Chapter

Cellulosic Fibers from Lignocellulosic Biomass for Papermaking Applications

Faten Mannai, Hanedi Elhleli, Ramzi Khiari and Younes Moussaoui

Abstract

This chapter gives a brief overview of the cellulose extraction from Opuntia (Cactaceae) fibers. The suitability of this food waste for pulp and paper production was investigated by the determination of the chemical composition and testing two procedures of delignification: chemical and semichemical pulping processes. Chemical pulping procedure was carried out by using soda-anthraquinone (soda-AQ) mixture, and semichemical pulping process was performed by softening the raw material using soda-hydrogen peroxide (soda-HP) mixture; this operation was followed by mechanical grinding. The obtained fibrous suspensions were characterized by measuring their dimension parameters (fiber length, fiber width, and fine elements), polymerization degree, and their retention water capacity. The effect of pulping process on yield and fiber characteristics in each pulp was studied. The surface morphologies of the produced papers were studied using scanning electron microscope (SEM), and results show the good distribution and individuality of fibers. The structural and mechanical properties of the prepared paper were presented and discussed. Mechanical strength results show the good tenacity of papers made from soda-HP pulping process.

Keywords: *Opuntia ficus-indica*, food waste, lignocellulosic fibers, deliberate fibers, fibrous suspension, pulping

1. Introduction

Opuntia ficus-indica is a xerophyte plant belonging to the Cactaceae family, well adapted to drought conditions thanks to its succulent nature that allows it to store extraordinary quantities of water [1]. *Opuntia ficus-indica* was used in traditional medicine for therapeutic, cosmetic, anticarcinogenic, anti-inflammatory, antioxidant, antiviral, and antidiabetic goods [2–4]. Thereby, *Opuntia ficus-indica* waste has received significant attention from numerous researches and was investigated because of its important chemical composition which has a high nutritional value, mainly due to their mineral, protein, dietary fiber, and phytochemical contents [4–9]. By-products of *Opuntia ficus-indica* were used by Bensadon et al. [4] as a source of good-quality antioxidant dietary fiber. Furthermore, *Opuntia ficus-indica* cladodes have interesting medical antioxidant activity [10, 11]. Likewise, methanol extract of *Opuntia ficus-indica* flowers has an anti-inflammatory effect on carrageenan-induced paw edema test [3]. The fruit syrup of *Opuntia ficus-indica* has a powerful antioxidant effect and exhibited effective antimicrobial activity against *Staphylococcus aureus* and *Staphylococcus epidermidis* [12]. In addition, Cactaceae waste was used also for nonfood applications by testing its ability to decontaminate wastewater through both the adsorption and coagulation-flocculation processes [13, 14], and manufacturing cactus fiber/polyester [15] and cactus fiber/polylactic acid [16] biocomposite materials (which contained cellulosic fibers obtained from cladodes) via rotational molding.

Otherwise, cellulosic fibers from *Opuntia ficus-indica* can be used as a raw material for papermaking. The pulp and paper industry is one of the largest and diversified industrial sectors in the world [17]. More than 400 million tons of paper are produced every year by different methods using many types of raw materials [17]. Traditionally, wood and forest resources were the basic sources of cellulosic fibers used by paper industries, but in recent years and in developing countries, about 60% of cellulose fibers originate from non-wood raw materials such as bagasse (sugarcane fibers), cereal straw, bamboo, reeds, esparto grass, jute, flax, and sisal [18]. For this reason, the selection of suitable non-wood fibers is critical for the yield of fibrous fraction, ease of processing, quality, and cost of the final fiber-based product [19].

The paper manufacturing process has several stages: raw material preparation, pulp manufacturing, pulp washing, chemical recovery, bleaching, stock preparation, and papermaking [17]. To realize the second stage, various pulping procedures have been utilized for the production of cellulosic fibers from non-wood raw material. These pulping methods might be classified mainly as chemical pulping, semichemical pulping, and mechanical pulping processes. In fact, chemical pulps are characterized by the highest production rate and represent almost pure celluloses; they are produced by combining heat and chemical treatment (kraft pulp) of wood chips with a mixture of sodium hydroxide and sodium sulfide [20]. The soda-anthraquinone (soda-AQ) process is similar to the kraft pulping procedure, which utilizes soda and anthraquinone catalysts. Soda-AQ can reduce the processing time and increases the pulp yield by protecting the carbohydrate compounds. Furthermore, semichemical pulping uses a combination of chemical and mechanical (i.e., grinding) processing to extract pulp fibers [21]. The raw material firstly is partially softened with chemicals, and mechanical methods complete the pulping process. One of the mild oxidant agents for chemical delignification is hydrogen peroxide. Hydrogen peroxide is characterized by its highest efficiency in bleaching and delignification when the reaction is conducted in alkaline medium with a stabilizing agent (diethylenetriamine pentaacetic acid (DTPA), ethylenediaminetetraacetic acid (EDTA)), etc. [22, 23]. Hydrogen peroxide readily decomposes to generate more active radicals which play a prominent role in dissolving lignin, hence releasing the fibers for papermaking [24]. The temperature and time of pulping in chemical and semichemical pulps depend on the type, composition, and source of lignocellulosic fibers. Indeed, in terms of yield, chemical pulping yields are between 45 and 55% but offer higher strength properties, and the fibers are more easily breached; and semichemical pulps, which apply to the category of chemical pulps, are obtained mainly from hardwoods with yields of between 65 and 85% (average ca. 75%) [21]. Moreover, pulp bleaching is often performed to produce special sorts of paper (such print and writing ones), while unbleached pulp can be used in various packing applications (including paperboards, food packaging, and large containment bags) [25]. In order to search for new sources of natural non-woody cellulosic fibers, *Opuntia ficus-indica* has been considered in this study as food waste source for pulp and papermaking production. It is a cactus from tropical, subtropical, arid, and semiarid regions, which exists in the form of a shrub or a tree with a height of up to 5 m and produces a sturdy trunk as it ages [26]. For paper manufacturing, the *Opuntia ficus-indica* fibers are firstly pulped by applying two different processes:

i. Chemical procedure using soda-anthraquinone mixture (soda-AQ).

ii. Semichemical procedure using a soft operation of delignification in soda-hydrogen peroxide (soda-HP) mixture followed by mechanical operation of fiber deliberation.

2. Experimental

2.1 Raw material preparation and chemical analyses

Manufacturing of pulp starts with raw material preparation [27]. The *Opuntia ficus-indica* stems (trunk) was harvested from southwest of Tunisia, debarked, cleaned, cut into chips (2–3 × 1–2 × 1.5–2 cm^3), dried at room temperature, and temporarily stored for further processing [28, 29]. The dried raw material was ground in accordance with the T 264 cm-07 standards, and its 40-mesh fractions were selected for chemical composition analyses. The ash, Klason lignin, holocellulose, and α-cellulose fractions in the raw materials were, respectively, quantified using the following TAPPI T211 om-07, T 222 om-06, procedure described by Wise et al. [30], and T 203 cm-99.

2.2 Pulping processing and testing

For paper manufacturing, two main step processes are followed in which the *Opuntia ficus-indica* chips are firstly converted into fibrous mass (pulp) and then the pulp is converted into paper. During pulping of this raw material, two procedures are used:

i. Semichemical pulping using soda-hydrogen peroxide (soda-HP).

ii. Chemical pulping using soda-anthraquinone (soda-AQ).

Multistep of pulping processes was followed to produce pulps from *Opuntia ficus-indica* chips as shown in **Figure 1**.

The delignification of *Opuntia ficus-indica* chips is carried out by adopting a chemical soda-HP process followed by mechanical deliberation operation of fibers [28]. The soda-HP pulping reaction is done under reflux, and the conditions are shown in **Figure 1** and listed in **Table 1**. After pulping, the obtained bleached soda-HP pulp was separated from white liquor by filtration and extensively washed several times with distilled water until neutrality. After that, a mechanical grinding operation is performed to more individualize and deliberate the fibrous suspensions. The obtained soda-HP pulp is purified by the classification of fibers. This operation is used to remove uncooked materials and other large-sized impurities by applying the standard T275 sp-12 method. The obtained soda-HP pulp was stored for further use.

Conventional soda-AQ pulping process was carried out in a rotating batch digesters with electrical heating equipped with thermocouples to monitor possible changes in temperature, and the processing time was automatically selected by the system [28, 29, 31, 32]. The soda-AQ pulping was performed by exploring different

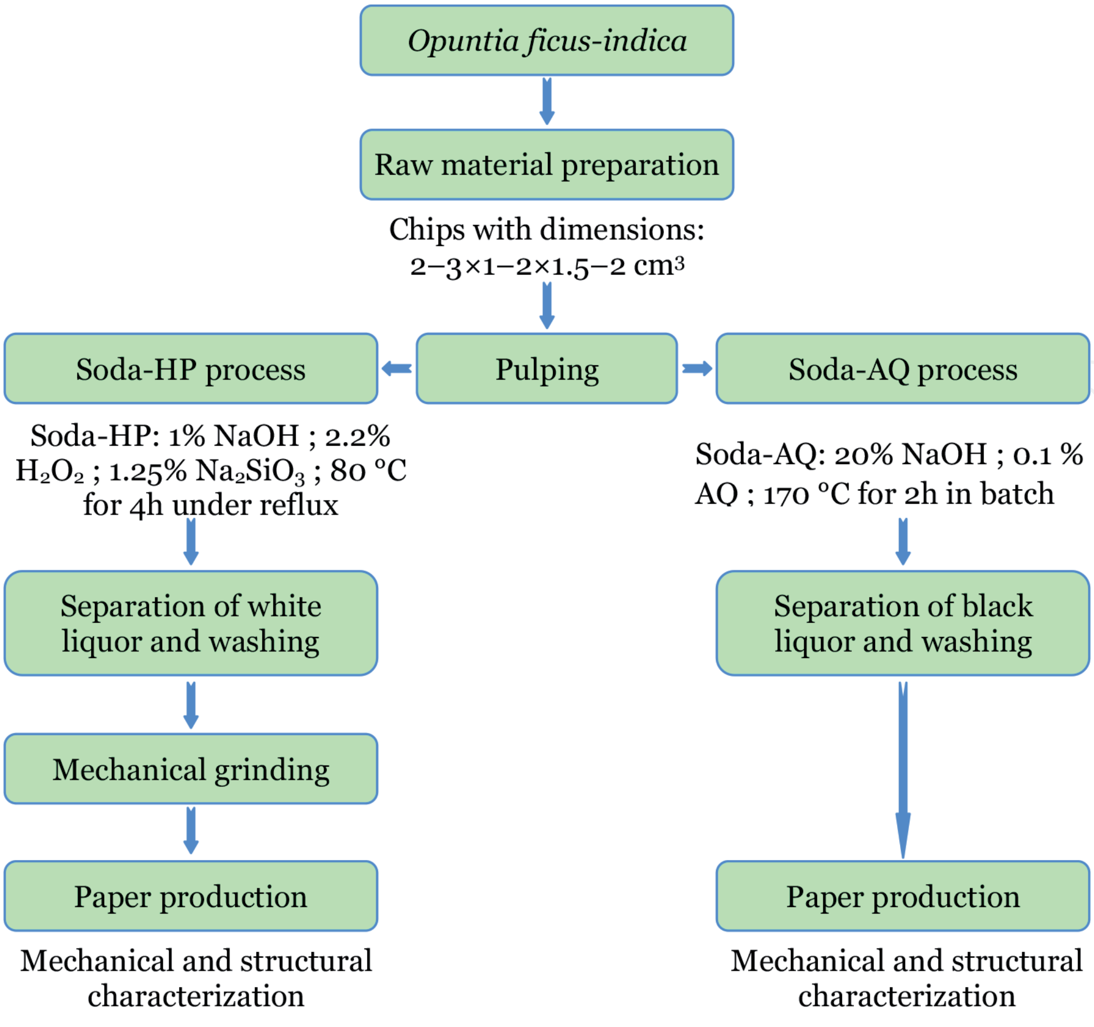

Figure 1.
The flowchart of Opuntia ficus-indica pulping processes.

Parameters	Soda-HP	Soda-AQ
NaOH (%)	1	20
H_2O_2 (%)	2.2	—
AQ concentration (o.d) (%)	—	0.1
Sodium silicate (o.d) (%)	1.25	—
pH	11	12
Liquor-to-raw material ratio	1:10	1:10
Temperature (°C)	80	170
Time (h)	4	2
Temperature ramping rate (°C/min)	—	2.4

Table 1.
Delignification conditions utilized for Opuntia ficus-indica fibers.

reaction conditions and parameters described in detail in **Table 1**. After processing, the obtained unbleached soda-AQ pulp was first separated from the black liquor, then carefully washed several times with distilled water, and stored for further use. Soda-AQ pulp will be exploited and characterized without bleaching operation.

The effect of pulping procedure on the yield and the properties of the deliberated fibers were studied using various ad hoc methods. Pulp yield (obtained from both processes) was determined as dry obtained on the basis of oven dried raw material. To examine the main morphological parameters (fiber dimensions) of the deliberated fibers (lengths, widths, and fractions of fine elements with lengths below 200 μm), 0.3 mg of pulp was suspended in 8 L distilled water and passed through the MorFi analyzer (LB01, developed by Techpap-France and the Paper Technical Centre). This technique is based on image analysis (using CCD camera), while more than 3000 fibers were observed in 2 min by the circulation of the fiber suspensions in a flat and transparent channel. The degree of polymerization (DP_v) was determined according to the procedure described by Sihtola et al. [33]. The intrinsic viscosity of the deliberated fibers (mPas) was measured in cupriethylenediamine (CED) according to the TAPPI T 230 om-99 standard. The water retention value (WRV) and degree of fiber swelling were estimated using a previously developed procedure [34]. The WRV was experimentally determined by the water retention measured after centrifugation at 3000 times for 15 min. The pulp drainability or Schopper-Riegler degree (°SR: ISO 5267-1) was evaluated by measuring the drainage capacity of a deliberated fibers in experimental conditions. Total ionic charges of the produced pulps (which correlated with the amount of the ionized chemical functions in the contained in the fibers) were determined by "Gran's method" [35].

2.3 Papermaking and testing

The deliberated fibers for each pulp were firstly disintegrated using a standard disintegrator T 205 sp-06 (shot 3000 rpm at room temperature) and then diluted to 2 g/L for paper preparation. Ten laboratory hand sheets with diameters of 20 cm via the standard ISO 5269-2 method were processed using Franc Rapid-Köthen sheet former apparatus. The produced hand sheets of papers were conditioned for 48 h at a temperature of 23°C and relative humidity of 50% before testing (ISO 187 standard). The structural and mechanical properties of paper samples were determined according to common standards: basis weights (ISO 536), thickness (ISO 534), air permeability (TAPPI: T452), tensile strength (NF Q 03-002), bursting strength (NF Q 03-053), and tear resistance (NF Q 03-011). Afterward, the bulk value and porosity of the produced paper sheets were calculated. The degree of bleaching (ISO 2470) and yellowness degree were detected by ELREPHO 2000 (yellowness degree measured by D 65/10 C and a 457 nm wavelength). The morphological analysis of each *Opuntia ficus-indica* paper was performed using the scanning electron microscopy (SEM, model Philips XL 30, USA).

3. Chemical properties of raw material

Table 1 shows the chemical composition results of *Opuntia ficus-indica* trunk, cladode [36], and other sources of fibers for comparison purposes. A very small fraction of minerals (5.5 wt%) was observed in the trunk [28] compared to the total minerals amount in the cladode (19.6 wt%). The lower fraction of minerals presents a major advantage, and the utilized raw material was silica free, which was extremely important for papermaking [29]. The total holocellulose contents (64 wt%) were higher than those of wood [20] and some annual plants [32, 37]. The α-cellulose rate was higher in the trunk (53.6 wt%) than those of cladode and other plants such as wood and annual plants [37]. The lower lignin amounts (**Table 2**) observed for *Opuntia ficus-indica* trunk and cladode (do not exceed 5 wt%) indicate that this plant was not a woody plant. Non-wood fibers are handled in ways specific to their composition [21]. For this reason, the processes used for *Opuntia*

Fiber plant	Ash	K.L	Holo	α-Cell
Opuntia ficus-indica trunk [28]	5.5	4.8	64.5	53.6
Opuntia ficus-indica cladode [36]	19.6	3.6	—	21.6
Soft-wood [4]	—	25–31	65–74	40–45
Annual plants [32, 37]	2–6.2	17–26	52–70	36–46

Table 2.
Chemical composition (ash (wt), Klason lignin (KL (wt%)), holocellulose (Holo (wt%)) and α-cellulose (α-cell (wt%))) of Opuntia ficus-indica trunk, and other values obtained for cladode (w/w%).

ficus-indica delignification was adapted in soft conditions to minimize degradation of the fibers and thus maximize pulp yield.

4. Effect of pulping process on yield and fiber characteristics

The produced pulps from soda-HP and soda-AQ processes are shown in **Figure 2**. The obtained pulps from both procedures show a high difference in terms of color. The soda-HP delignification process gives a bleached pulps and soda-AQ pulping process (without bleaching operation) conducted beige-brown (or kraft) color.

The pulp yield obtained for soda-HP pulping process of *Opuntia ficus-indica* fibers (about 80.8%) is 49% higher than that of soda-AQ pulping process (41.1%) (**Table 2**). The remarkable differences in the amounts of yields of the obtained pulps may be due to the differences in experimental conditions as well as the alkaline medium (or alkaline charge). However, the yield of the *Opuntia ficus-indica* pulp obtained from both soda-HP and soda-AQ processes is much higher than the magnitudes reported for most annual plants and agricultural crops (30–35%) [32, 37]. The high pulp yield is expected because of the higher cellulose content in raw material (**Table 2**). Due to the low content of lignin in the raw material, the kappa number of the produced *Opuntia ficus-indica pulp* was not calculated. The basic parameters that affected the paper properties are fiber's dimensions that include fiber length and fiber width [38]. The effect of soda-HP and soda-AQ pulping processes on morphological dimensions and characteristics was studied, and the obtained results are listed in **Table 3**. The deliberate fibers obtained from soda-HP pulping process present relatively high average fiber length of 764 μm than 737 μm of fibers made of soda-AQ pulping process. However, fiber length of *Opuntia ficus-indica* issued from both processes is in the range of hardwood fibers (0.7–1.5 mm) [39]. It was considered as short fiber species. The processing with soda-HP procedure gives thick individualized fibers with width of 38 μm. This value is in the range of hardwoods fibers (20.0–40.0) [40]. The fibers derived from soda-AQ pulping process are the thicker (45.6 μm) than fibers obtained from soda-HP pulping process and hardwood fibers. The processing with soda-AQ mixture and in high temperature (170°C) resulted in high fine element rate (29.3%).

The viscosity of the obtained soluble pulps represented by the DPv for soda-AQ pulp is around 600 (**Table 3**); this value was higher than those obtained from soda-HP pulp which represents about 500. The DPv values obtained for *Opuntia ficus-indica* pulps are lowest compared to those of woody fibers (1300–1500) and annual plants (900–1200) [32, 38]. The WRV obtained for soda-HP pulp is the same as that obtained for soda-AQ pulp. This may be explained by the presence of charged moieties (total charge of 21–22 μeq g^{-1}) associated with the fibers. In this context,

Figure 2.
Digital image of the produced pulps from Opuntia ficus-indica stems.

Pulps	Yield (%)	Fiber length (μm)	Fiber width (μm)	Fraction of fine elements (%)	DPv	WRV (%)	Total charge (μeq g^{-1})	°SR
Soda-HP [28]	80.8	764	38	16.3	500	67	21	11
Soda-AQ [29]	41.4	737	45.6	29.3	600	63.7	22	17

Table 3.
Effect of pulping process of Opuntia ficus-indica stems on yield, fiber dimensions, and other characteristics (degree of polymerization (DPv), water retention value (%), total charge, and Schopper degree (°SR)).

it was found that the lignin was totally oxidized according to the appearance of the hydration phenomenon (due to the presence of extra fiber water) [28, 41, 42]. The drainability of the *Opuntia ficus-indica* soda-HP pulp (°SR) is important (11) than those of soda-AQ pulp (17). The °SR observed for soda-HP pulp confirms the good quality of fibers. Thus, based on the properties established for the different pulps, we deduced that the most suitable one is processed by soda-HP procedure.

5. Evolution of pulping processes on paper properties

The morphological SEM analysis of the manufactured handmade paper sheets was performed in order to evaluate the effect of pulping processes on paper morphology (**Figure 3**). The fibers depicted on both sheet surfaces are long, swollen, well separated, homogeneous, and strongly linked together (fiber network). Fine elements were evenly distributed across the paper surface and mainly observed for soda-AQ paper. This is in agreement with MorFi's results.

Structural and mechanical parameters of handmade paper sheets produced from *Opuntia ficus-indica* soda-HP and soda-AQ pulps were estimated and listed in **Tables 4** and **5**, respectively. The obtained basic weight for sheets made from soda-AQ pulp (65.2 g m^{-2}) was 41% higher than that of the soda-HP papers. The corresponding bulk values obtained for the both handmade sheet papers are the same (2–2.2 cm^3 g^{-1}); these values were slightly lower than that of the Alfa paper (2.36 cm^3 g^{-1}) [43]. The low value of bulk is mainly due to the lower thickness of the

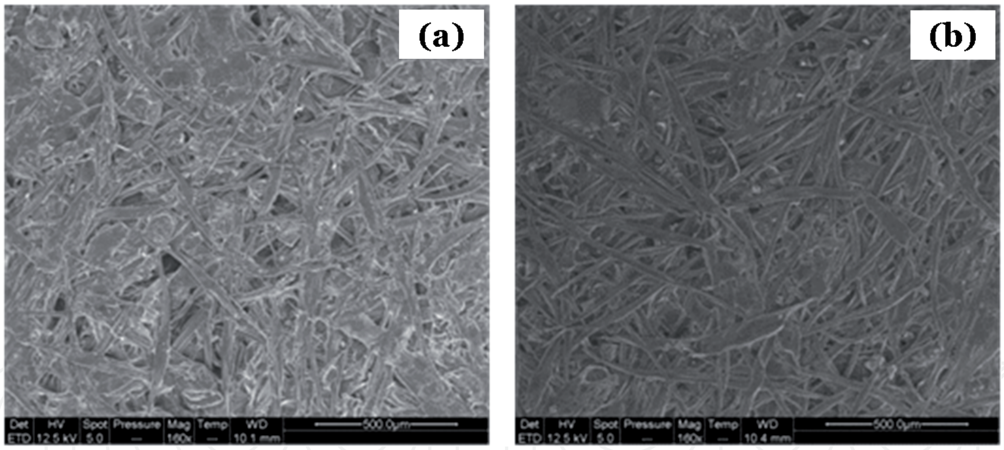

Figure 3.
SEM images [magnification ×500 µm] of surface morphologies of papers made from soda-HP pulp (a) and paper made from soda-AQ pulp (b) (the encircled zones show the presence of fine elements).

Paper samples	Basis weight ($g\ m^{-2}$)	Thickness (µm)	Bulk ($cm^3\ g^{-1}$)	Air permeability ($cm^3/s\ Pa\ m^2$)	Porosity (%)	D.B (%)	D.Y (%)
Soda-HP [28]	38.4	149	2.2	231.1	71.2	67.4	6.8
Soda-AQ [29]	65.2	135	2	229.2	68.6	—	—

Table 4.
Structural properties, degree of bleaching (D.B.%) and degree of yellow (D.Y.%), of papers produced from the Opuntia ficus-indica pulps.

Paper samples	Burst index ($kPa\ m^2\ g^{-1}$)	Tear index ($mNm^2\ g^{-1}$)	Breaking length (km)
Soda-HP [28]	0.67	19.2	1.9
Soda-AQ [29]	5.8	12	1.5

Table 5.
Mechanical strength properties of papers produced from the Opuntia ficus-indica pulps.

Opuntia ficus-indica papers. However, the porosity and air permeability measured for soda-HP papers were significantly higher than those obtained for soda-AQ papers. In addition, the important degree of bleaching was higher than ISO 11475 and 11476 for newsprint (65%) as well as for the yellow degree. Therefore, the soda-HP pulping process presents the advantage that relatively white papermaking sheets could be prepared without bleaching.

The burst index obtained for soda-AQ papers was 88% higher than soda-HP papers. The variation of burst index was mainly affected by fiber width and fine elements present in soda-AQ pulp [44]; it was dependent on pulping methods. The tear index noted for soda-HP papers was higher than those obtained for soda-AQ papers. This is due to the high fiber length which usually had a significant effect on the tearing strength of papers. The breaking length obtained for soda-HP papers was quite higher (1.9 Km) than those obtained for soda-AQ papers. The use of soda-HP pulping process affects the paper properties by increasing the fiber flexibility and strength [21]. Thus, the morphological, structural, and mechanical characteristics of the *Opuntia ficus-indica* fibers suggest their possible applications for producing paper from non-woody plants.

6. Conclusions

The study revealed that *Opuntia ficus-indica* can be potentially utilized as a suitable food waste source of non-wood fibers for papermaking application. Its chemical composition showed that the fibers were rich with biopolymers (cellulose 53.2 wt%) interconnected with natural resins confirmed by the low presence of lignin (4.8 wt%). In order to select the best conditions of delignification and reducing the lignin content, various pulping procedures were tested for this raw material. The alkaline peroxide pretreatment (soda-HP pulping process) of the raw material produced bleached pulp and high-quality pulp with good yield (80.8%). Fibrous mass produced using soda-AQ pulping process gives a brown pulp with a yield about 41.1%. The pulping method described in this chapter affects the pulp properties and paper characteristics. However, the papers obtained from soda-HP pulping process (eco-friendly process) have a white color, good structural properties, and high tear strength compared to those obtained from soda-AQ pulp. Thus, the results obtained in this study indicate that pulps for papermaking applications can be fabricated from agricultural waste, which represent a good option for the countries without forests.

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Conflict of interest

The authors declare no conflict of interest.

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