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Physico-Chemical Characterization of Some Saudi Lignocellulosic Natural Resources and their Suitability for Fiber Production

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Abstract. The valorization for fiber production of eight Saudi lignocellulosics: *Phoenix dactylifera* (surface fibers, leaflet and rachis) and wood from each of *Conocorpus erectus*, *Leucaena leucocephala, Simmondsia chinensis*, *Azadirachta indica* and *Moringa peregrina* were investigated. Fiber length, specific gravity and chemical composition: total extractives, lignin, holocellulose and ash contents of the eight lignocellulosic materials were determined. All the characteristics examined varied significantly due to the natural resource effect. The three lignocellulosic materials of date palm had higher contents of total extractives, lignin and ash; and lower holocellulose content compared to those from timber trees. Although the date palm leaflet possessed the longest fibers (1.31mm), followed by rachis (1.19mm). Their fiber manufacturing needs more chemicals and time and some problems arise upon chemical recovery process due to their chemical composition. The timber trees properties are more suitable for fiber products than the date palm resources although their shorter fibers. *Leucaena leucocephala* was the best resource due to its high content of holocellulose (70.82%), its reasonable fiber length (1.13mm) and to its specific gravity (0.597) approaching those of other hardwoods and its low contents of total extractives (9.74%), lignin (18.86%) and ash (1.22%) compared to the other resources examined.

Keywords: Fiber production, Lignin, Holocellulose, Fiber length, Pulping.

Introduction

World paper consumption was expected to rise above 400 million tons by this year, 2010 (Hurter and Riccio, 1998). High-yield fiber plants offer enormous potential to provide a productive new resource for pulp and fiber manufacturing sector (Mansfield and Weineisen, 2007). In view of shortage of conventional raw materials for fiber products along with increasing demand for them, valorization of non-woody plants and agriculture residues has been attracting renewable interests. Furthermore, it was reported by Prasad, *et al*. (2009) that short rotation industrial agroforestry plantations with the fast growing species are potential sources to fill the gap between production and demand, and to make the nation self reliant in pulpwood supply.

The date palm trunks are used as beams and rafters, leaves are used as a raw material for many of the rural industries. Furthermore, rachis and leaves can be viewed as sources of reinforcing fibers for polymeric matrices in composite materials (Khiari, 2010). Khristova, *et al*. (2005) found that fiber of date palm rachis is close to that of hardwoods and the open structure of date palm leaves which is easy for chemical to penetrate makes it possible to obtain strong pulp with soda process. *Leucaena leucocephala* is promising as a faster growing species for biomass and paper production, and it showed suitable physical characteristics of paper sheet (Lopez, *et al*., 2008). *Conocorpus erectus* and *Azadirachta indica* showed good adaptability to Saudi Arabia climate and are expected to share for solution of fiber shortage problem of the Kingdom. Jojoba, a relatively new crop that is adapted to hot, dry climate (Ash, *et al*., 2005), as well as, *Moringa perigrina* can be used to fill the gap between fiber production and demand. The length of fiber greatly affects the strength of pulp and the paper made from it (Kaila and Aittamaa, 2006). Accordingly, paper made from species with long fibers is expected to show higher quality than those with shorter fibers.

This study was conducted to evaluate the physico-chemical characterization of three date palm fibrous by-products (surface fibers, leaflet and rachis), as well as, woods from each of *Conocorpus erectus*, *Leucaena leucocephala, Simmondsia chinensis, Azadirachta indica* and *Moringa peregrina.*

Materials and Methods

Raw Material

Six Saudi natural resources were used as sources of the eight lignocellulosic materials in this investigation for their suitability for fiber production. The study was performed during 2009 at the Agricultural Research Station, Hada Al-Sham, King Abdulaziz University. The six species studied were date palm (*Phoenix dactylifera* L.), buttonwood (*Conocorpus erectus L.*), leucaena (*Leucaena leucocephala* (Lam) de wit), jojoba (*Simmondsia chinensis* (Link) C.K. Schneid), neem (*Azadirachta indica* A. Juss) and moringa (*Moringa peregrina* Forssk. ex. Fiori). The ages of the selected natural resources ranged from 5-20 years. Furthermore, their diameters outside bark varied between 5-40 cm.

Completely randomized design with three replications was used in this study according to El-Nakhlawy (2009). Eight lignocellulosic materials were used in this study namely, *Phoenix dactylifera* surface fibers, *Phoenix dactylifera* leaflet, *Phoenix dactylifera* rachis, *Conocorpus erectus* wood, *Leucaena leucocephala* wood, *Simmondsia chinensis* wood, *Azadirachta indica* wood and *Moringa peregrina* wood. Three healthy trees were chosen randomly from those grown at Hada Al-Sham.

Three different raw materials of date palm, namely surface fibers, leaflets and rachis were used for the study. For the surface fibers, samples representing three specified heights (base, medium and top) along each trunk were collected for the different determinations. For leaflets and rachis, samples representing four leaves at certain directions (North, South, East and West) and a constant height at the same girth were randomly taken from each palm for the different tests.

For the timber trees, one disc (20cm thick) at breast height was isolated from the stem, except for jojoba whereby the disc was cut at 30cm above the ground level due to its branching pattern.

Lignocellulosic Properties Determinations

The studied traits were: Fiber length (FL), specific gravity (SG), total extractives content (TEC), lignin content (LC), holocellulose content (HC) and ash content (AC).

For the measurement of fiber length (FL), assigned thin chips were exposed to maceration by Franklin method using glacial acetic acid and hydrogen peroxide as indicated by Megahed, *et al*. (1998) and Kherallah and Aly (1989). For the leaflet and rachis samples, extraction by acetone was applied before maceration in a Soxhlet apparatus to eliminate the organic chemicals that interfere with the maceration reagents used. A drop of macerated sample was taken on a slide, and the FL was measured under a projecting fiber suspension microscope after staining with 1% aqueous safranine. Twenty five fibers were measured from each of the three slides prepared to represent each tree (nine slides for each of the eight lignocellulosic materials). Accordingly, 1800 fibers were measured among the investigation.

For specific gravity (SG) determinations, five defect-free samples (2.5cm radially and tangentially and 2cm longitudinally each) were used from each tree (15 samples of each lignocellulosic material). Accordingly, 120 samples were specified for the eight resources used. The green samples assigned for this test were accurately re-saturated by water under vacuum (Hindi, 2001) and the saturated volume was measured by Pycnometric displacement of water (American Society for Testing and Materials, 1989a). The SG was calculated based on oven-dry weight and saturated volume.

The remainder mass of the collected samples were ground, sieved and specified for the determinations of total extractives content (TEC), lignin content (LC), holocellulose content (HC) and ash content (AC) of the eight lignocellulosic materials. For each determination, five samples were taken randomly from each tree (15 samples of each lignocellulosic material. Accordingly, 120 samples were specified for the eight resources used for each determination.

Total extractives content (TEC) was determined according to the *American Society for Testing and Materials* (1989b). Then, each of the TEC samples was divided into two equal portions. One of them was assigned for lignin content determination and the other for holocellulose test. Lignin was determined according to the *American Society for Testing and Materials* (1989c) using H_2SO_4 (72%). Holocellulose was determined according to the procedure stated by Wise, *et al*. (1946). To measure ash content, air-dried samples were ignited at 600°C until all carbon was eliminated (American Society for Testing and Materials, 1989d).

Statistical analysis of the recorded data was done according to El-Nakhlawy (2009) using the analysis of variance procedure and least significant difference test (LSD) at P≤0.05.

Results and Discussion

Characterization of the eight lignocellulosic materials is listed in Table 1. The statistical analyses indicated that the eight ligno-cellulosic materials tested were significantly different in their properties.

Fiber Length (FL)

Comparisons of the means using LSD test showed that Fl was significantly affected by the natural resources studied. Means presented in Table 1 revealed that *Phoenix dactylifera* leaflet had the longest fibers (1.31mm) followed by *Phoenix dactylifera* rachi*s* (1.19mm), *Leucaena leucocephala* (1.13mm) and *Azadirachta indica* (1.04mm). On the other hand, *Simmondsia chinens* was the shortest(0.5mm). Since, the length of fiber greatly affects the strength of the pulp and the paper made from it (Kaila and Aittamaa, 2006), paper made from *Leucaena leucocephala* and *Azadirachta indica* is expected to show higher quality than the others with shorter fibers.

The FL results are in agreement with previously published researches using other wood species (Kherallah and Aly, 1989, Megahed, *et al*., 1998, and Diaz, *et al*. 2007).

Specific Gravity (SG)

It was found that SG varied significantly due to natural resources effect. The SG means ranged from 0.369 for *Phoenix dactylifera* surface fibers to 0.645 for *Conocorpus erectus* wood .The higher SG values were found to be for the timber trees examined except for *Moringa peregrina* (0.43) that occupied a relatively moderate level. However, the lignocellulosics from date palm had lower SG value (Table 1). Higher SG values indicate that more cell wall materials can be used industrially. On the other hand, woody materials with low SG permit to pulping reagents to penetrate more easily into their lignocellulosic tissues through a short time. However, the quality of a final fibrous product and the cost of production control the required SG level of a parent raw material.

Total Extractives Content (TEC)

The lignocellulosic natural resources studied varied significantly from one resource to another in relation to the TEC parameter. The TEC value of date palm leaflet was the highest (37.06%), followed by rachis (26.81%), surface fibers (16.44%) and *Simmondsia chinensis* wood (15.01%). On the other hand, the timber trees possessed lower TEC contents with the lowest value for *Moringa peregrina* wood (Table 1).

The highest TEC content for date palm leaflet may be attributed to the open anatomical structure easily accessible for the chemicals (Khristova, *et al*., 2005). The presence of high extractives into the lignocellulosic tissues is unpreferred due to their interference with the maceration reagents used to separate the fibers. Therefore, the date palm leaflet, rachis and surface fibers, as well as, *Simmondsia chinensis* must be organic solvent-extracted before maceration. Certainly, this step will add additional cost to this industry. The remainder materials examined were suitable for fiber production specially *Moringa peregrina*, *Leucaena leucocephala* and *Azadirachta indica*. In addition, there is another defect of presence high content of extractives as reported by Lopez, *et al*. (2008) in which pulp yield is negatively correlated with the extractives content (ethanol-benzene and water soluble). Therefore a greater pulp yield could be supposed for materials with low contents of extractives.

Lignin Content

Statistical analysis showed a significant difference between the eight natural resources tested for the lignin content parameter. It can be seen from Table 1 that leaflet and surface fibers from the date palm had higher contents of lignin (36.44% and 31.3%, respectively), approaching to that for softwoods and quite high compared to the other materials studied, as well as, to typical amounts encountered in annual plants, non-wood and hardwood sources. Contrarily, the date palm rachis contained the lowest lignin value (14.28%) that is below that for hardwoods. Low lignin content of a lignocellulosic material reduces pulping time and chemical charge compared to those of other non-wood raw materials (Lopez, *et al*, 2008 and Diaz, *et al.*, 2007). Furthermore, higher contents of lignin are predicted to consume more chemicals upon the pulp industry (Khristova, *et al.*, 2005). The results are in agreement with those of Lopez, *et al*, (2008) and Megahed, *et al*. (1998).

Holocellulose

The HC results showed that the studied natural resources were found to be significantly different. *Leucaena leucocephala* was the superior for its HC value. This allows envisaging the valorization of such crop as cellulose derivatives and/or as lignocellulosic fibers for fiber-reinforced composite materials or papermaking applications (Khiari, 2010). On the other hand, date palm leaflet possessed the lowest HC value (Table 1).

Ash Content

The lignocellulosic resources examined were significantly different in their contents of ash. The three resources of date palm (surface fiber, leaflet and rachis) contained higher ash than the other resources (Table 1). High contents of ash will negatively impact the chemical recovery process and, therefore, could constitute a serious drawback (Khiari, 2010). However, the results were in agreement with those arising from other literature (Lopez, *et al.*, 2008 and 2009, Ververis, *et al.*, 2004, Amaducci, *et al.*, 2007 and Diaz, *et al.*, 2007).

Table 1. Mean values of fiber length (FL), specific gravity (SG), total extractives (TEC), lignin (LC), holocellulose (HC) and ash (AC) contents of some Saudi lignocellulosic natural resources.

Lignocellulosic material	FL mm	SG	TEC %	LC %	HC %	AC %
Phoenix dactylifera surface fibers	0.84 [e]	0.369 [d]	16.44 [c]	31.30 [b]	40.31[f]	11.82 [a]
Phoenix dactylifera leaflet	1.31 [a]	0.480 [b]	37.06 [a]	36.44 [a]	16.32 [g]	10.8 [b]
Phoenix dactylifera rachis	1.19 [b]	0.398 [c d]	26.81 [b]	14.28 [e]	48.34 [e]	12.27 [a]
Conocorpus erectus wood	0.93 [d]	0.645 [a]	12.93 [e]	28.83 [c]	57.68 [c]	0.91 [e]
Leucaena leucocephala wood	1.13 [b]	0.597 [a]	9.74 [f]	18.86 [d]	70.82 [a]	1.22 [de]
Simmondsia chinensis wood	0.50 [g]	0.638 [a]	15.08 [d]	28.18 [c]	53.11 [d]	2.31 [c]
Azadirachta indica wood	1.04 [c]	0.618 [a]	10.23 [f]	27.94 [c]	59.91 [b]	1.47 [d]
Moringa perigrina wood	0.68 [f]	0.430 [bc]	8.52 [g]	28.26 [c]	59.64 [b]	2.73 [c]

[*] Means within the same column followed by the same letter are not significantly different according to LSD at $P \leq 0.05$.

The timber trees properties are more suitable for fiber products than the date palm resources although their shorter fibers. *Leucaena leucocephala* is the best resource due to its high content of holocellulose, its reasonable fiber length and specific gravity approaching to those for hardwoods. Its contents of total extractives, lignin and ash are low compared to the other resources examined.

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التقييم الفيزيوكيميائي لبعض الموارد الطبيعية اللجنوسليلوزية السعودية وملاءمتها لصناعة الألياف

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المستخلص. تمت دراسة كفاءة ثمانية أنواع لجنوسليولوزية لإنتاج الألياف وهي: نخيل التمر *Phoenix dactylifera* (كل من الألياف السطحية والوريقات والعرق الوسطي للأوراق)، وأخشاب كل من البازروميا *Conocorpus erectus*، والليوسينا *Leucaena leucocephala*، والهوهوبا *Simmondsia chinensis*، والنيم *Azadirachta indica*، والمورنجا *Moringa peregrina*. تم قياس طول الألياف، والثقل النوعي، والتركيب الكيميائي، مثل كمية المستخلصات الكلية، واللجنين، والهلوسليلوز، والرماد لكل من المواد اللجنوسليلوزية الثمانية. ولقد وجد أن الصفات المدروسة اختلفت معنويًا باختلاف المواد المستخدمة في الدراسة. ولقد احتوت الثلاث مواد المأخوذة من نخيل التمر على كميات أكبر من المستخلصات الكلية، واللجنين، والرماد، وعلى كمية أقل من الهلوسليلوز. وبرغم أن وريقات النخيل ذات ألياف أطول (١,٣ مم)، يتبعها العرق الوسطي للورقة (١,١٩ مم)، إلا أن استخدامها في تصنيع الألياف يحتاج لكميات أكبر من الكيماويات خلال زمن أطول، مع ظهور بعض المشاكل خلال عملية استعادة الكيماويات، وذلك بسبب تركيبها الكيميائي. إن خواص الأشجار الخشبية أكثر ملائمة للمنتجات الليفية بالمقارنة بالمصادر الطبيعية

من نخيل التمر بالرغم من قصر أليافها. ولقد وجد أن الليوسينا هي أفضل المصادر بسبب ارتفاع محتواها من الهلوسليلوز (٧٠,٨٢٪)، واحتوائها على ألياف ذات طول معتدل (١,١٣ مم) مع ثقل نوعي (٠,٥٩٧) يقارب ما لصالدات الأخشاب الأخرى، وكذلك محتواها المنخفض من المستخلصات الكلية (٩,٧٤٪)، واللجنين (١٨,٨٦٪)، والرماد (١,٢٢٪)، وذلك بالمقارنة بباقي المصادر الأخرى المختبرة.