | 1.7 ± 0.08 | 10.6 ± 0.27 |
| 3 | | 3.4 ± 0.23 | 5.3 ± 0.66 | 0.6 ± 0.08 | 2.1 ± 0.18 | 11.3 ± 0.16 |
| 4 | | 3.4 ± 0.18 | 8.1 ± 0.65 | 0.6 ± 0.04 | 2.8 ± 0.07 | 14.8 ± 0.48 |
| 5 | Micho-acán | 1.6 ± 0.19 | 3.9 ± 0.17 | 0.5 ± 0.05 | 1.5 ± 0.04 | 7.4 ± 0.08 |
| 6 | | 3.9 ± 0.10 | 6.3 ± 0.16 | 0.7 ± 0.01 | 2.4 ± 0.07 | 13.2 ± 0.02 |
| 7 | | 2.5 ± 0.09 | 4.8 ± 0.32 | 0.9 ± 0.16 | 2.3 ± 0.09 | 10.6 ± 0.04 |
| 8 | | 3.1 ± 0.49 | 4.3 ± 0.31 | 0.9 ± 0.08 | 2.1 ± 0.01 | 10.5 ± 0.88 |
| 9 | | 2.4 ± 0.10 | 4.4 ± 0.23 | 0.8 ± 0.05 | 3.2 ± 0.10 | 10.8 ± 0.08 |
| 10 | Duran-go | 9.7 ± 0.04 | 8.7 ± 0.03 | 1.0 ± 0.07 | 4.0 ± 0.10 | 23.4 ± 0.04 |
| 11 | | 6.7 ± 0.38 | 3.5 ± 0.16 | 1.2 ± 0.05 | 3.8 ± 0.00 | 15.1 ± 0.16 |
| 12 | | 8.1 ± 0.99 | 4.7 ± 0.34 | 1.6 ± 0.17 | 2.6 ± 0.39 | 17.0 ± 1.91 |
| 13 | | 4.2 ± 0.36 | 5.7 ± 0.74 | 0.7 ± 0.33 | 2.1 ± 0.08 | 12.7 ± 0.12 |
| 14 | | 3.2 ± 0.43 | 7.3 ± 0.30 | 0.5 ± 0.11 | 1.4 ± 0.08 | 12.3 ± 0.17 |
| 15 | | 2.1 ± 0.10 | 5.0 ± 0.05 | 0.9 ± 0.06 | 2.2 ± 0.03 | 10.3 ± 0.12 |
| 16 | Oaxaca | 2.0 ± 0.06 | 3.8 ± 0.47 | 0.8 ± 0.01 | 2.8 ± 0.04 | 9.4 ± 0.51 |
| 17 | | 0.9 ± 0.11 | 2.6 ± 0.59 | 1.0 ± 0.22 | 1.6 ± 0.30 | 6.1 ± 0.63 |
| 18 | | 2.1 ± 0.04 | 2.9 ± 0.40 | 0.9 ± 0.05 | 2.3 ± 0.07 | 8.2 ± 0.32 |
| 19 | Nuevo León | 4.0 ± 0.24 | 6.9 ± 0.06 | 1.2 ± 0.31 | 1.9 ± 0.19 | 14.0 ± 0.69 |

In extracts obtained with non-polar solvents, fatty acids, resins, resin acids, terpenes, and stilbenes are often found in pine woods (Uprichard and Lloyd 1980; Fengel and Wegener 1983). In *Pinus pseudostrobus* wood, pinosylvin demethyl ether and pinosilvin dimethyl ether have been identified in petroleum ether extract and acetonic extract, respectively (Rutiaga-Quiñones 2001). In aqueous extracts of *Pinus pseudostrobus* wood, after successive extraction with organic solvents, small amounts of polysaccharides were detected from 0.5% to 2.4%, while in wood the polysaccharide content varied from 67.2% to 67.8% (Rutiaga-Quiñones 2001). In general, the average total extractives content obtained by the applied extraction sequence (Table 1) is higher compared to reported results for some pine woods (7.6% to 8.2%) (Bernabé-Santiago *et al*. 2013), (3.5% to 12.0%) (Honorato-Salazar *et al*. 2016), and (6.6% to 7.1%) (Pintor-Ibarra *et al*. 2017). This variation could be attributed to the extraction method used, the type of wood, and it is known that there are variations even within the same tree (Hillis 1987).

Holocellulose values ranged from 60.1% to 70.4% (Table 2), and in general coincided with data reported for coniferous woods (67.0% to 82.5%) (Fengel and Wegener 1983) and especially for pine woods (64.0% to 69.0%) (Rowell *et al*. 2005), (68.1% to 74.7%) (Bernabé-Santiago *et al*. 2013), (66.7% to 70.0%) (Honorato-Salazar *et al*. 2016), and (71.9% to 76.9%) (Pintor-Ibarra *et al*. 2017). The authors' results were also consistent with the reported values (63.6% to 67.4%) for holocellulose content, obtained by delignification method for four species of Mexican pines from the cembroid subsection (Revilla-González 2011).

For lignin, the results ranged from 20.5% to 25.8% (Table 2). These values are in the lower range reported for conifers (25.6% to 39.4%) (Fengel and Wegener 1983) and are generally less than the results of previous research with pine woods (24.0% to 28.5%) (Bernabé-Santiago *et al*. 2013), (26.3% to 28.6%) (Honorato-Salazar *et al*. 2016), and (29.6% to 32.5%) (Pintor-Ibarra *et al*. 2017). This variation could be due to the analysis method used, the type of wood used in the analysis, the origin of the wood, since it is known that the variation in chemical composition is influenced by the growing conditions, geographical location, age of the trees, among others (Hon and Shiraishi 2001).

In relation to the amount of mineral substances, the results ranged from 0.27% to 0.95% (Table 2). In general, they coincided with data reported for pine woods (Bernabé-Santiago *et al*. 2013; Correa-Méndez *et al*. 2014; Pintor-Ibarra *et al*. 2017) and were also within the typical range for pine woods (0.1% to 1.0%) (UNE-EN 14961-1 2011). Based on these results, lignocellulosic residues could be used to make class A2 pellets, whose requirement is an ash content of less than 1.5%, or to make class A1 pellets with materials whose ash content is below 0.7% (ISO 17225-2 2014).

The pH values range from 4.1 to 5.3 (Table 2), indicating that the pH of the samples is weakly acidic (Kollmann 1959). These values agree with previous reports for pine woods (Bernabé-Santiago *et al*. 2013; Pintor-Ibarra *et al*. 2017).

## Higher Heating Value

The values obtained from the calorific value calculated using the mathematical model based on the chemical composition (White 1987) range from 20.1 to 21.0 MJ/kg (Table 2). These results are higher than those reported for the same sawdust samples (18.8 to 20.7 MJ/kg), calculated using mathematical models based on ultimate analysis, proximal analysis, and the ash content (Rutiaga-Quiñones *et al*. 2020). They are also higher than range of typical variation reported for softwoods (18.5 to 19.8 MJ/kg) (UNE-EN 14961-1 2011). The calorific value is related to the chemical components of wood, mainly with the

content of lignin and extractives, in addition, in softwoods the content of resins increases the calorific value (White 1987).

**Table 2.** Holocellulose, Lignin, Ash, pH, and Calorific Value (HHV) in Biomass Samples

| Sample | Origin | Holocel-lulose (%) | Runkel Lignin (%) | Ash (%) | pH | HHV (MJ/kg) |
|---|---|---|---|---|---|---|
| 1 | Chihua-hua | 64.5 ± 0.58 | 24.1 ± 0.50 | 0.47 ± 0.01 | 4.6 ± 0.09 | 20.5 ± 0.05 |
| 2 | | 69.8 ± 0.51 | 20.9 ± 0.46 | 0.45 ± 0.01 | 4.1 ± 0.03 | 20.2 ± 0.05 |
| 3 | | 67.0 ± 0.74 | 22.4 ± 0.06 | 0.73 ± 0.03 | 4.2 ± 0.02 | 20.3 ± 0.02 |
| 4 | | 65.5 ± 0.61 | 23.6 ± 0.06 | 0.53 ± 0.01 | 4.2 ± 0.01 | 20.6 ± 0.03 |
| 5 | Micho-acán | 69.5 ± 0.60 | 23.5 ± 0.12 | 0.91 ± 0.06 | 4.8 ± 0.20 | 20.2 ± 0.01 |
| 6 | | 67.8 ± 0.19 | 24.2 ± 0.08 | 0.50 ± 0.01 | 4.2 ± 0.05 | 20.6 ± 0.004 |
| 7 | | 68.8 ± 0.66 | 25.5 ± 0.49 | 0.43 ± 0.06 | 4.2 ± 0.01 | 20.7 ± 0.20 |
| 8 | | 66.4 ± 0.14 | 24.0 ± 0.08 | 0.64 ± 0.02 | 4.6 ± 0.12 | 20.4 ± 0.07 |
| 9 | | 68.8 ± 0.39 | 24.1 ± 0.79 | 0.33 ± 0.01 | 5.3 ± 0.24 | 20.4 ± 0.07 |
| 10 | Duran-go | 60.4 ± 0.87 | 20.5 ± 0.15 | 0.58 ± 0.06 | 4.2 ± 0.07 | 21.0 ± 0.01 |
| 11 | | 61.3 ± 0.77 | 24.4 ± 0.37 | 0.73 ± 0.02 | 4.3 ± 0.06 | 20.7 ± 0.04 |
| 12 | | 60.1 ± 0.79 | 25.8 ± 0.50 | 0.55 ± 0.07 | 4.1 ± 0.06 | 20.9 ± 0.09 |
| 13 | | 68.5 ± 0.19 | 22.9 ± 0.53 | 0.27 ± 0.05 | 4.1 ± 0.23 | 20.5 ± 0.03 |
| 14 | | 68.7 ± 0.31 | 21.7 ± 0.84 | 0.75 ± 0.01 | 4.1 ± 0.03 | 20.3 ± 0.05 |
| 15 | | 68.4 ± 0.89 | 22.8 ± 0.21 | 0.95 ± 0.01 | 4.2 ± 0.01 | 20.3 ± 0.01 |
| 16 | Oaxaca | 69.4 ± 0.16 | 23.6 ± 0.01 | 0.66 ± 0.10 | 4.1 ± 0.01 | 20.3 ± 0.04 |
| 17 | | 70.4 ± 0.70 | 24.5 ± 0.22 | 0.34 ± 0.03 | 4.5 ± 0.03 | 20.1 ± 0.03 |
| 18 | | 69.3 ± 0.46 | 24.7 ± 0.32 | 0.48 ± 0.04 | 4.6 ± 0.04 | 20.3 ± 0.001 |
| 19 | Nuevo León | 67.2 ± 0.62 | 22.7 ± 0.04 | 0.50 ± 0.11 | 4.4 ± 0.09 | 20.5 ± 0.05 |

Table 3 summarizes the results obtained in this study by region of collection of the lignocellulosic samples. Except for the values obtained for the ash content, significant statistical differences were found in the other results ($p < 0.05$). A higher extractives content is observed for the Durango samples, which is reflected in a higher calorific value (Table 3), the same pattern was observed for the samples from Oaxaca, where the least extractives amount tends to with a lower calorific value. Telmo *et al.* (2010), found that in 17 wood fuels, the calorific value was explained by 43.6% of the quantity of extractives and 56.4% of Klason lignin, emphasizing that softwoods tend to have a higher calorific value as a consequence of the extractives content.

**Table 3.** Summary of the Chemical Composition and Calorific Value of Biomass Samples by Collection Origin

| Origin | Extractives (%) | Holocellulose (%) | Runkel Lignin (%) | Ash (%) | pH | HHV (MJ/kg) |
|---|---|---|---|---|---|---|
| Chihuahua | 12.1 a | 66.7 a | 22.9 a | 0.55 a | 4.3 a | 20.4 a |
| Michoacán | 10.5 a | 68.0 a | 24.3 b | 0.56 a | 4.6 b | 20.4 a |
| Durango | 15.1 b | 64.5 b | 23.0 a | 0.64 a | 4.2 a | 20.6 b |
| Oaxaca | 7.9 c | 69.7 c | 24.3 b | 0.49 a | 4.4 b | 20.2 a |
| Nuevo León | 14.0 b | 67.2 a | 22.7 a | 0.64 a | 4.4 b | 20.5 b |
| Equal lowercase letters in the direction of the columns indicate statistical equality ($p < 0.05$) | | | | | | |

## CONCLUSIONS

1. According to the solvents used, the total extractives content in biomass samples had the following behavior: acetone > cyclohexane > hot water > methanol.
2. The extractives concentration by origin of biomass samples had the following behavior: Durango > Nuevo León > Chihuahua > Michoacán > Oaxaca.
3. By origin of the biomass samples, the amount of holocellulose from highest to lowest was as follows: Oaxaca > Michoacán > Nuevo León > Chihuahua > Durango.
4. The Runkel lignin concentration in the studied samples had the following behavior according to the origin: Oaxaca/Michoacán > Durango > Chihuahua > Nuevo León.
5. The ash concentration by origin of biomass samples had the following behavior: Durango/Nuevo León > Michoacán > Chihuahua > Oaxaca.
6. The pH value, from higher to lower acidity, varied in the biomass samples according to their origin as follows: Durango > Chihuahua > Nuevo León/Oaxaca > Michoacán.
7. The HHV by origin of biomass samples had the following behavior: Durango > Nuevo León > Chihuahua/Michoacán > Oaxaca.
8. Based on the results obtained, the pine sawdust has the potential to be used in the production of densified solid biofuels.

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