ELSEVIER

Bioresource Technology 77 (2001) 101-108

BIORESOURCE TECHNOLOGY

# Utilization of by-products from the tequila industry. Part 2: potential value of *Agave tequilana* Weber *azul* leaves

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Received 14 April 2000; received in revised form 20 October 2000; accepted 21 October 2000

**Abstract**

The leaves of the agave plant are left in the field after harvesting the heads for tequila production. Different types of agave leaves were isolated, classified, and their content in the total plant determined. The usable fractions were collected and their properties determined. Of the total wet weight of the agave plant, 54% corresponds to the agave head, 32% corresponds to materials which could be usable for sugar and fiber production which leaves 14% of the wet plant without apparent utility. The fractions with higher total reducing sugars (TRS) content were the fresh fraction of partially dry leaves stuck to the head and the leaf bases with a TRS content of 16.1% and 13.1%, respectively. The highest TRS concentration (16–28%) is in the agave head which is used for tequila production. The leaves are 90–120 cm long and 8–12 cm wide and contain fiber bundles that are 23–52 cm long and 0.6–13mm wide. The ultimate fiber length is approximately 1.6 mm with an average width of 25 μm. There are several types of leaf fibers that can be utilized depending on what part of the plant they come from and what product is desired. Agave leaf fibers were pulped using a soda pulping process and the pulp was hand formed into test sheets. Test sheets made from pulped agave leaf fibers had a breaking length comparable to paper made from both pine and eucalyptus fibers, but the tear index and burst index were lower than the other two papers. ©2001 Elsevier Science Ltd. All rights reserved.

***Keywords:*** Agave plant; Agave leaves; Nonwood fiber pulp; Tequila by-products; Agave paper properties

## 1. Introduction

The diverse varieties of agave found throughout Mexico have been used since time immemorial to make different alcoholic beverages. Owing to numerous factors affecting the characteristics of agave including temperature, humidity, natural cycles, and average yearly rainfall. the product obtained in each region is completely different from that found in any other region. Tequila manufacture in a traditional distillery, begins with the cooking of *Agave tequilana* Weber *azul* plant heads to hydrolyze the polymers present in the plant, mainly inulin, into fermentable sugars. Agaves are cyultivated in arid and semi-arid regions worldwide. The genus contains 140 species, which constitute most of the family Agavaceae. Such plants are well adapted to arid and semi-arid environments as they utilize crassulacean acid metabolism (CAM), which was independently documented on three continents in the late 1960s for *Agave americana* (Gentry, 1982; Nobel, 1988). CAM plants fix atmospheric $CO_2$ mainly at night, when temperatures are lower than during the daytime. thereby decreasing the accompanying transpirational loss of water from plant to the atmosphere (Nobel et al., 1998). *Agave tequilana* Weber *azul* is an economically important CAM species cultivated in Mexico and is the only one of the species of Agavaceae that is appropriate for the production of tequila because of the high inulin concentration present in the plant. Inulin is a linear fructose polymer found in the agave "head" in large amounts. During heating of the head, hydrolysis of this polymer to fructose occurs which is used in the production of tequila.

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PII: S0960-8524(00)00167-X

*Agave tequiluna* Weber *azul* is a succulent plant, radiately spreading, 1.2–1.8 m tall with short thick stems 30–50 cm tall at maturity; panicle 5–6 m tall, large densely branched with 20–25 large diffusive dicompound umbels of green flowers with roseate stamens; flowers 68–75 mm long on small bracteolate pedicels 3–8 mm long (Gentry, 1982). The commercially important part of *A. tequilanu* Weber *azul* for tequila production is the stem and attached leaf bases, commonly termed "head" or "piña" because it resembles a pineapple fruit. Leaves are cut off near their bases and the "piñas" are harvested 7–9 years after planting but before the inflorescence is produced by this monocarpic perennial. *Agave tequilana* Weber *azul* leaves are 90–120 cm long × 8–12 cm width, lanceolate, acuminate, firm fibrous, mostly rigidly outstretched, concave, ascending to horizontal, widest through the middle, narrowed and thickened toward the base, generally bluish to gray green, sometimes cross-zoned, the margin straight to undulate or repand; teeth generally regular in size and spacing rarely irregular, mostly 3–6 mm long through mid-blade, the slender cusps curved or flexed from low pyramidal bases, light brown to dark brown, 1–2 cm apart, rarely remote and longer; spine generally short, 1–2 cm long, rarely longer, flattened or openly grooved above the broad base which is dark brown (Gentry, 1982). The fiber in the leaves is composed of agrate bundles of short fibers. The fiber bundles vary in length with an average length of 40 cm (23–52 cm) and an average width of 0.12 mm (0.613 mm). The average length of the ultimate fiber is 1.6 mm with an average width of 25 μm (unpublished results).

*Agave tequilana* Weber *azul* is cultivated only in very restricted regions established as protected territories by the tequila denomination of origin. These territories include Jalisco state and some municipalities in the states of Nayarit (8 municipalities), Guanajuato (6 municipalities), Michoacán (29 municipalities) and Tamaulipas (12 municipalities) (Fig. 1 and Table 1). The major land surface where *A. tequilana* Weber *azul* is cultivated is in the Jalisco state covering more than 482 millions $m^2$. In the states of Michoacán and Nayarit agave is planted but there are no tequila distilleries. In Guanajuato (Corralejo town) and Tamaulipas states there is only one distillery in each state with minimal production. The growth of *A. tequilana* Weber *azul* is authorized in states where there is no tequila production. These lands are held in reserve for a continuous expansion of the tequila industry. Currently Jalisco produces about 90% of the total tequila

Fig. 1. Protected territories by the tequila denomination of origin, Jalisco and Nayarit, Guanajuato, and Tamaulipas states with 8, 6, 29 and 12 municipalities, respectively.

**Table 1**
Statistics on plantations of agave plants and protected territories by the tequila denomination of origin where tequila is produced (NOM, 1994; CRT, 1995)

| Jalosco Municipality | Producers | $m^2$ | Agave plants |
|---|---|---|---|
| Acatic | 22 | 20,883,000 | 6,644,537 |
| Amatitán | 615 | 75,128,300 | 19,390,160 |
| AntonioEscobedo | 22 | 3,894,000 | 1,329,302 |
| Arandas | 290 | 64,145,100 | 25,603,814 |
| Arenal | 239 | 20,183,800 | 6,314,692 |
| Atotonilco | 376 | 79,609,500 | 28,314,692 |
| Ayotán | 53 | 12,536,300 | 4,725,521 |
| Hostotipaquillo | 10 | 1,410,000 | 485,135 |
| Jesus Maria | 19 | 9,801,700 | 3,729,869 |
| Magdalena | 8 | 2,827,000 | 1,034.76 |
| Tala | 69 | 16,780,200 | 5,946,096 |
| Tepatitlán | 221 | 67,235,100 | 24,944,066 |
| Tequila | 392 | 53,702,200 | 15,087,880 |
| Teuchitlán | 3 | 2,210,000 | 800,164 |
| Tlajomulco | 2 | 1,125,700 | 517,874 |
| Tototlán | 41 | 16,523,400 | 4,594,957 |
| Zapopan | 10 | 1,277,000 | 430,456 |
| Zapotlán | 3 | 3,855,500 | 1,053,169 |
| Zapotlanejo | 57 | 19,034,700 | 7,093,306 |
| Rest of Jalisco state (22) | 46 | 85,685,400 | 3,181,146 |
| Guanajuato [a] | 1 | 250,000 | 90.000 |
| Nayarit [b] | 0 | 30,000 | 108,013 |
| Tamaulipas [c] | 1 | 2,550,000 | 1,500,000 |
| Michoacan [d] | | | |
| Total | 2500 | 485,198,400 | 162,919610 |

[a] Municipalities: Abasolo, Manuel Doblado, Cuerámaro, Huanimaro, Pénjamo, Purísima del Roncón.
[b] Municipalities: Ahuacatlán, Amatlán de Cañas, Ixtlán, Jala, Jalisco, San Pedro de Lagunillas, Santa María del Oro, Tepic.
[c] Municipalities: Aldama, Altamira, Antiguo de Morelos, Gómez Farias, González, Llera, Mante, Nuevo Morelos, Ocampo, Tula, Xicoténcatl.
[d] Municipalities: Briseñas de Matamoros, Chavinda, Chilchota, Churintzio, Cotija, Ecuandureo, Jaconda, Jiquilpan, Maravatio, Nuevo Parangaricutiro, Numarán, Pajacuarán, Peribán, La Piedad, Régules, Los Reyes, Sahuayo, Tacitaro, Tangamandapio, Tangancicuero, Tanhuato, Tingüindin, Tocumbo, Venustiano Carranza, Villamar, Vistahermosa, Yurécuaro, Zamora, Zináparo.

production, concentrated in the regions of Amatitán, Arandas, Atotonilco, El Arenal, Tepatitlán and Tequila. Approximately 8% of the production comes from the south municipalities of Jalisco (mainly Ixtlahuacán del Rio, Jocotepec, Tlajomulco, Tonaya and Venustiano Carranza) and the remainder comes from the sum of Tamaulipas and Guanajuato states (NOM, 1994).

In previous studies, we have investigated the use of waste agave bagasse for the production of animal feed and fiberboards and the production of agave head waste from the tequila factory (Idarraga et al., 1999; Iñiguez et al., 2000). The purpose of the current study was to identify and evaluate the potential value of the leaves left in the field after agave heads were harvested on the basis to their fiber and sugar content. More specifically, the project had the following objectives: measure weight and determine the percentage of useful leaf material; evaluate the quantity of reducing sugars in the useful material; and evaluate the quality of the useful material for making paper.

## 2. Methods

### 2.1. Leaf collection and analysis

Several components of agave plants are shown in Fig. 2 at the time of harvest of agave heads from mature plantations in the region of Amatitán, Jalisco. Of the total plant dry matter, three primary components were considered in this project: (a) leaves, (b) heads, and (c) products derived from agave head polishing. The sum of these three components was considered as the total usable weight of the agave plant. The components were weighed individually. Materials coming from the agave head polishing (the final trimming of leaf bases to smooth the head) were divided into: (d) leaf bases ($P$) (material separated by the worker at the time of agave head polishing) and. (e) plant base (material between the head and the agave root). Leaves were subdivided into three groups: (f), young leaves, (g), mature leaves, and (h), dry leaves. Mature leaves were subdivided into: (i). whole fresh leaves ($B_1$, $B_2$, $B_3$) and (j), partially dry

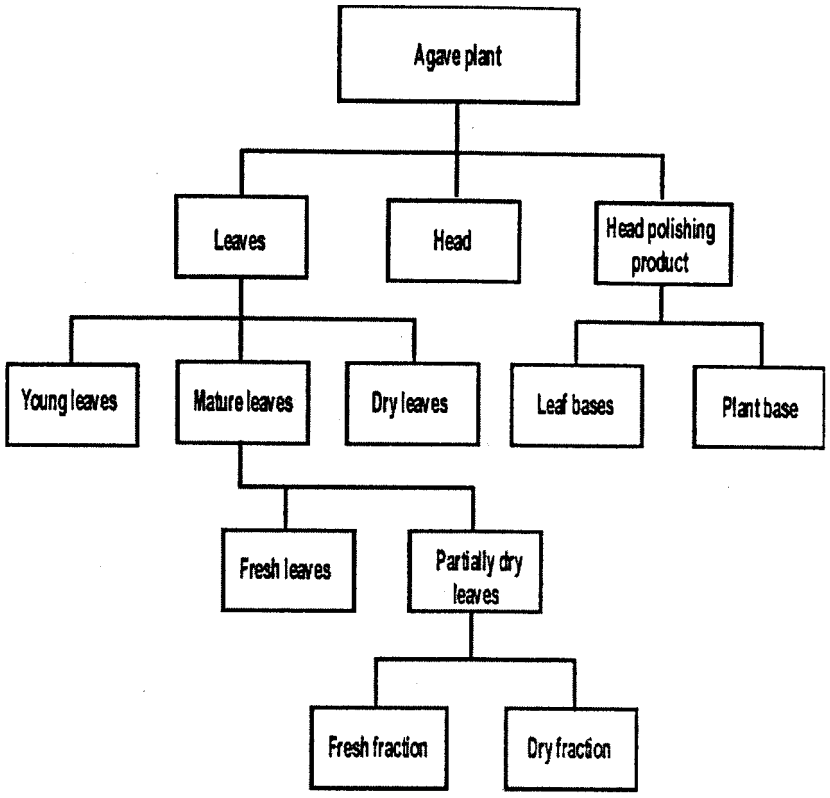


Fig. 2. Agave plant components collected for evaluation.

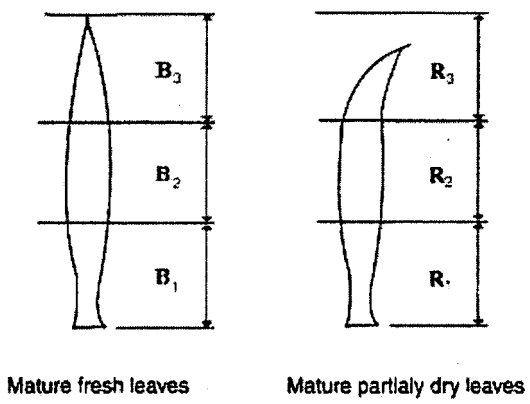


Fig. 3. Identification of leaf fractions

leaves. Partially dry leaves were subdivided in: (k), fresh fraction ($R_1$, $R_2$) and (1), dry fraction ($R_3$). The material of each fraction was weighed and its fraction in the total plant was determined. Useful material was considered as the sum of whole fresh leaves ($B_1$, $B_2$, $B_3$), fresh fraction of partially dry leaves ($R_1$, $R_2$) and leaf bases ($P$) (see Fig. 3).

### 2.2. Reducing sugar analysis

Reducing sugar concentration in the usable material of agave plants was calculated according to Lane–Eynon general volumetric method (AOAC, 1990). This is the official method to determine total reducing sugars contained in the raw material used for the production of tequila (DOF, 1992). Dry matter was determined by drying at 105°C for 24 h. Sugar contents were determined on whole fresh leaves $B_1$, $B_2$ and $B_3$, (where $B_1$ is the nearest part of the agave head and $B_3$ the leaf tip) and fresh fraction of partially dry mature leaves $R_1$, $R_2$, (where $R_1$ is the nearest fraction to the agave head). Sugar analysis was also carried out on the leaf bases (P). It was calculated as the percentage of those fractions ($B_1$, $B_2$, $B_3$, $R_1$, $R_2$ and P) in relation to the useful material as well as to the whole agave plant. Samples of fractions were chopped to small pieces with a kitchen knife and refrigerated (–20°C) for subsequent analyses.

### 2.3. Fiber analysis and manufacture of pulp and paper

Whole mature leaves were analyzed for their fiber content and the suitability of this fiber for pulp and paper production. Whole mature leaves were depithed in a lab depither similar to those used to obtain fibers from henequen leaves (Desfibradora del noroeste de Yucatán, S.A. de C.V. Av. Felipe Carrillo Puerto Km. 8.5 int. A. Carr. Mérida–Progreso. Mérida Yucatán 97110). This depither separated the fibrous bundles from the parenchymatic tissue. The parenchyma tissue, because of its

dimensions and chemical composition is not useful for making pulp and paper. Yields of fibrous bundles and pith were determined and fiber dimensions were measured. Fibrous material was analyzed for lignin, a-cellulose, hemicellulose and ash contents as well as for its extract content based on weight loss after ethanol-benzene, ethanol, and hot water extractions.

Fibrous bundles were subjected to a chemical soda pulping process using 20% NaOH (on oven dry weight) in a reactor (digester) at a fiber to liquor ratio of 1:10 at 175°C for 1.5 h. This time does not include the 2 h required to raise the reactor temperature from room temperature to the cooking temperature. The cooked material was washed thoroughly with water and then screened to remove the uncooked materials (rejects). Degree of delignification was evaluated by the Kappa number method. The screened pulp was refined in a Jokro mill until it reached levels of 40–450 ml Schopper Riegler (SR).

Standard sheets of 60 ± 1 g/m$^2$ were prepared from the unbleached pulp using a TAPPI standard hand sheets former. The sheets were air dried and then conditioned at 65% RH and 27°C for 2 h. The sheets were then tested for tensile strength, tear and burst index, and compared with sheets made from pine and eucalyptus pulps.

All the chemicals used were of analytical reagent grade. Methods of analysis were TAPPI Standards Test Methods (TAPPI, 1980).

## 3. Results and discussion

Distribution of different separated components from 30 different agave plants selected at random from 10 different agave plantations in the field study is shown in Table 2. The average wet weight of the whole agave plant was 53.2 kg (19.5–126.1 kg) and the average wet weight of the heads was 30.7 kg (8.2–66.3 kg). The standard deviation of weights of agave plants and heads showed a wide variation of 36.6 and 25.2, respectively, at harvesting time. This could be due to many factors such as soil characteristics, temperature, hours of sunlight, plant health, or insect attacks during the growing cycle. Moreover, the average ratio of wet head weight to wet whole plant weight was approximately 0.54 with a standard deviation of 0.111. This means there is a constant relationship between the wet head weight and the rest of the wet agave plant.

Based on data from Table 2, 54% of the wet agave plant represents the agave head, which is the raw material for tequila production. The rest of the wet plant or 46% is left in the fields and is not utilized. A 25% fraction of this residue corresponds to mature leaves (whole fresh leaves and fresh fraction of partially dry leaves), 4% to a dry fraction of mature partially dry leaves, 7% to dry leaves, 7% to leaf bases, 2% to young leaves, and 1% to head bases.

During the sampling process it was found that not all the agave plants contained young leaves. This is logical since agave plants used in this study were mature, so the content of young leaves was low. The fraction of young leaves was so small that it was not considered as part of a useful fraction for utilization. The same was true for dry leaves (7%) and the head bases (1%). These fractions were also too small for consideration for sugar or fiber production. Based on the data collected, only the material from leaf bases. whole fresh leaves, and the fresh fraction of partially dry mature leaves had enough mass to be considered for utilization.

Of the total wet weight of the agave plant, 54% thus corresponds to the agave head, 32% corresponds to materials which could be useful for sugar and fiber production which leaves only 14% of the wet plant without apparent utility. The useful fraction is composed of fresh fraction leaves $B_1$ . $B_2$ and $B_3$ of whole

**Table 2**
Distribution of components of wet agave plants

| Item | Average weight (kg) [a] | Average ratio of wet item weight to whole agave plant (%)[b] |
|---|---|---|
| Whole plant | 53.2 (36.6) | |
| Head | 30.7 (25.2)[c] | 54.0(11.1)[c] |
| Leaves | | |
| Young leaves | 0.7 (0.9) | 2.0 (2.6) |
| Dry leaves | 3.2 (2.0) | 7.0 (2.4) |
| Mature leaves | 14.1 (4.8) | 29.0 (6.0) |
| Fresh leaves ($B_1$, $B_2$, $B_3$) and fresh fraction leaves ($R_1$, $R_2$) | 12.3(7.0) | 25.0 (9.9) |
| Dry fraction leaves ($R_3$ ) | 1.9 (1.5) | 4.0 (1.1) |
| Head polishing | | |
| Leaf bases ($P$) | 3.6 (2.9) | 7.0 (2.2) |
| Plant bases | 0.9 (1.9) | 1.0 (2.4) |

[a] Arithmetic mean of 30 agave plants.
[b] From 30 agave plants.
[c] Standard deviation.

Table 3
Distribution of useable fractions of the agave plant after head removal

| Item | Mature leaves | | | | | Leaf bases' |
|---|---|---|---|---|---|---|
| | Whole fresh leaves[a] | | | Partially dry leaves[b] | | |
| Relation of useable fractions of agave plant | $B_1$ | $B_2$ | $B_3$ | $R_1$ | $R_2$ | $P$ |
| Arithmetic mean (%)[d] | 15.1(2,8)[c] | 9.2 (2.4) | 4.6 (1.0) | 30.8 (2.1) | 18.7 (2.1) | 21.6 (9.0) |
| Dry matter (%) | 19.2 | 25.0 | 24.5 | 19.8 | 22.5 | 25.4 |
| Total reducing sugars (%)[f] | 9.7 | 5.5 | 3.3 | 16.1 | 8.7 | 13.1 |

[c] Product of agave head polishing in harvesting time.
[a] Divided into three fractions. $B_1$ . $B_2$ and $B_3$, being $B_1$ the nearest part of the agave head and $B_3$ the leaf tip.
[b] Divided into two fractions. $R_1$ and $R_2$, being $R_1$ the nearest fraction of the agave head. Dry leaf tip fraction was discarded
[d] Arithmetic mean of 30 agave plants.
[e] Standard deviation.
[f] TRS on a wet basis.

mature leaves, fresh fraction leaves $R_1$ and $R_2$ of partially dry mature leaves and leaf bases $P$ (Table 2). Based on data from Table 3, 29% of useful material corresponds to whole fresh leaves, 49% to fresh fraction of partially dry leaves and 22% to leaf bases, leaving fresh fraction of partially dry leaves the highest proportion of the useful material. Also in Table 3 the total reducing sugars and the dry matter contents of those fractions are shown. Based on the data in this table the fractions with higher total reducing sugars (TRS) content were the fresh fraction of partially dry leaves stuck to the head ($R_1$) and the leaf bases ($P$) with a TRS content of 16.1% and 13.1%, respectively. The next fractions with higher TRS content were the nearest fraction to the head of whole fresh leaves ($B_1$) with a TRS content of 9.7% and $R_2$ the fraction between $R_1$ and the dry fraction of partially dry leaves with a TRS content of 8.7%. The fraction with less TRS content was the tip of the whole fresh leaves with a value of 3.3% It also appears from whole mature leaves data that the nearest fraction to agave head had less dry matter content, but higher TRS content (Table 3). For example. $B_3$, that is the leaf tip fraction, contained 3.3% of TRS while fraction $B_1$, that is the nearest fraction to agave head, contained 9.7% of TRS. The same trend occurred in fractions $R_1$ and $R_2$ from partially dry mature leaves. The nearest fraction to the agave head ($R_1$) had a TRS content of 16.1%, while the next fraction $R_2$ had a TRS content of 8.7%). This indicates that in agave plants exists a TRS concentration gradient toward the agave head, It can be stated that the average TRS concentration in agave heads is approximately 23%. TRS concentration in agave heads can vary from 16% to 28% (Valenzuela, 1997). Finally from Table 3 it can be deduced that 32% of the useful material from agave plants contains 9.4% of TRS when the percentages of distribution of useful fractions of agave plants and its sugar contents are considered. This amount of sugars corresponds to approximately 40% of the average TRS content of the agave heads used for tequila production. The potential amount of sugars that could be used after harvesting the agave heads can be calculated from data in Table 1 where there are reported 162,919,610 agave plants. based on data from the last census of the tequila regulating council (CRT, 1995). However, given the 32% useful material of agave plants not used, and considering the sugar content of this material (9.4%), the average weight of agave head (50 kg, Valenzuela, 1997) and the total consumption of agave heads in 1998 (672,130,177 kg CRT, 1998) it can be estimated that in 1998. 4,301.633 kg of useful material equivalent to 404,353 kg of sugar were not utilized. Mature agave leaves could thus be utilized as energy and fiber sources for animal feed or as a sugar source for alcohol production.

The yield (dry basis) of fibrous bundles after depithing whole mature leaves with a water content of 77.6% was 25.8%. Sugar cane bagasse has a fibrous bundle content of about 50% (Geplacea/Undp. 1990). The fiber length ($L$), fiber diameter ($D$) and the $L/D$ ratio of the fiber contained in the separated fibrous bundles as well a comparison of this fiber to softwood (pine) and hardwood (eucalyptus) fibers used in papermaking (Fernandez et al., 1999a; Dinwoodie, 1965) are shown in Table 4. The length and diameter of the agate fiber was higher for pine fiber. but shorter for eucalyptus fiber. and the $L/D$ relationship was smaller for agave fiber

Table 3
Characteristics of agave fiber in comparison to pine and eucalyptus fibers

| Fiber | Fiber longitude ($L$) (mm) | Fiber diameter ($D$) (μm) | $L/D$ relationship |
|---|---|---|---|
| Agave | 1.58 | 25.00 | 63.00 |
| Pinus | 4.20 | 39.00 | 108.00 |
| Eucalyptus | 0.94 | 18.29 | 97.98 |

Table 5
Chemical composition of agave fibrous bundle and agave head in comparison to pine and eucalyptus fibers

| Item(%) | Agave fibrous bundle | Agave head [a] | Pinus ocarpa [b] | Eucalyptus[c] |
|---|---|---|---|---|
| Ethanol–benzene extracts | 5.3 | 3.1 | 6.5 | 3.8 |
| Ethanol extracts | 2.9 | 5.5 | N.D.[d] | 5.5 |
| Hot water extracts | 5.8 | 3.9 | 1.8 | 0.3 |
| Lignin | 15.9 | 16.8 | 27.4 | 18.2 |
| a -cellulose | 64.8 | 64.9 | 47.5 | 49.4 |
| Hemicellulose | 5.1 | 5.5 | 16.2 | 21.2 |
| Ash | 1.0 | 2.0 | 0.20 | 0.4 |

[a] Sanjuán (1987).
[b] Zavala et al. (1998).
[c] Fernandez et al. (1999b).
[d] N.D. Not determined.

Table 6
Properties of agave paper sheets

| Sheets | Refination degree (°SR) | Property | | |
|---|---|---|---|---|
| | | Breaking length (m) | Tear index (m $Nm^2/g$) | Burst index (kPa $m^2/g$) |
| Agave | 45 | 6333 | 6.9 | 2.2 |
| Pine[a] | 45 | 6925 | 8.2 | 5.2 |
| Eucalyptus[b] | 40 | 5367 | 7.4 | 4.5 |

[a] Zavala et al. (1998).
[b] Fernandez et al. (l999b).

than pine and eucalyptus fibers. A chemical composition of agave fibrous bundles compared to pine and eucalyptus is shown in Table 5 (Fernandez et al., 1999b; Zavala et al., 1998). The a -cellulose content of agave fiber was higher than either of the woods, which meant that agave leaves could give a high yield of bleached chemical pulp. The lignin content of agave leaves fiber was lower than either pine or eucalyptus. Lower lignin content would result in less chemicals used in the pulping process. Because of the chemical composition of agave fibrous bundles, a pulping process used for hardwoods might be appropriate for pulping agave (Taques et al., 1998).

Strength properties of agave sheets are given in Table 6. Agave sheets have a higher breaking length than eucalyptus sheets but lower than pine sheets. Tear and burst index values were lower for agave sheets than for pine and eucalyptus sheets. This means that it might be best to mix agave leaf pulp with other pulps to obtain a good quality paper. The fiber content of mature agave leaves is too low for a good fiber source for pulp and paper production. For the production of 1 t of pulp 38.4 t of mature agave leaves would be needed. Economic viability is only possible in small-scale projects to produce handmade or other value added papers.

## 4. Conclusions

Based on the results of this study 54% of the wet agave plant is the agave head which serves as the raw material for tequila production while the rest of the wet plant is left in the fields and is not utilized. From this residue, only the material from leaf bases, whole fresh leaves, and the fresh fraction of partially dry mature leaves have enough mass to be considered for sugar and fiber production. The fractions with higher TRS content were the fresh fraction of partially dry leaves stuck to the head and the leaf bases with a TRS content of 16.1% and 13.1%, respectively. Handmade paper sheets made from agave leaf fibers had a breaking length comparable with paper made from both pine and eucalyptus fiber, but the tear index and burst index were lower than for the other two products. Agave leaf fibers could be potentially utilized for many paper products including handicraft, geotextiles, filters, packaging and composites.

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