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Papermaking as Potential Use of Fibers from Mexican *Opuntia ficus-indica* Waste

Elaboración de papel como uso potencial de fibras a partir de desechos de *Opuntia ficus-indica*

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ABSTRACT

The papermaking potential of *Opuntia ficus-indica* (*OFI*) waste fibers was studied in this research. Alpha cellulose, lignin, hollocellulose, ethanol/benzene extractives and ash content were determined as 53.7±0.1%, 2.4±0.3%, 61.6±5.7%, 7.1±0.3% and 26.4±0.1%, respectively. The average fiber length, width, lumen, and cell wall thickness were found to be 1.1±0.3 mm, 18.8±6.1µm, 12.1±5.4 µm, and 4.3±1.0 µm. Soda pulping was conducted using 20 and 28% sodium hydroxide, cooking temperatures of 160 and 175 °C, cooking times of 60 and 120 min, and liquor- to fiber ratio of 9:1. Soda pulping with 28% sodium hydroxide, 175 °C and 120 min showed a lower Kappa number of 29.60±1.7 and a total yield of 32.2±1.6%. In general, tensile strength index (36.0±5.0 Nm/g), stretch (1.7±0.3%), breaking length **(**3.7±0.5 km), burst index (3.2±0.4 KPa.m^2/g), tear index (7.3±0.0 mN.m^2/g), folding endurance (166 times) and porosity (> 120 s) of *OFI* pulp were comparable with wood and non-wood pulps.

Keywords: *Opuntia ficus-indica* waste, Fiber morphology, Pulp properties, Paper properties, Non-wood paper.

RESUMEN

En esta investigación se estudió el potencial del uso de desechos de fibras de *Opuntia ficus-indica (OFI)* para elaborar papel. La estrategia metodológica que se siguió fue: caracterización de materia prima, análisis morfológico de las fibras, pulpeo soda mediante un diseño completamente al azar con un arreglo factorial 2^3, refinación de pulpa, formación y caracterización de hojas. El contenido de alfa-celulosa, lignina, holocelulosa, extractivos en etanol/benceno y cenizas fue de 53.7±0.1%, 2.4±0.3%, 61.6±5.7%, 7.1±0.3% y 26.4±0.1%, respectivamente. El promedio de la longitud de las fibras, ancho, lumen y espesor de la pared celular fue 1.1±0.3 mm, 18.8±6.1µm, 12.1±5.4 µm, 4.3±1.0 µm. Las condiciones de pulpeo fueron: 20 y 28% de hidróxido de sodio, 160 y 175 °C, durante 60 y 120 min y una relación licor/fibra de 9:1. El proceso de pulpeo con 28% de hidróxido de sodio, 175°C y 120 min mostró un menor número de Kappa (29.60±1.7) y un rendimiento total de 32.2±1.6%. En general el índice de tensión (36.0±5.0 Nm/g), deformación (1.7±0.3%), longitud de ruptura (3.7±0.5 km), índice de estallido (3.2±0.4 KPa. m^2/g), índice de rasgado (7.3±0.0 mN.m^2/g), resistencia al doblez (166 times) y porosidad (> 120 s) de la pulpa obtenida fueron comparables con valores reportados para pulpa de fibras maderables y no maderables.

Palabras clave: Desecho *de Opuntia ficus-indica*, Morfología de las fibras, Propiedades de la pulpa, Propiedades de papel, Papel de fibras no maderables.

INTRODUCCIÓN

Nowadays, there is a trend to substitute single-use plastic packaging for environmental-friendly packaging due to the growing awareness of the environmental damages associated (Herbes *et al.*, 2018). Paper and paperboard are tagged as environmental-friendly packaging and can substitute several kinds of plastic.

In 2016, the world production of paper and paperboard was 409 million tons (FAO, 2016). The paper industry is also associated with environmental problems because of its high energy consumption and emissions, high consumption of chemicals and it is a sector with highly intensive use of natural resources, like wood, non-wood fibers, and water (Wang *et al.*, 2016; IEA, 2017; Man *et al.*, 2018; Sun *et al.*, 2018). The environmental problems related to raw materials for the paper industry require search and study of diverse alternatives that mitigate their effects. The principal natural resource used to produce paper is pulp fibers, which can be obtained from wastepaper pulp, wood pulp and pulp from other fibers. The use of wood to produce paper has caused a great deforestation problem. As a result, in recent years, several research groups have investigated the technical viability of using pulp derived from agro-industry waste, straw, bagasse and other non-wood fibers (Rainey and Covey, 2016;

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Gonzalo *et al.*, 2017; Kaur *et al.*, 2017). Commercially, pulps derived from straw, bagasse, bamboo, esparto, reeds, grass, flax and hemp are used for paper production (FAO, 2017). The viability for the use of non-wood fibers as a raw material for papermaking will depend on the chemical and morphological properties of the fibers, as well as the availability of this resource, its proximity to the paper factories and, as it has recently been emphasized, the amount of water used during the crop irrigation process (Man *et al.*, 2018). Therefore, it is necessary to continue exploring the use of other non-wood fibers as a raw material for papermaking.

In Mexico, arid and semi-arid lands constitute 49.20% of the national territory. Mexico is also considered one of the main diversity centers of cacti. In fact, there are 586 species of cacti and the highest number of native species in Mexico (Ortega-Baes *et al.*, 2010). The cacti of greatest economic importance in the world are the *Opuntia* spp., commonly named "nopales" in Mexico (Inglese *et al.*, 2018). The economic importance of nopales resides in its nutritional value, functional and therapeutic properties, and its potential use in the cosmetics and bioenergy industries (Ciriminna *et al.*, 2019). The worldwide-cultivated species of *Opuntia* genus is *Opuntia ficus-indica* (*OFI*). *Opuntia* spp. are characterized by growing in extreme temperature conditions (-10 to 70 °C) and scarce water availability (50 mm or less annual precipitation) (Nobel and Bobich, 2002; Ginestra *et al.*, 2009; Inglese *et al.*, 2018). They also need 20 to 30 days to develop a harvestable size and they have very low lignin content (3.6 to 16%) (Rodriguez-Felix and Cantwell, 1988; Malainine *et al.*, 2003). Mexico has a great availability of wild (83) and cultivated (4) species of *OFI* (Guzmán *et al.*, 2003). The cultivated area of nopales in Mexico is around 77,878 ha (SIAP 2017). Actually, there is a big interest to valorize nopales, for example, it has been used as biofuel feedstocks and coagulant in wastewater bioremediation (Vishali and Karthikeyan, 2015; Yang *et al.*, 2015; Barbera and Gurnari, 2018). The production of nopales for foodstuff uses generates wastes at agricultural and industrial levels, for example, in Mexico City, each year dethorning process of nopal generates around 40,000 tons of waste (Marin-Bustamante *et al.*, 2017). This can become a focus of contamination (insects and microorganisms). Recently, several publications reveal the possible applications of nopales in science and materials engineering. About this, three research topics were identified: I) Activated charcoal production from *OFI* (Ouhammou *et al.*, 2017), II) *OFI* as polymer matrix reinforcement (Malainine *et al.*, 2004; Malainine *et al.*, 2005; Greco *et al.*, 2013; Greco and Maffezzoli, 2015) and III) *OFI* as raw material for papermaking (Mannai *et al.*, 2016; Mannai *et al.*, 2018). The last topic will be explored in this study. It is important to mention that during the construction of the theoretical framework of this research, only two papers about this topic were found. Tunisian *OFI* fibers have high cellulose (53%) and low lignin (4.8%) content with long and thick fibers. The Tunisian fibers have chemical and morphological characteristics favorable for papermaking. *OFI* pulp was obtained by two methods, soda-hydrogen peroxide (80.8% of yield) and soda-anthraquinone (41.1% of yield) process. The paper sheets obtained, by both methods, had acceptable physical properties. In agreement with this data, *OFI* fibers can be used as raw material for papermaking (Mannai *et al.*, 2016; Mannai *et al.*, 2018). However, it is necessary to continue exploring the properties of *OFI* fibers from other regions and other processes.

The aim of the present work was to explore an alternative for processing Mexican *Opuntia ficus-indica* waste to produce paper as a strategy for further valorization. Chemical characterization of the raw material was conducted and the morphological analysis of the fibers. Optimal parameters of the paper preparation procedure based on the soda process were established. Finally, laboratory paper sheets were manufactured and subjected to mechanical testing.

MATERIALS AND METHODS

Raw material

OFI waste cladodes were obtained from a commercial cultivation area in La Victoria, Hermosillo, Sonora (México). OFI waste cladodes did not have quality for food consumption or processing. The cladodes were two years old and 89.23% moisture content. The cladodes size was 41.8±7.5 cm in length, 14.7±2.7 cm in width, and 797.0±287.8 g in weight.

Preparation of raw material

Cladodes were shredded to reduce the particle size and be ready to be ground by a hammer mill (own design) equipped with two different screen sizes (10.82 and 3.16 mm). The ground material was fully washed with water, and sundried to constant moisture. Finally, fibers bundles were manually selected and stored at 20 °C.

Characterization of the raw material

Chemical properties of cladodes were determined in accordance with TAPPI standards for different components such as moisture (TAPPI 412 om-02), lignin (TAPPI 222 om-15), α-cellulose (TAPPI 203 cm-09), water-soluble (TAPPI 207 om-08), 1% NaOH soluble (TAPPI 212 om-18), extractives (TAPPI 204 om-17), and ashes (TAPPI 211 om-16). Holocellulose was quantified according to Mannai *et al*. (2018).

Morphological studies

The fibers morphology was characterized according to TAPPI 259 om-16. Previously, fibers were treated with 28% NaOH at 176-199 °C for 1 h under constant stirring to remove the cellular content. Characteristics of fibers, including length (L), diameter (D), wall thickness (W), and lumen width (l) were measured with an optical microscope (DMR Leica, Wetzlar, HE, Germany). Additionally, the Runkel ratio (2W/l), flexibility coefficient (l/D), slenderness coefficient (L/D), and stiffness coefficient (2W/D) were obtained (Ogbonnaya *et al*., 1997; Ververis *et al*., 2004).

Pulping

Fibers were cooked with NaOH in a 1 L laboratory scale, oil heated, and cylindrical batch reactor (own design).

The cylindrical reactor containing fibers, was introduced in the oil bath when the temperature was 80 °C. The time to reach cooking temperatures was 1 hour after which, it was kept constant. Cooking conditions were as stated in Table 1. The cooked material was filtered (325 mesh) and washed to remove the wastewater.

Table 1. Cooking conditions for *OFI* waste fibers pulping.
Tabla 1. Condiciones de cocción para la pulpa de fibras de desecho de *OFI*.

Parameter	Low value	High value
OFI fiber weight, g	34.10±0.23	
Liquid/solid ratio	9/1	
Active alkali (NaOH), %	20	28
Temperature, °C	160	175
Time, min	60	120

Pulp beating

The pulp was beaten in a Jokro-Muhle refiner (P.J & Söhne Gmbh, Düren, NW, Germany) at 150 rpm for 25 min. Next, the pulp was screened using a 0.15 mm aperture size sieve (Lorentzen and Wettre, Kista, STO, Sweden) to remove the uncooked material and to evaluate the screened and rejected yield. Additionally, total yield was calculated by adding accepted and rejected yields according to TAPPI 274 sp-13. Kappa number of the pulp was determined according to TAPPI 236 om-13.

Paper sheets formation and characterization

OFI waste paper sheets were prepared with a sheet former used according to TAPPI 205 sp-18. The tensile index according to TAPPI 494 om-13, burst index in accordance to TAPPI 403 om-16, tear index consistent to TAPPI 414 om-12, breaking length and stretch in accordance to TAPPI 494 om-13, folding consistent to TAPPI 423 om-16 and porosity according to TAPPI 460 om16 were determined.

Statistical analysis

For cooking conditions optimization, a completely randomized design with a 2^3 factorial arrangement was used. The factors were temperature (160 and 175 °C), reaction time (60 and 120 min), and active alkali (20 and 28%), while the response variables were screening yield, rejects, total yield, and Kappa number. For paper sheet characterization, descriptive statistics and simple linear regression analyses were used.

RESULTS AND DISCUSSION

Characterization of the raw material

Figure 1 shows the chemical composition of *OFI* waste. The content of lignin in *OFI* waste is 2.4±0.3%, much lower than values reported for any wood and non-wood material used for papermaking. In general, non-wood plants have less lignin than woods. Our results exhibit that lignin content was in the range (2.51-4.80%) reported for *OFI* cladodes (Malainine *et al.*, 2003; Mannai *et al.*, 2016; Cheikh Rouhou *et al.*, 2018). In addition, our results showed that *OFI* waste cladodes has only 8.30, 18.0, and 10.04% of lignin, that other authors reported for chili paper residue, rice straw, sugarcane bagasse, respectively (Gonzalo *et al.*, 2017; Mulyantara *et al.*, 2017; Kaur *et al.*, 2018). The low lignin content is convenient since one of the principal targets in the pulping process is the elimination of lignin from the fibers. *OFI* waste had a holocellulose content of 61.6%, value also close to that reported for *OFI* trunks (64.5%) and rice straw (60.7%), but lower than that reported for chili paper residue (88.0%) and sugarcane bagasse (76.7%) (Rodríguez *et al.*, 2008; Zhao *et al.*, 2008; Mannai *et al.*, 2016; Gonzalo *et al.*, 2017). The *OFI* waste cellulose content was 53.7±1.0%, very similar to that reported for *OFI* trunks (53.6%) and miscanthus residue (51.0%), higher than the reported for rice straw (41.2%), and lower than that reported for sugarcane bagasse (44.9%) (Zhao *et al.*, 2008; Rodríguez *et al.*, 2010; Gonzalo *et al.*, 2017; Mannai *et al.*, 2018). Our results contrast with the 21.60% published by Malainine *et al.* (2003) and the 26.70% published by Cheikh Rouhou *et al.* (2018) for *OFI* cladodes. This apparent lack of agreement can be attributed to the season and age of the plants and edaphic factors at the cultivation sites. The ethanol-benzene extractables content in the *OFI* waste cladodes was 7.10±0.3%. This value is very close to that reported for *OFI* trunks (9.8%) but higher than the published data for sugarcane bagasse (3.2%), and chili paper residue (2.2%) (Zhao *et al.*, 2008; Mannai *et al.*, 2016; Gonzalo *et al.*, 2017; Cheikh Rouhou *et al.*, 2018). In agreement with this result, some *OFI* waste cladodes components will precipitate upon pulping and leave stains in the resulting paper sheets (Rodríguez *et al.*, 2008). Cladodes contain different compounds that can be considered extractables. These compounds include chlorophyll, flavonoids like kaempferol, quercetin, methyl-3-kaempferol, kaempferol 3-methyl ether, quercetin 3-methyl ether, narcissin, (+)-dihydrokaempferol, (+)-dihydroquercetin and eriodictyol; terpenoids like (6S, 9S)-3-oxo-α-ionol-β-D-glucopyranoside and corchoionoside C; carotenoids like cryptoxanthin, β-carotene and lutein (Jaramillo-Flores *et al.*, 2003; Lee *et al.*, 2003; Stintzing and Carle,

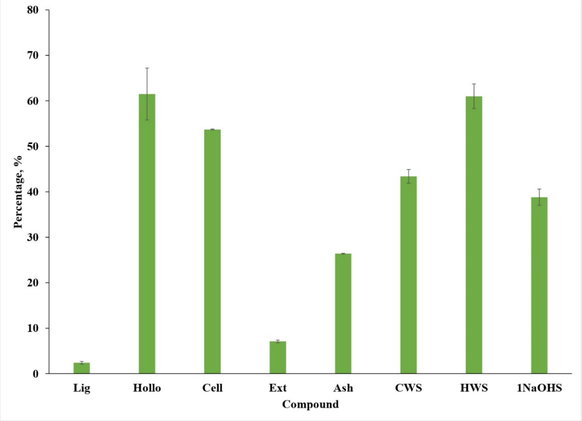

Fig. 1 Chemical characterization of *Opuntia ficus-indica*. Lig: lignin; Hollo: hollocelulose; Cell: α-cellulose: Ext: extractives; CWS: cold-water solubles; HWS: hot water solubles; 1NaOHS: 1% NaOH solubles.
Fig. 1. Caracterización química de *Opuntia ficus-indica*. Lig: lignina; Hollo: hollocelulosa; Célula: α-celulosa: Ext: extractivos; CWS: solubles en agua fría; HWS: solubles en agua caliente; 1NaOHS: solubles en NaOH al 1%.

2005). Additionally, some pyrones (opuntioside I, opuntioside II, opuntioside III, 4-eyhoxyl-6-hydroxymethyl-αpyrone,7-O-β-D-glucopyranosyl-(1→4)-β-D-glucopyranoside) and fatty acids have been extracted from other Opuntias (Qiu *et al.*, 2002; Qiu *et al.*, 2007).

The cladodes ashes content was 26.4±0.1%, close to the 23.31% reported for Cheikh Rouhou *et al*. (2018) a little higher than 19.60% reported by Malainine *et al*. (2003) and higher than the 5.50% reported by Mannai *et al*. (2018) for *OFI*. This result can be attributed to the presence of different minerals such as calcium, potassium, iron, and magnesium, being the most abundant calcium oxalate, which is water-insoluble (Stintzing and Carle, 2005; Rodríguez-Garcia *et al.*, 2007). In fact, it is higher than the value reported for wood and non-wood materials used for papermaking. The content of substances soluble in hot water in *OFI* waste cladodes was 61.0±2.7 %, higher than the reported for the same species of genus Opuntia (25%-cladodes and 36.3%-trunks), eucalyptus, sugarcane bagasse, cotton stalks, and rice straw (Jiménez *et al.*, 2007; Rodríguez *et al.*, 2008; Mannai *et al.*, 2016; Mannai *et al.*, 2018). This property indicates that different components of cladodes such as inorganic compounds, tannins, gums, sugars, and starches will be drawn with hot water. Finally, 1% NaOH solution extracted low molecular weight carbohydrates of *OFI* waste cladodes. The content of substances soluble in 1% NaOH was 38.8±1.80. This value is higher than the 29.6% reported for *OFI* trunks (Mannai 2016). Zhong *et al*. (2010) and Majdoub *et al*. (2001) reported arabinose, galactose, galacturonic acid, glucose, glucuronic acid, xylose, rhamnose, uronic acid like low molecular weight carbohydrates in *OFI* cladodes.

In sum, the chemical properties of *OFI* waste cladodes showed the suitability of this raw material as a non-wood source for papermaking.

Morphological studies

Figure 2 shows an *OFI* waste fiber and Table 2 its morphological characteristics. The size for these fibers was: length 1.1±0.3 mm, diameter 18.80±6.1 µm, wall thickness 4.30±1.0 µm, and lumen width 12.10±5.4 µm. In general, the *OFI* waste fibers are longer than the data published for other *Opuntia* species. Gibson (1977) reported that the size for *Opuntia* species falls in the range of 629 to 330 µm in length, 38.3 to 16.20 µm in diameter, and 5.50 to 2.60 µm in wall thickness. The length of the *OFI* waste fibers is higher than the reported for *OFI* cladodes fibers (0.74 µm) and *OFI* trunks fibers (0.76 µm) (Mannai *et al.*, 2016; Mannai *et al.*, 2018). Fiber length of *OFI* waste is in the range of hardwoods (0.7 -1.5 mm) and within the range (1.0-2.5 mm) of non-wood fibers such as sugarcane bagasse, rapeseed straw, kenaf and sunflower stalk. The length of *OFI* waste fibers was similar to that of rapeseed straw, sunflower stalk and eucalyptus, but smaller than sugarcane bagasse, kenaf and pine fibers (Ashori *et al.*, 2006; Khristova *et al.*, 2006; Mazhari Mousavi *et al.*, 2013; Gominho *et al.*, 2014; Gulsoy and Ozturk, 2015; Rudi *et al.*, 2016). Fibers with low (<1) Runkel ratio are assessed to be efficient for sheet formation by the combination effect of thin walls and wide lumen. On the other hand, fibers with high Runkel ratio (>1) are considered not to be efficient for sheet formation because these are indicative of tubular and noncollapsible fibers, providing a low bonding area (Villar *et al.*, 2009). In agreement with the obtained Runkel ratio for *OFI* waste fibers (0.80±0.44), these are suitable as raw material for papermaking.

Figure 2. *Opuntia ficus-indica* fibers.
Figura 2. Fibras de *Opuntia ficus-indica*.

Table 2. Morphological characteristics of *OFI* waste fibers.
Tabla 2. Características morfológicas de las fibras de desecho de *OFI*.

Parameter	Value
Fiber length, mm	1.1±0.3
Fiber diameter, µm	18.8±6.1
Fiber lumen width, µm	12.1±5.4
Fiber wall thickness, µm	4.3±1.0
Elasticity coefficient	0.60±0.11
Slenderness coefficient	60.9±17.8
Runkel ratio	0.80±0.44

OFI results are presented as mean ± SD of seven replicates.

According to the flexibility ratio, there are four groups of fibers (San *et al.*, 2016): I) High elastic fibers with an elasticity ratio >75, II) Elastic fibers with an elasticity ratio between 55-75, III) Rigid fibers with an elasticity ratio between 30-50, and IV) Highly rigid fibers with an elasticity ratio <30. The flexibility ratio of *OFI* fibers was found to be 0.60±0.11; therefore, these are elastic fibers. The flexibility ratio shows the degree of fiber bonding in paper sheet (Anupam *et al.*, 2016). Because *OFI* fibers have efficient elasticity, they are suitable for paper production. The slenderness ratio shows the fibers ability to bond with each other. If fibers have a high slenderness ratio, then these can collapse easily resulting in good surface contact and inter-fiber bonding during the formation of paper. The recommendable slenderness ratio for fibers to papermaking is > 30. Along with the slenderness ratio (60.9±17.8), *OFI* waste fibers are suitable to make paper.

In summary, anatomical ratios of *OFI* waste fibers show the suitability of these as a non-wood source for papermaking.

Pulp analysis

Figure 3 presents the general process of *OFI* waste fibers pulping, and Table 3 shows the cooking conditions and pulp properties obtained from *OFI* waste fibers. The screening yields were in the range of 14.0 to 41.8% and time and % NaOH showed statistically significant differences ($p \le 0.05$) on this property. This variable was inversely proportional to reaction time. Surprisingly, even when *OFI* waste cladodes contain low levels of lignin, the pulp yield was directly proportional to % NaOH. This behavior should be more attributed to the composition of the lignin itself than to its concentration. The exact proportion of syringyl, guaiacyl and p-hydroxyphenyl units of lignin in *OFI* fibers has not been reported. However, considering our results, we can hypothesize that the guaiacyl units are in a greater proportion in the lignin of *OFI* waste fibers because these structures are more difficult to hydrolyze during the alkaline pulping. Guaiacyl units have one methoxyl group and a free carbon-5 available for carbon–carbon inter-unit bonds, which make them resistant to lignin depolymerization in pulping. In contrast, siryngyl units are relatively unbranched and have a lower degree of condensation and therefore are easier to delignify (Del Río *et al*., 2007).

From all the pulping conditions, treatments 2 and 6 applied the lowest level of time and the highest level of % NaOH giving screening yields of 41.8 and 36.3%, respectively. However, the pulps delignification degree obtained with these treatments was not enough. Rejects values ranged between 0.1 and 29.5%. According to this variable, the samples were grouped in two categories. In the first, the rejects were up to 21.0% and the pulps obtained with the treatments 1, 3, 4, 5 and 7 classified in this category. In the second category, the rejects were lower than 0.2% and the pulps obtained with the treatments 2, 6 and 8 classified here. Pulps obtained from treatments 2 and 6 showed the highest pulping yield but did not have an adequate delignification. The total yield for *OFI* waste pulp was in the range of 32.2 to 45.0%. These values are in the range of yields reported for non-wood species such as rice straw, Leucaena, and morning glory (Dutt *et al*., 2004; López *et al*., 2008; Rodríguez *et al*., 2008).

Figure 3. General process of *Opuntia ficus-indica* pulping. a) Raw material; b) Hammer mill c) Dry raw material; d) Fiber bundles; e) Cooking; f) Beating; g) Screening; h) Paper sheets forming; i) Final paper sheet.
Figura 3. Proceso general de despulpado de *Opuntia ficus-indica*. a) Obtención de la materia prima; b) Reducción de materia prima; c) Secado de materia prima; d) Obtención de haces de fibrosos; e) Cocción; f) Refinación; g) Depuración; h) Formación de hojas de papel; i) Hoja de papel.

Table 3. Cooking conditions and properties obtained from *OFI* waste pulp.
Tabla 3. Condiciones de cocción y propiedades de la pulpa obtenida residuos de *OFI*.

Treatment	Temperature, °C	Time, min	NaOH, %	Screening yield, %	Reject, %	Total yield, %	Kappa number
1	160	60	20	24.0±0.9	21.0 ± 4.0^{b}	45.0±4.9	70.0 ± 0.4^{a}
2	160	60	28	41.8±2.8	0.2 ± 0.3^{a}	42.0±2.5	59.6 ± 2.1^{ab}
3	160	120	20	19.7±1.3	23.3 ± 2.2^{b}	43.0±0.8	53.0 ± 1.9^{bc}
4	160	120	28	20.6±0.4	21.5 ± 1.1^{b}	42.1±1.5	47.1 ± 0.3^{bd}
5	175	60	20	18.2±4.6	22.1 ± 3.9^{b}	40.4±0.7	37.3 ± 0.2^{de}
6	175	60	28	36.3±7.7	0.1 ± 0.0^{a}	36.4±7.6	38.5 ± 8.8^{df}
7	175	120	20	14.0±4.8	29.5 ± 1.5^{b}	43.5±6.3	42.4 ± 1.7^{cdg}
8	175	120	28	32.1±1.6	0.2 ± 0.0^{a}	32.2±1.6	29.6 ± 1.7^{efg}

Results are presented as mean ± SD of two replicates.
Means within a column followed by different letters differ significantly at $p < 0.05$.

The Kappa number ranged between 29.6 and 70.0 for all the pulp treatments. This number expresses the lignin content in the pulp and how easy it is bleached. Severe pulping conditions favored the Kappa number. The pulp with the highest Kappa number was obtained with 20% NaOH, 60 min and 160 °C (treatment 1). On the other hand, the pulp with the lowest Kappa number was obtained with treatments 5 and 8, which consisted of 20% NaOH for 60 min at 175 °C, and 28% NaOH for 120 min at 175 °C, respectively. Kappa numbers for both treatments were transformed into residual lignin giving 4.8 and 3.8% for treatments 5 and 8, respectively. Both values are in the range (2-5%) suggested for materials suitable for bleaching pulps (García-Hortal *et al.*, 2008)**.** Nevertheless, the lignin residual value for the pulp of treatment 5 was close to the upper limit and the rejects were high. On the other hand, the lignin residual value for the pulp of treatment 8 was at good level and the rejects were low. According to these results, the best conditions for pulping were 28% NaOH for 120 min at 175 °C (treatment 8). In conclusion, *OFI* waste fibers exposed with a soda pulping process (28% NaOH, 175 °C and 120 min) produced pulps with a low Kappa number.

Characterization of paper sheets

Figure 3i exhibits a paper sheet of *OFI* waste pulp and Table 4 shows the paper sheet properties produced with the *OFI* waste pulp. Tensile strength index is a property that is influenced by three factors: fiber length, fiber strength and fiber bonding ability. Generally, softwood pulps have a higher tensile index than hardwood ones, and non-wood pulps have a low tensile strength index in comparison with softwood and hardwood pulp. In agreement with this, tensile strength index for *OFI* waste pulp (36.0±5.0 Nm/g) is low in comparison with that of pine (76.1 Nm/g) and poplar (40.40 Nm/g) pulps. However, it is in the same range of pulp derived from non-wood fibers like sugarcane bagasse (38.2 Nm/g), rice straw (41.7 Nm/g) and sunflower stalks (43.64 Nm/g) (Khristova *et al.*, 2006; Fišerová and Gigac, 2011; Sarwar Jahan *et al.*, 2012; Mazhari Mousavi *et al.*, 2013; Danielewicz *et al.*, 2015).

The burst index is a measure of the resistance to rupture of paper. Several authors suggest that this property is intimately related to the cell wall thickness and length of the fibers (Horn, 1974). The longer the fibers are, the greater the burst index. On the other hand, the greater the cell wall thickness of the fiber, the lower the burst index. *OFI* waste burst index (3.2±0.4 kPam^2/g) is higher than the reported by Mannai et al. (2018) for *OFI* papers (5.8 kPam^2/g). However, the fibers (0.76 mm) used for Mannai *et al* (2018) were shorter than the fibers used for us on this research. This difference could be influenced by the conditions of the pulping process. The *OFI* waste burst index is in the range published for wood and non-wood papers sheets like *Agave tequilana* (2.2 kPam^2/g), rapeseed straw (3.42 kPam^2/g), sunflower stalks (2.89 kPam^2/g), kenaf (3.2 kPam^2/g), pine (2.6 kPam^2/g) and poplar (2.3 kPam^2/g) (Khristova *et al.*, 1998; Iñiguez-Covarrubias *et al.*, 2001; Boeva *et al.*, 2007; Villar *et al.*, 2009; Koray Gulsoy, and Eroglu 2011; Mazhari Mousavi *et al.*, 2013). Tear index reflects the work needed to tear paper and it has a relationship with the length of the fibers (Shmulsky and Jones, 2011). *OFI* waste paper sheets presented a tear index lower than the reported for *OFI* cladodes sheets (12 mNm^2/g) and *OFI* trunks sheets (19.2 mNm^2/g). These differences can be attributed to *OFI* fiber length, *OFI* cladodes fibers were 0.74 mm and *OFI* trunks fibers were 0.76 mm (Mannai *et al.*, 2016; Mannai *et al.*, 2018). *OFI* waste paper sheets tear index was in the range published for paper sheets of wood and non-wood fibers like *Agave tequilana* (6.9 mNm^2/g), sugarcane bagasse (6.3 mNm^2/g), and poplar (4 mNm^2/g) (Iñiguez-Covarrubias *et al.*, 2001; Khristova *et al.*, 2006). This result is consistent with the reported for paper made with a fiber of similar length as that of rapeseed straw (1.03 mm; 4.90 mNm^2/g) and sunflower stalks (1.27 mm; 6.0 mNm^2/g) (Khristova *et al.*, 1998; Ai and Tschirner, 2010; Mazhari Mousavi *et al.*, 2013). However, Mannai *et al*. (2018) indicated a higher tear index (12 mNm^2/g) for *OFI* cladodes. The breaking length expresses the length of the paper, which would just break under its own weight. It is a measure of the resistance of paper to direct tension under specific conditions of rate of extension (Ghasemian *et al.*, 2012). Higher values are preferred for a good quality paper. The breaking length obtained for *OFI* waste pulps was 3.7±0.5 km. This value is higher than the reported for *OFI* cladodes paper sheets (1.5 km), *OFI* trunks paper sheets (1.9 km), and rice straw paper sheets (2.7 km) but lower than the reported for *Eulaliopsis binata* paper sheets (5.3 km) (Tyagi *et al.*, 2004; Rodríguez *et al.*, 2008; Mannai *et al.*, 2018).

Table 4. Properties of paper sheets obtained from *OFI* waste pulp.
Tabla 4. Propiedades de las hojas de papel obtenidas a partir de pulpa residual de *OFI*.

Property	Value
Tensile index, Nm/g	36.0±5.0
Stretch, %	1.7±0.3
Breaking length, km	3.7±0.5
Burst index, KPa.m^2/g	3.2±0.4
Tear index, mN.m^2/g	7.3±0.0
Folding endurance, times	166
Gurley's porosity, s	> 120

Results are presented as mean ± SD of ten replicates

Stretch reflects the capability of paper to conform a desired contour, or to resist nonuniform tensile stress and it is important in all types of papers. The stretch % obtained for *OFI* waste paper sheets was higher (1.7±0.3%) than the value reported for sheets produced with vine shoots pulp (1.49%) and *Cyanara cardunculus* (1.40%) but lower than the published data for sheets produced with rice straw (1.94-2.40 %) and eucalyptus (3.56%) (Gominho *et al.*, 2001; Mutjé *et al.*, 2005; Jiménez *et al.*, 2007; Rodríguez *et al.*, 2008). Oluwadere and Ashimiyu (2007) found that cell wall thickness and fiber length were the main factors which influenced the strength properties like stretch % in paper sheets obtained from *Leu-*

caena leucocephala pulp. In agreement with this finding, the lower stretch % of sheets produced with *OFI* waste pulp compared to that of sheets produced with eucalyptus pulp can be explained as follow. Although the fiber length for *OFI* (1.1 mm) and eucalyptus (0.93 mm) is similar, the wall thickness is thicker for the eucalyptus fibers (6.1 µm) than for *OFI* fibers (4.3 µm) (Gominho *et al.*, 2014). Therefore, the lower stretch % obtained for the *OFI* waste sheets shows its poor flexibility compared to the high flexibility reported for eucalyptus.

Folding endurance is a property that can be used as an indicator of paper durability. This property measures the capacity of paper of keeping its folding line without breaking during repeating folding. Additionally, it reflects the degree to which the paper keeps its physical properties in relation to the frequency of use (Ponce-Jiménez *et al.*, 2002). *OFI* waste sheets presented a holding endurance (166 times) in the range for non-wood pulps. Folding endurance keep a positive correlation with fiber length (Ona *et al.*, 2001). This correlation can be observed in the paper derived from *OFI* waste fibers with similar length, such is the case of switchgrass (folding endurance = 210; 0.78 mm fiber length), alfalfa (folding endurance = 180; 0.78 mm fiber length), *Ipomea carnea* (folding endurance = 128; 0.62 mm fiber length) and *Cannabis sativa* (folding endurance = 223; 1.11 mm fiber length) (Dutt *et al.*, 2004; Ai and Tschirner, 2010).

Finally, regarding the porosity test, *OFI* waste paper sheets were not permeable to air because its pores were closed. This value is opposite to the reported for *OFI* cladodes (68.6 %) and *OFI* trunks (71.2%) paper sheets (Mannai *et al.*, 2016; Mannai *et al.*, 2018). Air resistance is a property governed by internal structure (type and length of fibers, degree of hydration, orientation and compaction of the fibers, as well as the type and amount of fillers and sizing) (T-460-om-16 2016). For example, paper sheet of kenaf (core fiber) had similar behavior than *OFI* waste paper. Kenaf paper had low air permeability, this was attributed to the fact that the fibers were small. The small size of the fibers facilitated the filling of voids in the paper sheets (Villar *et al.*, 2009). Santos *et al*. (2008) reported that pulp fibers with thinner fiber walls (more collapsible), generate paper structures with lower air permeability. In general, the pulp properties of *OFI* waste fibers were comparable to those of wood and non-woods pulp paper like pine, rapeseed straw, sugarcane bagasse and rice straw.

The main components of paper are recycled pulp (40-43%), chemical pulp (35-39%), mechanical pulp (8-11%), mineral fillers (8-11%) and chemical products (2-3%). The pulps come from different sources and origins. The mix of these components to form the structure of the paper allows high degrees of freedom to design a paper that satisfy the end use specifications of the product (Turrado-Saucedo *et al.*, 2008). According with the results of this research, the stretch and tear indexes of virgin *OFI* pulp agree to those specified for paper kraft (1.5%), bond paper (7.0 mNm^2/g), and coating base paper (6.25 to 8.75) (Escoto-García, 2004; Zanuttini *et al.*, 2008). The *OFI* pulp has its own properties which can be modified by mixing it with pulp from different sources to satisfy paper type specifications.

CONCLUSIONS

The intrinsic properties, chemical characteristics, and fiber morphology of Mexican *OFI* waste reveals that this non-wood plant can be used as a raw material for paper making. Mechanical properties of paper sheets obtained by the soda process reflect the quality of *OFI* fibers and the adequate conditions of processing applied (time, temperature, and active alkali). It remains to be further clarified whether our findings could be applied to the processing of different types of paper.

The use of *OFI* waste as a raw material for papermaking shows an option to utilize this unused byproduct. Therefore, an approach with respect to the technological, economical, and commercial viability needs to be developed to enable the sustainable utilization of *OFI* waste for papermaking to achieve a complete valorization.

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