Industrial Crops and Products 30 (2009) 241–249

Contents lists available at ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

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

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ARTICLE INFO

Article history:
Received 6 February 2009
Received in revised form 14 April 2009
Accepted 16 April 2009

Keywords:
Jute
Corchorus capsularis
Nonwood
Lipids
Fatty acids
Waxes
Sterols
Triterpenoids
Isomultiflorenone
Bauerenone

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.

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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doi:10.1016/j.indcrop.2009.04.001

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.

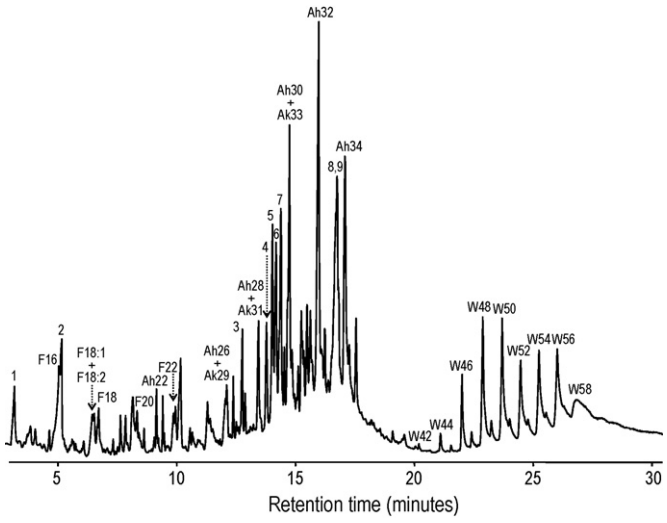

Fig. 1. GC–MS chromatogram of the underivatized lipid extracts from jute (*C. capsularis*) fibers. F(*n*): *n*-fatty acid series; Ak(*n*): *n*-alkane series; Ah(*n*): *n*-fatty alcohol series; W(*n*): high molecular weight ester wax series; *n* denotes the total carbon atom number. Other compounds reflected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

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

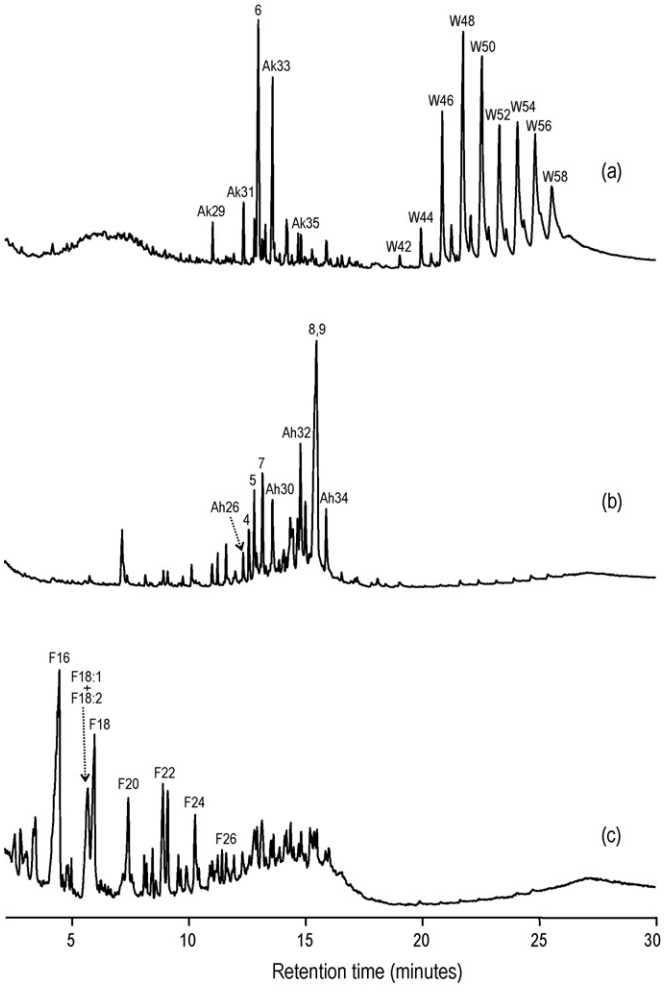

Fig. 2. GC–MS chromatograms of the different SPE fractions isolated from the extracts of jute fibers. (a) Fraction A, eluted with 8 mL of hexane; (b) fraction B, eluted with 10 mL of chloroform; and (c) fraction C, eluted with 10 mL diethyl ether:acetic acid (98:2). F(*n*): *n*-fatty acids series; Ak(*n*): *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) stigmasterol; (6) isomultiflorenone; (7) sitosterol; (8 and 9) unidentified triterpenoid compounds with epoxydammarane-type structure.