Chemical composition of lipophilic extractives from jute (*Corchorus capsularis*) fibers used for manufacturing of high-quality paper pulps

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ABSTRACT

The chemical composition of the lipophilic extractives from jute (*Corchorus capsularis*) fibers, which are used for high-quality paper pulp manufacturing, was thoroughly studied. The extractives content was low (0.4%), and its composition was studied by gas chromatography–mass spectrometry. For a better characterization of the different homologous series and compounds present in minor amounts, the extract was also fractionated by solid-phase extraction. The most predominant lipophilic compounds present in jute fibers were high molecular weight ester waxes (24% of total extract), followed by free fatty acids (17%), free fatty alcohols (17%) and α -hydroxyfatty acids (14%). Additionally, significant amounts of alkanes (6%), ω -hydroxyfatty acids (6%), sterols (6%), steroid and triterpenoid ketones (3%) and steryl glycosides (1%) were also identified, together with minor amounts of mono- and diglycerides.

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1. Introduction

Jute (*Corchorus capsularis*) is an herbaceous annual plant from the Tiliaceae family, mostly grown in Southeast Asian countries. Jute fibers are separated from the bast or outer region of the stem after retting of the whole plant and are mainly used for the manufacture of cordage, carpets, bagging and wrapping materials, with an annual production of 2.65 million tons. In addition, jute fibers are a good source of different grades of paper pulp (Akhtaruzzaman and Shafi, 1995; Jahan, 2001). With the increasing concern over forest destruction and the increasing demand of paper pulp, the importance of jute for this purpose may increase. There have been previous published studies describing the characteristics of jute fibers, including the content of their main organic fractions, particularly lignin and carbohydrates, and its behavior during pulping (Akhtaruzzaman and Shafi, 1995; Jahan, 2001; Jahan et al., 2007, 2008). However, in order to maximize the exploitation of this fiber for paper pulp production, a more complete understanding of its chemistry is required.

In the present study, we report the chemical composition of lipophilic compounds from jute fibers, which up to our knowledge,

has not been addressed before. It is well known that the content and composition of lipophilic compounds present in raw materials cause significant environmental and technical problems in the manufacturing of paper pulp (Back and Allen, 2000). During pulping, lipids are released from the fibers forming colloidal pitch, which can deposit in either pulp or machinery causing production troubles and important economic losses (Hillis and Sumimoto, 1989; Back and Allen, 2000). In the manufacture of alkaline pulps, a large part of the lipids originally present in the raw material is removed during the cooking and bleaching stages. However, some chemical species survive these processes and are found as pulp extractives (Gutiérrez et al., 2001a; Gutiérrez and del Río, 2003a; Bergelin and Holmbom, 2003; Freire et al., 2005, 2006), suspended in process waters (Gutiérrez et al., 2001b) or forming the so-called pitch deposits in circuits, equipments and final product (del Río et al., 1998, 2000; Silvestre et al., 1999; Bergelin et al., 2005; Gutiérrez and del Río, 2005). Pitch deposition is a serious problem in the pulp and paper industry since it is responsible for reduced production levels, higher equipment maintenance costs, higher operating costs and an increased incidence of defects in the final products (Back and Allen, 2000). Moreover, such extractives might contribute to the toxicity of paper pulp effluents and products (Ali and Sreekrishnan, 2001; Christianson-Heiska et al., 2008).

In this paper, the composition of the lipophilic compounds was carried out by gas chromatography (GC) and gas

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chromatography–mass spectrometry (GC–MS) using short- and medium-length high-temperature capillary columns, respectively, with thin films, which enables the elution and analysis of intact high molecular weight lipids such as waxes or sterol glycosides (Gutiérrez et al., 1998). Additionally, and for a more detailed characterization of the different homologous series and other minor compounds, the extract was fractionated by a simple solid-phase extraction (SPE) method using aminopropyl-phase cartridges, as described previously (Gutiérrez et al., 1998).

2. Materials and methods

2.1. Sample

Jute (*C. capsularis*) fibers were supplied by CELESA pulp mill (Tortosa, Spain). Jute fibers were air-dried. The dried samples were milled using a knife mill (Janke and Kunkel, Analysenmühle), and successively extracted with acetone in a Soxhlet apparatus for 8 h and hot water (3 h at 100 °C). The acetone extracts were evaporated to dryness, and resuspended in chloroform for chromatographic analysis of the lipophilic fraction. Two replicates were used for each sample.

2.2. Solid-phase extraction (SPE) fractionation

The lipid extracts were fractionated by a SPE procedure using aminopropyl-phase cartridges (500 mg) from Waters Division of Millipore (Mildford, MA, USA), as already described (Gutiérrez et al., 1998). Briefly, the dried chloroform extracts were taken up in a minimal volume (<0.5 mL) of hexane:chloroform (4:1) and loaded into the cartridge column previously conditioned with hexane (4 mL). The cartridge was loaded and eluted by gravity. The column was first eluted with 8 mL of hexane and subsequently with 10 mL of chloroform and finally with 10 mL of diethyl ether:acetic acid (98:2). Each isolated fraction was dried under nitrogen and analyzed by GC and GC–MS.

2.3. GC and GC–MS analyses

An HP 5890 gas chromatograph (Hewlett Packard, Hoofddorp, Netherlands) equipped with a split–splitless injector and a flame ionization detector (FID) was used for GC analyses. The injector and the detector temperatures were set at 300 °C and 350 °C respectively. Samples were injected in the splitless mode. Helium was used as the carrier gas. The capillary column used was a high-temperature, polyimide coated fused silica tubing DB5-HT (5 m × 0.25 mm I.D., 0.1 µm film thickness; J&W Scientific). The oven was temperature-programmed from 100 °C (1 min) to 350 °C (3 min) at 15 °C min^{−1}. Peaks were quantified by area, and a mixture of standards (octadecane, palmitic acid, sitosterol, cholesteryl oleate, and sitosteryl 3β-D-glucopyranoside) was used to elaborate calibration curves. The data from the two replicates were averaged.

The GC–MS analysis were performed on a Varian Star 3400 gas chromatograph (Varian, Walnut Creek, CA) coupled with an ion-trap detector (Varian Saturn) equipped with a high-temperature capillary column (DB-5HT, 15 m × 0.25 mm i.d., 0.1 µm film thickness; J&W Scientific). Helium was used as carrier gas at a rate of 2 mL/min. The oven was heated from 120 °C (1 min) to

Fig. 1. GC–MS chromatogram of the underivatized lipid extracts from jute (*C. capsularis*) fibers. F(n): *n*-fatty acid series; Ak(n): *n*-alkane series; Ah(n): *n*-fatty alcohol series; W(n): high molecular weight ester wax series; *n* denotes the total carbon atom number. Other compounds reflected are: (1) *trans*-coniferyl alcohol; (2) *trans*-sinapyl alcohol; (3) stigmasta-3,5,7-triene; (4) campesterol; (5) stigmastanol; (6) isomultiflorenone; (7) sitosterol; (8 and 9) unidentified triterpenoid compounds with epoxydammarane-type structure.

Fig. 2. GC–MS chromatograms of the different SPE fractions isolated from the extracts of jute fibers. (a) Fraction A, eluted with 8 mL of hexane; (b) fraction B, eluted with 10 mL of chloroform; and (c) fraction C, eluted with 10 mL diethyl ether:acetic acid (98:2). F(n): *n*-fatty acids series; Ak(n): *n*-alkane series; Ah(n): *n*-fatty alcohol series; W(n): high molecular weight ester wax series; *n* denotes the total carbon atom number. Other compounds are: (4) campesterol; (5) stigmastanol; (6) isomultiflorenone; (7) sitosterol; (8 and 9) unidentified triterpenoid compounds with epoxydammarane-type structure.

Table 1Composition of lipids (mg/kg) from jute (*C. capsularis*) fibers.

Compounds	Mass fragments	MW	Formula	Abundance
<i>n</i>-Alkanes				48.5
<i>n</i> -Heptacosane	57/71/85/380	380	C ₂₇ H ₅₆	1.4
<i>n</i> -Octacosane	57/71/85/394	394	C ₂₈ H ₅₈	1.0
<i>n</i> -Nonacosane	57/71/85/408	408	C ₂₉ H ₆₀	6.3
<i>n</i> -Triacontane	57/71/85/422	422	C ₃₀ H ₆₂	1.5
<i>n</i> -Hentriacontane	57/71/85/436	436	C ₃₁ H ₆₄	8.0
<i>n</i> -Dotriacontane	57/71/85/450	450	C ₃₂ H ₆₆	2.5
<i>n</i> -Tritriacontane	57/71/85/464	464	C ₃₃ H ₆₈	20.6
<i>n</i> -Tetraatriacontane	57/71/85/478	478	C ₃₄ H ₇₀	1.8
<i>n</i> -Pentatriacontane	57/71/85/492	492	C ₃₅ H ₇₂	5.4
Fatty acids				130.2
<i>n</i> -Tetradecanoic acid	60/73/129/228	228	C ₁₄ H ₂₈ O	2.4
<i>n</i> -Pentadecanoic acid	60/73/129/242	242	C ₁₅ H ₃₀ O	5.5
<i>n</i> -Hexadecanoic acid	60/73/129/256	256	C ₁₆ H ₃₂ O ₂	30.1
<i>n</i> -Heptadecanoic acid	60/73/129/270	270	C ₁₇ H ₃₄ O ₂	1.5
9,12-Octadecadienoic acid	67/81/280	280	C ₁₈ H ₃₂ O ₂	1.1
9-Octadecenoic acid	55/69/264	282	C ₁₈ H ₃₄ O ₂	18.9
<i>n</i> -Octadecanoic acid	60/73/129/284	284	C ₁₈ H ₃₆ O ₂	11.3
<i>n</i> -Nonadecanoic acid	60/73/129/298	298	C ₁₉ H ₃₈ O ₂	0.5
<i>n</i> -Eicosanoic acid	60/73/129/312	312	C ₂₀ H ₄₀ O ₂	6.6
<i>n</i> -Heneicosanoic acid	60/73/129/326	326	C ₂₁ H ₄₂ O ₂	2.6
<i>n</i> -Docosanoic acid	60/73/129/340	340	C ₂₂ H ₄₄ O ₂	9.8
<i>n</i> -Tricosanoic acid	60/73/129/354	354	C ₂₃ H ₄₆ O ₂	3.1
<i>n</i> -Tetracosanoic acid	60/73/129/368	368	C ₂₄ H ₄₈ O ₂	5.0
<i>n</i> -Pentacosanoic acid	60/73/129/382	382	C ₂₅ H ₅₀ O ₂	3.9
<i>n</i> -Hexacosanoic acid	60/73/129/396	396	C ₂₆ H ₅₂ O ₂	2.5
<i>n</i> -Heptacosanoic acid	60/73/129/410	410	C ₂₇ H ₅₄ O ₂	1.0
<i>n</i> -Octacosanoic acid	60/73/129/424	424	C ₂₈ H ₅₆ O ₂	2.3
<i>n</i> -Nonacosanoic acid	60/73/129/438	438	C ₂₉ H ₅₈ O ₂	1.1
<i>n</i> -Triacontanoic acid	60/73/129/452	452	C ₃₀ H ₆₀ O ₂	6.5
<i>n</i> -Hentriacontanoic acid	60/73/129/466	466	C ₃₁ H ₆₂ O ₂	1.1
<i>n</i> -Dotriacontanoic acid	60/73/129/480	480	C ₃₂ H ₆₄ O ₂	7.9
<i>n</i> -Tritriacontanoic acid	60/73/129/494	494	C ₃₃ H ₆₆ O ₂	0.4
<i>n</i> -Tetraatriacontanoic acid	60/73/129/508	508	C ₃₄ H ₆₈ O ₂	5.1
α-Hydroxyfatty acids				102.8
2-Hydroxytricosanoic acid	73/397/441 ^b	456 ^b	C ₂₇ H ₅₆ O ₃ Si ^b	20.6
2-Hydroxytetracosanoic acid	73,411,455 ^b	470 ^b	C ₂₈ H ₅₈ O ₃ Si ^b	54.8
2-Hydroxypentacosanoic acid	73,425,469 ^b	484 ^b	C ₂₉ H ₆₀ O ₃ Si ^b	27.4
ω-Hydroxyfatty acids				34.5
22-Hydroxydocosanoic acid	73,395,411,427 ^b	442 ^b	C ₂₆ H ₅₄ O ₃ Si ^b	30.6
24-Hydroxytetracosanoic acid	73,423,439,455 ^b	470 ^b	C ₂₈ H ₅₈ O ₃ Si ^b	3.9
Fatty alcohols				127.0
<i>n</i> -Hexadecanol	75/103/299 ^a	314 ^a	C ₁₉ H ₄₂ OSi ^a	2.1
<i>n</i> -Octadecanol	75/103/327 ^a	342 ^a	C ₂₁ H ₄₆ OSi ^a	2.4
<i>n</i> -Docosanol	75/103/383 ^a	398 ^a	C ₂₅ H ₅₄ OSi ^a	8.9
<i>n</i> -Tetracosanol	75/103/411 ^a	426 ^a	C ₂₇ H ₅₈ OSi ^a	2.1
<i>n</i> -Hexacosanol	75/103/439 ^a	454 ^a	C ₂₉ H ₆₂ OSi ^a	3.3
<i>n</i> -Octacosanol	75/103/467 ^a	482 ^a	C ₃₁ H ₆₆ OSi ^a	12.0
<i>n</i> -Triacontanol	75/103/495 ^a	510 ^a	C ₃₃ H ₇₀ OSi ^a	24.2
<i>n</i> -Dotriacontanol	75/103/523 ^a	538 ^a	C ₃₅ H ₇₄ OSi ^a	30.8
<i>n</i> -Tetraatriacontanol	75/103/551 ^a	566 ^a	C ₃₇ H ₇₈ OSi ^a	28.0
<i>n</i> -Hexatriacontanol	75/103/579 ^a	594 ^a	C ₃₉ H ₈₂ OSi ^a	6.2
<i>n</i> -Octatriacontanol	75/103/607 ^a	622 ^a	C ₄₁ H ₈₆ OSi ^a	6.2
<i>n</i> -Tetracontanol	75/103/635 ^a	650 ^a	C ₄₃ H ₉₀ OSi ^a	1.0
Steroid hydrocarbons				16.1
Ergosta-3,5,7-triene	135/143/380	380	C ₂₈ H ₄₄	0.9
Stigmastatetraene	83/143/157/392	392	C ₂₉ H ₄₄	5.4
Stigmasta-3,5,7-triene	135/143/394	394	C ₂₉ H ₄₆	6.0
Stigmasta-3,5-diene	81/147/381/396	396	C ₂₉ H ₄₈	3.7
Sterols				43.4
Campesterol	55/145/213/382/400	400	C ₂₈ H ₄₈ O	6.8
Stigmasterol	55/83/255/394/412	412	C ₂₉ H ₄₈ O	9.6
Sitosterol	145/213/396/414	414	C ₂₉ H ₅₀ O	20.7
Stigmastanol	215/416	416	C ₂₉ H ₅₂ O	1.0
7-Oxo-campesterol	135,161,381,414	414	C ₂₈ H ₄₀ O ₂	1.0
7-Oxo-sitosterol	135,161,395,428	428	C ₂₉ H ₄₂ O ₂	4.3
Epoxydammarane-type triterpenoids				64.8
Unknown compound peak 8	143/367	n.d.	n.d.	51.7
Unknown compound peak 9	143/365	n.d.	n.d.	13.1

Table 1 (Continued)

Compounds	Mass fragments	MW	Formula	Abundance
Tocopherols				3.1
γ-Tocopherol	151/191/416	416	C ₂₈ H ₄₈ O ₂	1.0
α-Tocopherol	165/205/430	430	C ₂₉ H ₅₀ O ₂	2.1
Steroid/triterpenoid ketones				25.1
Stigmasta-3,5-dien-7-one	174/269/410	410	C ₂₉ H ₅₀ O	2.9
Stigmast-4-en-3-one	124/229/412	412	C ₂₉ H ₄₈ O	2.6
β-Amyrenone	189/203/218/424	424	C ₃₀ H ₄₈ O	1.0
Isomultiflorenone	205/245/257/424	424	C ₃₀ H ₄₈ O	17.6
Baurenone	205/245/257/424	424	C ₃₀ H ₄₈ O	1.0
Steryl glycosides				8.4
Campesteryl 3β-D-glucopyranoside	204/217/361/383 ^a	850 ^a	C ₄₆ H ₉₀ O ₆ Si ₄ ^a	1.0
Stigmasteryl 3β-D-glucopyranoside	204/217/361/395 ^a	862 ^a	C ₄₇ H ₉₀ O ₆ Si ₄ ^a	1.9
Sitosteryl 3β-D-glucopyranoside	204/217/361/397 ^a	864 ^a	C ₄₇ H ₉₂ O ₆ Si ₄ ^a	5.5
Waxes				181.2
Ester C ₄₂	57/257/313/620	620	C ₄₂ H ₈₄ O ₂	0.7
Ester C ₄₄	57/257/285/341/648	648	C ₄₄ H ₈₈ O ₂	2.9
Ester C ₄₅	57/243/257/662	662	C ₄₅ H ₉₀ O ₂	1.1
Ester C ₄₆	57/257/285/676	676	C ₄₆ H ₉₂ O ₂	14.5
Ester C ₄₇	57/243/257/271/690	690	C ₄₇ H ₉₄ O ₂	3.1
Ester C ₄₈	57/257/285/704	704	C ₄₈ H ₉₆ O ₂	31.4
Ester C ₄₉	57/243/257/271/718	718	C ₄₉ H ₉₈ O ₂	3.4
Ester C ₅₀	57/257/285/313/752	732	C ₅₀ H ₁₀₀ O ₂	30.2
Ester C ₅₁	57/257/271/285/746	746	C ₅₁ H ₁₀₂ O ₂	2.0
Ester C ₅₂	57/257/285/313/760	760	C ₅₂ H ₁₀₄ O ₂	24.4
Ester C ₅₃	57/313/327/341/774	774	C ₅₃ H ₁₀₆ O ₂	1.7
Ester C ₅₄	57/313/341/368/788	788	C ₅₄ H ₁₀₈ O ₂	26.5
Ester C ₅₅	57/341/355/369/802	802	C ₅₅ H ₁₁₀ O ₂	1.6
Ester C ₅₆	57/341/369/397/816	816	C ₅₆ H ₁₁₂ O ₂	27.0
Ester C ₅₈	57/341/369/397/844	844	C ₅₈ H ₁₁₆ O ₂	10.7
Monoglycerides				7.5
2,3-Dihydroxypropylhexadecanoate	73/147/203/371/459 ^a	474 ^a	C ₂₅ H ₅₄ O ₄ Si ^a	2.6
2,3-Dihydroxypropyloctadec-9-enoate	73/129/147/397/485 ^a	500 ^a	C ₂₃ H ₅₆ O ₄ Si ^a	1.9
2,3-Dihydroxypropyloctadecanoate	73/147/203/399/487 ^a	502 ^a	C ₂₇ H ₅₈ O ₄ Si ^a	1.9
2,3-Dihydroxypropyltriacontanoate	73/147/203/567/655 ^a	670 ^a	C ₃₉ H ₈₂ O ₄ Si ^a	0.5
2,3-Dihydroxypropyldotriacontanoate	73/147/203/595/683 ^a	698 ^a	C ₄₁ H ₈₆ O ₄ Si ^a	0.6
Diglycerides				13.5
1,2-Dipalmitoylglycerol	73/129/313/625 ^a	640 ^a	C ₃₈ H ₇₆ O ₅ Si ^a	1.0
1,3-Dipalmitoylglycerol	73/129/313/371/625 ^a	640 ^a	C ₃₈ H ₇₆ O ₅ Si ^a	3.7
1,2-Oleoylpalmitoylglycerol	73/129/313/339/651 ^a	666 ^a	C ₄₀ H ₇₈ O ₅ Si ^a	1.2
1,3-Oleoylpalmitoylglycerol	73/129/339/371/651 ^a	666 ^a	C ₄₀ H ₇₈ O ₅ Si ^a	3.6
1,2-Distearoylglycerol	73/129/341/681 ^a	696 ^a	C ₄₂ H ₈₄ O ₅ Si ^a	1.4
1,3-Distearoylglycerol	73/129/341/399/681 ^a	696 ^a	C ₄₂ H ₈₄ O ₅ Si ^a	2.6
Total identified compounds				806.1

^a TMS ether.^b TMS ether, methyl ester.

380 °C (5 min) at 10 °C/min. The temperature of the injector during the injection was 120 °C, and 0.1 min after injection was programmed to 380 °C at a rate of 200 °C/min and held for 10 min. The temperature of the transfer line was set at 300 °C. Bis(trimethylsilyl)trifluoroacetamide (BSTFA) silylation were used where required. Compounds were identified by comparing their mass spectra with mass spectra in the Wiley and NIST libraries, by mass fragmentography and, when possible, by comparison with authentic standards.

3. Results and discussion

The total acetone extractives of jute fibers accounts for 0.4% of dry material. This content is similar to that found in other nonwood fibers (i.e. flax, hemp, kenaf, sisal, abaca or giant reed) commonly used for papermaking (Gutiérrez and del Río, 2003a; Gutiérrez et al., 2006, 2008; del Río and Gutiérrez, 2006; Coelho et al., 2007).

The underivatized and silylated lipophilic extracts from jute fibers were analyzed by GC and GC–MS using short- and medium-length high-temperature capillary columns, respectively, with thin films, according to the method previously described (Gutiérrez et al., 1998). The GC–MS chromatogram of the underivatized lipid

extracts from jute fibers is shown in Fig. 1. For a better characterization of the compounds present in the total extracts of jute fibers, the extracts were also subjected to a SPE fractionation according to the method developed by Gutiérrez et al. (1998). The chromatograms of the different SPE fractions are shown in Fig. 2. The first fraction (A), eluted with hexane, was enriched in hydrocarbons, ester waxes and triterpenoids and steroid ketones. The second fraction (B), eluted with chloroform, contained sterols, fatty alcohols and mono- and diglycerides. The final fraction (C), eluted with diethyl ether-acetic acid (98:2), was enriched in free fatty acids. The identities and abundances of the main lipophilic compounds identified among the jute lipophilic extractives are detailed in Table 1.

The most predominant lipophilic compounds present in jute fibers were series of high molecular weight ester waxes (24% of total identified compounds), followed by series of free fatty acids (17%), free fatty alcohols (17%) and α-hydroxyfatty acids (14%). Additionally, lower amounts of other aliphatic series such as *n*-alkanes (6%) and ω-hydroxyfatty acids (6%), were also found. Steroid and triterpenoid compounds were also present among jute lipophilic extracts, together with minor amounts of tocopherols as well as mono- and diglycerides. The structures of the main lipophilic compounds present in jute fibers are depicted in Fig. 3 and the

Fig. 3. Structures of the main lipophilic compounds identified in jute (*C. capsularis*) fibers and referred in the text. (I) hexadecanoic acid, dotriacontanyl ester; (II) *n*-trtriacontane; (III) hexadecanoic (palmitic) acid; (IV) 9-octadecenoic (oleic) acid; (V) 2-hydroxytetracontanoic acid; (VI) 22-hydroxydocosanoic acid; (VII) *n*-dotriacontanol; (VIII) α -tocopherol; (IX) campesterol; (X) stigmasterol; (XI) sitosterol; (XII) sitosteryl 3 β -D-glucopyranoside; (XIII) stigmasterane-3,5,7-triene; (XIV) stigmasta-3,5-dien-7-one; (XV) stigmast-4-en-3-one.

distributions of the main aliphatic series present in jute fibers are represented in the histograms of Fig. 4.

High molecular weight ester waxes (formed by long-chain fatty acids esterified to long-chain fatty alcohols) were found in a high abundance (181.2 mg/kg) among the jute extracts. High molecular weight ester waxes are also prominent among the extracts of other bast fibers, such as flax, hemp or kenaf (Gutiérrez and del Río, 2003a,b; Gutiérrez et al., 2006). They were present in the range from C_{42} to C_{58} , with a strong even-over-odd carbon atom predominance and the C_{48} , C_{50} , C_{52} , C_{54} and C_{56} homologues being the most abundant. Waxes with unsaturated fatty acids could not be detected, despite the relatively high amount of oleic acid being present in free form, as shown below. Each chromatographic ester peak consisted of a complex mixture of different long-chain fatty

acids esterified to different long-chain fatty alcohols that coelute together in the same peak. The identification and quantitation of the individual long-chain esters present in each chromatographic peak was accomplished based on the mass spectra of the peaks. Fig. 5 shows the mass spectrum of the chromatographic peak corresponding to the long-chain ester wax C_{44} . The mass spectra of long-chain esters are characterized by a base peak produced by a rearrangement process involving the transfer of 2 H atoms from the alcohol chain to the acid chain giving a protonated acid ion (Sharkey et al., 1959; Moldovan et al., 2002). The fragments at m/z 229, 257, 285, 313 and 341 of the mass spectra in Fig. 5 correspond therefore to protonated tetradecanoic, hexadecanoic, octadecanoic, eicosanoic and docosanoic acids, respectively. Therefore, this mass fragment gives information about the number of carbon atoms in

Fig. 4. Distribution of the main aliphatic series identified in the extracts of jute (*C. capsularis*) fibers. The histograms are scaled up to the abundance of the major compound in the series.

Fig. 5. Mass spectrum of the chromatographic peak corresponding to long-chain ester wax C_{44} . The fragments at m/z 229, 257, 285, 313 and 341 correspond to protonated tetradecanoic, hexadecanoic, octadecanoic, eicosanoic and docosanoic acids, respectively.

the acid moiety whereas the molecular ion (M^+ 648 in this particular case) gives information on the total number of carbon atoms in the ester (a C_{44} ester in this particular case), being then possible to determine the individual contribution of esters to every chromatographic peak by mass spectrometry. Quantitation of individual esters was accomplished by integration of the areas of the chromatographic profiles of the characteristic ions for the acidic moiety. A detailed structural characterization of the high molecular weight ester waxes present in the extracts of jute fibers is shown in Table 2. The distribution of the series of esterified fatty acids and fatty alcohols is shown in Fig. 4. The esterified fatty acids ranged from C_{14} to C_{30} and the esterified fatty alcohols ranged from C_{16} to C_{40} . The acid moiety of the waxes was exclusively constituted by saturated fatty acids with a very strong predominance of the even carbon atom number homologues. Among these, the most predominant one was hexadecanoic (C_{16}) acid followed by docosanoic (C_{22}) and tetracosanoic (C_{24}) acids. Among the alcohol moiety of the high molecular weight ester waxes, the most predominant was

Table 2Composition and abundance (mg/kg) of the individual high molecular weight ester waxes present in jute (*C. capsularis*) fiber extracts.

Individual esters	Fatty acid:fatty alcohol	Abundance
Ester C₄₂		0.7
Hexadecanoic acid, hexacosyl ester	C ₁₆ :C ₂₆	0.4
Octadecanoic acid, tetracosyl ester	C ₁₈ :C ₂₄	0.1
Eicosanoic acid, docosyl ester	C ₂₀ :C ₂₂	0.2
Ester C₄₄		2.9
Tetradecanoic acid, triacontyl ester	C ₁₄ :C ₃₀	0.5
Hexadecanoic acid, octacosyl ester	C ₁₆ :C ₂₈	1.7
Octadecanoic acid, hexacosyl ester	C ₁₈ :C ₂₆	0.2
Eicosanoic acid, tetracosyl ester	C ₂₀ :C ₂₄	0.1
Docosanoic acid, docosyl ester	C ₂₂ :C ₂₂	0.3
Tetracosanoic acid, heneicosyl ester	C ₂₄ :C ₂₀	0.1
Ester C₄₅		1.1
Tetradecanoic acid, hentriacontyl ester	C ₁₄ :C ₃₁	0.1
Pentadecanoic acid, triacontyl ester	C ₁₅ :C ₃₀	0.8
Hexadecanoic acid, nonacosyl ester	C ₁₆ :C ₂₉	0.2
Ester C₄₆		14.5
Tetradecanoic acid, dotriacontyl ester	C ₁₄ :C ₃₂	1.7
Hexadecanoic acid, triacontyl ester	C ₁₆ :C ₃₀	10.8
Octadecanoic acid, octacosyl ester	C ₁₈ :C ₂₈	0.5
Eicosanoic acid, hexacosyl ester	C ₂₀ :C ₂₆	0.2
Docosanoic acid, tetracosyl ester	C ₂₂ :C ₂₄	0.2
Tetracosanoic acid, docosyl ester	C ₂₄ :C ₂₂	0.4
Hexacosanoic acid, heneicosyl ester	C ₂₆ :C ₂₀	0.1
Octacosanoic acid, octadecyl ester	C ₂₈ :C ₁₈	0.1
Triacontanoic acid, hexadecyl ester	C ₃₀ :C ₁₆	0.5
Ester C₄₇		3.1
Pentadecanoic acid, dotriacontyl ester	C ₁₅ :C ₃₂	1.8
Hexadecanoic acid, hentriacontyl ester	C ₁₆ :C ₃₁	0.9
Heptadecanoic acid, triacontyl ester	C ₁₇ :C ₃₀	0.4
Ester C₄₈		31.4
Tetradecanoic acid, tetratriacontyl ester	C ₁₄ :C ₃₄	1.5
Hexadecanoic acid, dotriacontyl ester	C ₁₆ :C ₃₂	24.7
Octadecanoic acid, triacontyl ester	C ₁₈ :C ₃₀	3.5
Eicosanoic acid, octacosyl ester	C ₂₀ :C ₂₈	0.7
Docosanoic acid, hexacosyl ester	C ₂₂ :C ₂₆	0.2
Tetracosanoic acid, tetracosyl ester	C ₂₄ :C ₂₄	0.2
Hexacosanoic acid, docosyl ester	C ₂₆ :C ₂₂	0.3
Octacosanoic acid, heneicosyl ester	C ₂₈ :C ₂₀	0.1
Triacontanoic acid, octadecyl ester	C ₃₀ :C ₁₈	0.2
Ester C₄₉		3.4
Pentadecanoic acid, tetratriacontyl ester	C ₁₅ :C ₃₄	1.6
Hexadecanoic acid, tritriacontyl ester	C ₁₆ :C ₃₃	1.1
Heptadecanoic acid, dotriacontyl ester	C ₁₇ :C ₃₂	0.6
Octadecanoic acid, hentriacontyl ester	C ₁₈ :C ₃₁	0.1
Ester C₅₀		30.2
Tetradecanoic acid, hexatriacontyl ester	C ₁₄ :C ₃₆	0.3
Hexadecanoic acid, tetratriacontyl ester	C ₁₆ :C ₃₄	18.8
Octadecanoic acid, dotriacontyl ester	C ₁₈ :C ₃₂	5.7
Eicosanoic acid, triacontyl ester	C ₂₀ :C ₃₀	3.5
Docosanoic acid, octacosyl ester	C ₂₂ :C ₂₈	1.1
Tetracosanoic acid, hexacosyl ester	C ₂₄ :C ₂₆	0.5
Hexacosanoic acid, tetracosyl ester	C ₂₆ :C ₂₄	0.1
Octacosanoic acid, docosyl ester	C ₂₈ :C ₂₂	0.2
Ester C₅₁		2.0
Pentadecanoic acid, hexatriacontyl ester	C ₁₅ :C ₃₆	0.2
Hexadecanoic acid, pentatriacontyl ester	C ₁₆ :C ₃₅	0.2
Heptadecanoic acid, tetratriacontyl ester	C ₁₇ :C ₃₄	0.5
Octadecanoic acid, tritriacontyl ester	C ₁₈ :C ₃₃	0.2
Nonadecanoic acid, dotriacontyl ester	C ₁₉ :C ₃₂	0.2
Eicosanoic acid, hentriacontyl ester	C ₂₀ :C ₃₁	0.2
Heneicosanoic acid, triacontyl ester	C ₂₁ :C ₃₀	0.5
Ester C₅₂		24.4
Tetradecanoic acid, octatriacontyl ester	C ₁₄ :C ₃₈	0.4
Hexadecanoic acid, hexatriacontyl ester	C ₁₆ :C ₃₆	1.9
Octadecanoic acid, tetratriacontyl ester	C ₁₈ :C ₃₄	4.2
Eicosanoic acid, dotriacontyl ester	C ₂₀ :C ₃₂	9.0
Docosanoic acid, triacontyl ester	C ₂₂ :C ₃₀	7.4
Tetracosanoic acid, octacosyl ester	C ₂₄ :C ₂₈	0.8
Hexacosanoic acid, hexacosyl ester	C ₂₆ :C ₂₆	0.3

Table 2 (Continued)

Individual esters	Fatty acid:fatty alcohol	Abundance
Octacosanoic acid, tetracosyl ester	C ₂₈ :C ₂₄	0.2
Triacontanoic acid, docosyl ester	C ₃₀ :C ₂₂	0.2
Ester C₅₃		1.7
Pentadecanoic acid, octatriacontyl ester	C ₁₅ :C ₃₈	0.1
Heptadecanoic acid, heptatriacontyl ester	C ₁₇ :C ₃₇	0.1
Heneicosanoic acid, dotriacontyl ester	C ₂₁ :C ₃₂	1.0
Tricosanoic acid, triacontyl ester	C ₂₃ :C ₃₀	0.5
Ester C₅₄		26.5
Tetradecanoic acid, tetracontyl ester	C ₁₄ :C ₄₀	0.3
Hexadecanoic acid, octatriacontyl ester	C ₁₆ :C ₃₈	0.7
Octadecanoic acid, hexatriacontyl ester	C ₁₈ :C ₃₆	0.4
Eicosanoic acid, tetratriacontyl ester	C ₂₀ :C ₃₄	4.6
Docosanoic acid, dotriacontyl ester	C ₂₂ :C ₃₂	14.0
Tetracosanoic acid, triacontyl ester	C ₂₄ :C ₃₀	5.9
Hexacosanoic acid, octacosyl ester	C ₂₆ :C ₂₈	0.6
Ester C₅₅		1.6
Heneicosanoic acid, tetratriacontyl ester	C ₂₁ :C ₃₄	0.4
Tricosanoic acid, dotriacontyl ester	C ₂₃ :C ₃₂	0.8
Pentacosanoic acid, triacontyl ester	C ₂₅ :C ₃₀	0.4
Ester C₅₆		27.0
Hexadecanoic acid, tetracontyl ester	C ₁₆ :C ₄₀	0.5
Octadecanoic acid, octatriacontyl ester	C ₁₈ :C ₃₈	0.1
Eicosanoic acid, hexatriacontyl ester	C ₂₀ :C ₃₆	0.8
Docosanoic acid, tetratriacontyl ester	C ₂₂ :C ₃₄	5.9
Tetracosanoic acid, dotriacontyl ester	C ₂₄ :C ₃₂	15.8
Hexacosanoic acid, triacontyl ester	C ₂₆ :C ₃₀	3.5
Octacosanoic acid, octacosyl ester	C ₂₈ :C ₂₈	0.4
Ester C₅₈		10.7
Tetracosanoic acid, tetratriacontyl ester	C ₂₄ :C ₃₄	3.9
Hexacosanoic acid, dotriacontyl ester	C ₂₆ :C ₃₂	6.8

dotriacontanol (C₃₂) followed by tetratriacontanol (C₃₄) and triacontanol (C₃₀). Therefore, the predominant ester wax found among the jute lipophilic extracts was C₄₈ (31.4 mg/kg), which is mostly constituted by hexadecanoic acid (C₁₆) esterified to dotriacontanol (C₃₂) (structure I in Fig. 3), followed by ester wax C₅₀ (30.2 mg/kg), mostly constituted by hexadecanoic acid (C₁₆) esterified to tetratriacontanol (C₃₄), ester wax C₅₆ (27.0 mg/kg), mostly constituted by tetracosanoic acid (C₂₄) esterified to dotriacontanol (C₃₂), and by ester wax C₅₄ (26.5 mg/kg), mostly constituted by docosanoic acid (C₂₂) esterified to dotriacontanol (C₃₂), as detailed in Table 2.

Free fatty acids were the second most important class of lipophilic compounds identified in jute extracts (130.2 mg/kg), ranging from tetradecanoic (C₁₄) to tetratriacontanoic (C₃₄) acids, with a strong even-over-odd carbon atom predominance. Hexadecanoic (palmitic, III) and 9-octadecenoic (oleic, IV) acids were the most abundant, followed by octadecanoic (stearic, C₁₈) and docosanoic (C₂₂) acids. Relatively high amounts of α -hydroxyfatty acids (102.8 mg/kg), ranging from C₂₃ to C₂₅ with C₂₄ (V) as the most abundant, and ω -hydroxyfatty acids (34.5 mg/kg) ranging from C₂₂ to C₂₄ (VI), the latest being the most abundant, were also identified. Series of α -hydroxyfatty acids have been reported previously in both bast (i.e. flax, hemp, kenaf) and leaf (i.e. sisal, abaca, curaua) fibers (Gutiérrez and del Río, 2003a; Gutiérrez et al., 2004, 2006, 2008; del Río and Gutiérrez, 2006; Marques et al., 2007). However, ω -hydroxyfatty acids have only been detected in leaf fibers (del Río and Gutiérrez, 2006; Gutiérrez et al., 2008; Marques et al., 2007), and this is the first report of the presence of these compounds in a bast fiber.

Series of *n*-fatty alcohols were present in the jute extracts in important amounts (127.0 mg/kg), ranging from C₁₆ to C₄₀ and with the presence of exclusively the even carbon atom numbered homologues. Dotriacontanol (C₃₂, VII) was the most abundant homologue. Interestingly, the distribution of the series of free

fatty alcohols parallels the distribution of the series of esterified fatty alcohols in the long-chain ester waxes, as also observed in the ester wax series present in other bast fibers such as flax, hemp or kenaf (Gutiérrez and del Río, 2003a,b; Gutiérrez et al., 2004, 2006). A series of *n*-alkanes was also identified among the extracts of jute fibers (48.5 mg/kg) ranging from heptacosane (C_{27}) to pentatriacontane (C_{35}) with a strong odd-over-even carbon atom number predominance, tritriacontane (C_{33} , II) being the most predominant. Finally, other aliphatic series present in smaller amounts are monoglycerides (7.5 mg/kg) and diglycerides (13.5 mg/kg), although triglycerides could not be found in jute extracts.

Sterols were also present in significant amounts (43.4 mg/kg) among the lipophilic extracts of jute fibers. Sitosterol (XI) was the most abundant among the free sterols, followed by stigmasterol (X) and campesterol (IX), with minor amounts of stigmastanol, 7-oxocampesterol and 7-oxositosterol. Steryl glycosides, such as campesteryl, stigmasteryl and sitosteryl 3 β -D-glucopyranosides (XII) were also identified although in minor amounts (8.4 mg/kg), the latter being the most predominant. The identification of steryl glycosides was accomplished (after BSTFA derivatization of the lipid extract) by comparison with the mass spectra and relative retention times of authentic standards (Gutiérrez and del Río, 2001).

Steroid hydrocarbons (ergosta-3,5,7-triene, XIII, stigmastatriene, stigmasta-3,5,7-triene and stigmasta-3,5-diene) and steroid ketones (stigmasta-3,5-dien-7-one, XIV and stigmasta-4-en-3-one, XV) were also present. Several triterpenoid ketones were also identified, the most abundant being isomultiflorenone (D:C friedoolean-8-en-3-one) with minor amounts of bauerenone (D:C friedours-7-en-3-one) and β -amyrenone. The identification of the bauerenone and β -amyrenone was accomplished after SPE fractionation due to their low abundance and coelution with other major lipids. The mass spectra of the triterpenoid ketones present in jute fiber extracts (shown in Fig. 6) are similar to those reported in the literature (Budzikiewicz et al., 1963; Talapatra et al., 1968).

Finally, two compounds (peaks 8 and 9 in Figs. 1 and 2), with similar and characteristic mass spectra (Fig. 7), were also detected in important amounts among the jute lipophilic extracts. These compounds coelute very closely and were concentrated in the SPE B fraction, where alcohols and sterols concentrate, indicating the possible presence of hydroxyl groups in their structure. The mass spectra of both compounds are characterized by a base peak at m/z 143, which is indicative of the presence of a hydroxypropyltetrahydrofuran side-chain (Fig. 7c), and are similar to the mass spectrum of the epoxy-dammarane triterpene capsugenin, a triterpenoid with a hydroxypropyltetrahydrofuran side-chain (Fig. 7d), previously identified in the extracts of jute leaves (Hasan et al., 1984; Quader and Ahmed, 1987; Quader et al., 1990). The molecular weight of these compounds could not be determined despite they were subjected to different derivatizations, although the fragments at m/z 444 and m/z 442 seem to correspond to a loss of water $[M-18]^+$. The mass spectrum reported for capsugenin is very similar to the mass spectra of unknown compounds 8 and 9, with the same base peak at m/z 143 and a similar fragmentation profile. Therefore, these unknown compounds 8 and 9 seem to be structurally related to capsugenin.

We have reported the detailed chemical composition of the lipophilic extractives present in jute fibers. The knowledge of the chemical composition of lipophilic components of jute fiber will assist to predict and control the eventual pitch problems that may occur during pulping and bleaching and to establish appropriate methods and strategies for their control. In general terms, the lipids present in jute fibers can be classified into two principal groups, namely fatty acids (including α - and ω -hydroxyfatty acids) and neutral components, including wax esters, long-chain *n*-fatty alcohols, alkanes, and steroids and triterpenoids. The differ-

Fig. 6. Mass spectra and chemical structures of triterpenoid ketones identified among jute lipophilic extractives. (a) Isomultiflorenone; (b) bauerenone; and (c) β -amyrenone.

ent lipid classes have different behavior during cooking. The wax esters, which are the most abundant lipophilic compounds in jute fibers, are hydrolyzed during alkaline cooking and the fatty acids are dissolved. At sufficiently high pH (as in alkaline pulping), the acids dissociate and form fatty acid soaps and can thus dissolve in water to quite a high extent. On the other hand, alkanes, fatty alcohols, sterols and triterpenols, steroid hydrocarbons and ketones, and steryl glycosides do not form soluble soaps under the alkaline pulping conditions and therefore survive cooking and can be found intact in the pulp. These compounds have a very low solubility in water and are difficult to remove, and therefore can be at the origin of pitch deposition. The low amounts of these neutral compounds in jute fibers, and particularly the low abundances of free and conjugated sterols, which have a high propensity to form pitch deposits (del Río et al., 1998, 2000; Gutiérrez and del Río, 2001) would point to a low pitch deposition tendency of the lipophilics from jute fibers. On the other hand, fatty acid soaps are effective solubilizing agents facilitating the removal from pulp of sparingly soluble neutral substances. The ratio of saponifiables-to-unsaponifiables has been suggested to be a better index for predicting pitch problems than the total amount of lipids (Back and Allen, 2000). In fact, the higher abundances of unsaponifiable compounds (neutrals) with respect to the saponifiable ones are the main cause for pitch problems during pulping of some woods, such as aspen or eucalypt (Chen et al., 1995; del Río et al., 1998, 2000). In jute fibers the content of free fatty acids (including α - and ω -hydroxyfatty acids) is high (around 33% of total lipids), and therefore the fatty acid soaps formed during the cooking may possess sufficient micellar-forming properties to carry the less polar compounds into solution.

Fig. 7. Mass spectra of two unknown compounds present in jute (*C. capsularis*) fiber extracts. (a) Mass spectrum of unknown compound 8; (b) mass spectrum of unknown compound 9; (c) structure of the mass fragment at m/z 143; (d) structure of capsugenin (20,24-epoxy-3,12,25,30-tetrahydroxydammarane).

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References

- Akhtaruzzaman, A.F.M., Shafi, M., 1995. Pulping of jute. *Tappi J.* 78, 106–112.
- Ali, M., Sreekrishnan, T.R., 2001. Aquatic toxicity from pulp and paper mill effluents: a review. *Adv. Environ. Res.* 5, 175–196.
- Back, E.L., Allen, L.H., 2000. Pitch Control Wood Resin and Deresination. Tappi Press, Atlanta, GA.
- Bergelin, E., Holmbom, B., 2003. Deresination of birch kraft pulp in bleaching. *J. Pulp Paper Sci.* 29, 29–34.
- Bergelin, E., Moller, R., Holmbom, B., 2005. Analysis of pitch and deposit samples in kraft pulp production. *Pap. Puu-Pap. Tim.* 87, 399–403.
- Budzikiewicz, H., Wilson, J.M., Djerassi, C., 1963. Mass spectrometry in structural and stereochemical problem XXXII. Pentacyclic triterpenes. *J. Am. Chem. Soc.* 85, 3688–3699.
- Chen, T., Wang, Z., Zhou, Y., Breuil, C., Aschim, O.K., Yee, E., Nadeau, L., 1995. Using solid-phase extraction to assess why aspen causes more pitch problems than softwoods in kraft pulping. *Tappi J.* 78, 143–149.
- Coelho, D., Marques, G., Gutiérrez, A., Silvestre, A.R.D., del Río, J.C., 2007. Chemical characterization of the lipophilic fraction of Giant reed (*Arundo donax*) fibres used for pulp and paper manufacturing. *Ind. Crops Prod.* 26, 229–236.

- Christianson-Heiska, I.L., Haavisto, T., Paranko, J., Bergelin, E., Isomaa, B., 2008. Effects of the wood extractives dehydroabietic acid and betulinol on reproductive physiology of zebrafish (*Danio rerio*)—a two-generation study. *Aquat. Toxic.* 86, 388–396.
- del Río, J.C., Gutiérrez, A., González-Vila, F.C., Martín, F., Romero, J., 1998. Characterization of organic deposits produced in kraft pulping of *Eucalyptus globulus* wood. *J. Chromatogr. A* 823, 457–465.
- del Río, J.C., Romero, J., Gutiérrez, A., 2000. Analysis of pitch deposits produced in kraft pulp mills using a totally chlorine free bleaching sequence. *J. Chromatogr. A* 874, 235–245.
- del Río, J.C., Gutiérrez, A., 2006. Chemical composition of abaca (*Musa textilis*) leaf fibers used for manufacturing of high quality paper pulps. *J. Agric. Food Chem.* 54, 4600–4610.
- Freire, C.S.R., Silvestre, A.J.D., Pascoal Neto, C., 2005. Lipophilic extractives in *Eucalyptus globulus* kraft pulps. Behaviour during ECF bleaching. *J. Wood Chem. Technol.* 25, 67–80.
- Freire, C.S.R., Silvestre, A.J.D., Pascoal Neto, C., Evtuguin, D.V., 2006. Effect of oxygen, ozone and hydrogen peroxide bleaching stages on the contents and composition of lipophilic extractives of *Eucalyptus globulus* kraft pulps. *Biores. Technol.* 97, 420–428.
- Gutiérrez, A., del Río, J.C., González-Vila, F.J., Martín, F., 1998. Analysis of lipophilic extractives from wood and pitch deposits by solid-phase extraction and gas chromatography. *J. Chromatogr. A* 823, 449–455.
- Gutiérrez, A., del Río, J.C., 2001. Gas chromatography/mass spectrometry demonstration of sterol glycosides in eucalypt wood, kraft pulp and process liquids. *Rapid Commun. Mass Spectrom.* 15, 2515–2520.
- Gutiérrez, A., Romero, J., del Río, J.C., 2001a. Lipophilic extractives from *Eucalyptus globulus* pulp during kraft cooking followed by TCF and ECF bleaching. *Holzforchung* 55, 260–264.
- Gutiérrez, A., Romero, J., del Río, J.C., 2001b. Lipophilic extractives in process waters during manufacturing of totally chlorine free kraft pulp from eucalypt wood. *Chemosphere* 44, 1237–1242.
- Gutiérrez, A., del Río, J.C., 2003a. Lipids from flax fibers and their fate in alkaline pulping. *J. Agric. Food Chem.* 51, 4965–4971.
- Gutiérrez, A., del Río, J.C., 2003b. Lipids from flax fibers and their fate in alkaline pulping (addition/correction). *J. Agric. Food Chem.* 51, 6911–6914.
- Gutiérrez, A., Rodríguez, I.M., del Río, J.C., 2004. Chemical characterization of lignin and lipid fractions in kenaf bast fibers used for manufacturing high-quality papers. *J. Agric. Food Chem.* 52, 4764–4773.
- Gutiérrez, A., del Río, J.C., 2005. Chemical characterization of pitch deposits produced in the manufacturing of high-quality paper pulps from hemp fibers. *Biores. Technol.* 96, 1445–1450.
- Gutiérrez, A., Rodríguez, I.M., del Río, J.C., 2006. Chemical characterization of lignin and lipid fractions in industrial hemp bast fibers used for manufacturing high-quality paper pulps. *J. Agric. Food Chem.* 54, 2138–2144.
- Gutiérrez, A., Rodríguez, I.M., del Río, J.C., 2008. Chemical composition of lipophilic extractives from sisal (*Agave sisalana*) fibers. *Ind. Crops Prod.* 28, 81–87.
- Hasan, C.M., Isla, A., Ahmed, M.-D., Waterman, P.G., 1984. Capsugenin, a dammarane triterpene from *Corchorus capsularis*. *Phytochemistry* 23, 2583–2588.
- Hillis, W.E., Sumimoto, M., 1989. Effect of extractives on pulping. In: Rowe, J.W. (Ed.), *Natural Products of Woody Plants II*. Springer-Verlag, Berlin, pp. 880–920.
- Jahan, M.S., 2001. Evaluation of additive in soda pulping of jute. *Tappi J.* 84, 1–11.
- Jahan, M.S., Al-Maruf, A., Quaiyyum, M.A., 2007. Comparative studies of pulping of jute fiber, jute cutting and jute caddis. *Bangladesh J. Sci. Ind. Res.* 42, 425–434.
- Jahan, M.S., Kanna, G.H., Mun, S.P., Chowdhury, D.A.N., 2008. Variations in chemical characteristics and pulpability within jute plant (*Corchorus capsularis*). *Ind. Crops Prod.* 28, 199–205.
- Marques, G., Gutiérrez, A., del Río, J.C., 2007. Chemical characterization of lignin and lipophilic fractions from leaf fibers of curaua (*Ananas erectifolius*). *J. Agric. Food Chem.* 55, 1327–1336.
- Moldovan, Z., Jover, E., Bayona, J.M., 2002. Systematic characterisation of long-chain aliphatic esters of wool wax by gas chromatography-electron impact ionisation mass spectrometry. *J. Chromatogr. A* 952, 193–204.
- Quader, M.A., Ahmed, M.-D., 1987. Capsugenin 30-O- β -diglucopyranoside: a new glycoside from the leaves of *Corchorus capsularis*. *J. Nat. Prod.* 50, 479–481.
- Quader, M.A., Gray, A.I., Waterman, P.G., 1990. Capsugenin-25 30-O- β -diglucopyranoside: a new glycoside from the leaves of *Corchorus capsularis*. *J. Nat. Prod.* 53, 527–530.
- Sharkey Jr., A.G., Shultz, J.L., Friedel, R.A., 1959. Mass spectra of esters. Formation of rearrangement ions. *Anal. Chem.* 31, 87–94.
- Silvestre, A.J.D., Pereira, C.C.L., Pascoal Neto, C., Evtuguin, D.V., Duarte, A.C., Cavaleiro, J.A.S., Furtado, F.P., 1999. Chemical composition of pitch deposits from ECF *Eucalyptus globulus* bleached kraft pulp mill: Its relationship with wood extractives and additives in process streams. *Appita J.* 52, 375–382.
- Talapatra, S.K., Sengupta, S., Talapatra, B., 1968. A pentacyclic triterpene alcohol from *Evodia fraxinipolia* Hook F. *Tetrahedron Lett.* 57, 5963–5968.