



Effect of harvest time and field retting duration on the chemical composition, morphology and mechanical properties of hemp fibers

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ABSTRACT

The large variability in the mechanical properties of hemp fibers is an issue in relation to their use in high-grade composites. The objective of the present study was to determine the optimal growth stage for harvesting hemp fibers for use in composites and to evaluate the effect of field retting time on mechanical performance of the fibers. Reduction in bast content and thickness of the primary bast fiber layer in stems were found to be highly significant ($P < 0.01$) with plant maturity. A significant increase in the secondary fiber fraction occurred with maturity, reaching a maximum value of 10% at seed maturity. A highly significant reduction in cellulose deposition in fiber cell walls was reflected by reduced fiber wall thickness with plant maturity and was related to the development and ripening of hemp seeds. A statistically significant increase in lignin deposition and a slight decrease in pectins in hemp fiber cell walls were also noted with stem maturity. Microscopy observations and histochemical analyzes corroborated the results from the chemical analyzes and revealed variations in morphological aspects and spatial micro-distributions of carbohydrates and lignin within the cell structure of the hemp stems between early- and late growth phases. Fibers harvested at the beginning of flowering exhibited high tensile strength and strain, which decreased with plant maturity. Reduction in strength was related to the increase in proportion of secondary fibers and decrease in cellulose deposition leading to inferior properties of fibers. A negative effect of field retting occurred only after extended field retting (i.e., 70 days) which was presumably due to accelerated degradation of cellulose by the action of microorganisms.

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1. Introduction

Hemp fibers are cellulose-rich cells that are attractive as reinforcement agents in composite materials due to their low cost and density, good mechanical properties and potential sustainability and biodegradability (Islam et al., 2011; Thygesen et al., 2005, 2011). The hemp fibers suitable for composites are the primary- and secondary fibers (i.e., bast fibers) situated in the cortex of the hemp plant stem. The bast fibers encircle the core xylem and originate from the procambium and correspond to sclerenchyma primary cells (Crônier et al., 2005; Esau, 1943). Their morphological features differ significantly from those of xylem fibers. In addition, the morphology and chemical composition of bast fibers vary with

maturity (Charlet et al., 2007; Duval et al., 2011; Mediavilla et al., 2001). This results in large variations in the mechanical properties of the fibers (Placet et al., 2012), and these variations are generally considered a major barrier for using hemp fibers in composites where high reliability and stability of fiber properties are required.

The main chemical components of hemp fiber cell walls are cellulose, hemicelluloses, lignin and pectin and the fibers are bound together by a pectin and lignin-rich middle lamella (ML) (Love et al., 1994; Nykter et al., 2008). For high-grade composites, the ML-fiber-fiber bonding must be degraded to obtain individual fibers and/or small fiber bundles. Therefore to increase the ease of fiber extraction from plants and reduce fiber breakage, the stems are normally retted before mechanical separation (termed “decortication”). The retting stage is critical for the broad use of hemp fibers with respect to economic aspects and fiber quality (Keller et al., 2001; Mediavilla et al., 2001).

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In field retting (also known as dew-retting), plant stems are spread out in the fields where they are attacked mainly by fungi. The pectinolytic enzymes expressed by the fungi can degrade the pectin in the middle lamella regions between fibers (Henriksson et al., 1997). Field retting is still widely used due to its low cost (Bacci et al., 2010), but is limited to geographic regions where weather conditions are suitable for fungi proliferation. Field retting also causes many problems such as increased scattering of fiber properties, insecurity of fiber supply due to poor weather conditions and may also cause delays in the planting of subsequent crops. Due to these disadvantages, more efficient and controllable methods have been investigated, including chemical treatment (Song and Obendorf, 2006), mechanical defibration (Vignon et al., 1996), and enzymatic retting (Li and Pickering, 2008). However, those methods require high energy input or expensive enzymes and/or may generate costly wastes (Tahir et al., 2011).

For natural fibers, their mechanical performance (e.g., tensile strength) largely depends on a number of crucial physical and chemical parameters, and the mechanical performances of natural hemp fibers were recently shown to be highly dependent on fiber diameter (Duval et al., 2011; Marrot et al., 2013; Placet et al., 2012). Tensile strength is found to decrease as the fiber diameter increases and this diameter dependence is closely related to both the number of fiber defects (i.e., kinks or dislocations) and number of single fibers contained in the fiber bundles (Fan, 2010).

Hemp contains secondary bast fibers situated outside the vascular cambium. These secondary fibers are shorter (approx. 2 mm long) and thinner (approx. 15 μm in diameter) than primary fibers (i.e., tenths of mm in length and 18–24 μm in diameter) (Mishra 2009; Sankari 2000a). According to Amaducci et al. (2008), these secondary fibers are primarily located at the bottom of the plant stem. Their formation has been reported to cause a reduction in both fiber yield and quality after flowering (Mediavilla et al., 2001). Thus, the mechanical properties of hemp fibers are dependent on many parameters such as fiber diameter, defects, chemical composition, and the presence/proportion of secondary fibers.

The aims of the present work were to provide an improved understanding of the reduction of mechanical properties of hemp fibers in relation to morphological features and chemical composition during growth and field retting.

2. Materials and methods

2.1. Raw material

2.1.1. Cultivation and harvest

The hemp (*Cannabis sativa* L.), variety USO-31, was sown at a rate of 45 kg/ha on May 5th 2013 in France (N 48.85°, E 3.02° (WGS84)) by hemp cultivation companies (Planète Chanvre and Bafa Neu GmbH). The seeds were sown with the seed drill 3–4 inches deep. The hemp plants were fertilized with 80 kg/ha N, 45 kg/ha K and 45 kg/ha P. The monoecious hemp plants were harvested at two developmental stages: (1) at the beginning of flowering (i.e., early harvest on July 18th 2013); and (2) seed maturity (i.e., late harvest on Sep 6th 2013). Using the definition of hemp growth stages given by Mediavilla et al. (1998), the two stages selected correspond to codes 2101 and 2204. Precipitation was not well distributed over the season (beginning of May to the end of September, 2013). From sowing date to the early harvest date, the weather was cool with an average temperature of 14.5 °C and precipitation was relatively evenly distributed with a total of 115 mm. Between early and late harvest (first retting period for early harvest sample, 50 days in total), the weather was quite hot (especially during daytime) with an average temperature of 19.6 °C and dry with a precipitation of 52 mm. From late harvest to the end of field retting (20 days in

total), the weather was humid with a total precipitation of 41 mm and cool with an average temperature of 14.2 °C (Fig. 1).

2.1.2. Sampling and storage

In this study, sampling involved a complete randomized block design with three replications. With each harvest (i.e., early- and late harvest), in every replicate, 10 m² above-ground part of hemp was harvested and the number of plants was determined. Considering the high dependence of morphological feature, chemical composition and mechanical properties on hemp stem sections, only the bottom section (one third above the base of the stem) of the plant was investigated (Charlet et al., 2007; Duval et al., 2011). A randomized sample of 20 bottom sections of plants per replication was used to determine the diameter of hemp stem and bast content.

A flow diagram of the setup for this study is presented in Fig. 2. For early harvest, a randomized sample of 80 plants per replication was divided into 4 groups (Group 1–Group 4). For late harvest, a randomized sample of 100 plants per replication was divided into 5 groups (Group 1–Group 5).

2.2. Cell wall isolation

The bast fibers were air-dried at 40 °C with an air flow of 150 m³/(m² grid h) (Maskinfabrikken Thisted, Denmark Type 150) and ground with a crushing microfine grinder (IKA, MF 10.1) to a particle size of 1 mm. Samples of about 3 g were then extracted in a Soxhlet apparatus (Gerhardt EV6 All/16 No. 10-0012) for 5 h using a 300 mL solution of toluene-ethanol-acetone (4:1:1 by volume) (Sluiter et al., 2008; Özmen et al., 2013). For each sample, the final residue was dried at 50 °C for 12 h. and the resulting residue was designated as cell wall residue (CWR).

2.3. Chemical analysis of CWR

Chemical analyzes were done using two-step sulfuric acid hydrolysis at 72% and 4% (w/w), according to the method of the US National Renewable Energy Laboratory (Sluiter et al., 2011). After acid hydrolysis, the hydrolysate was collected for monosaccharide analysis. Hemp fibers are characterized by their low lignin content, and the lignin content of hemp bast fibers is usually characterized by Klason lignin. (Charlet et al., 2007; Gutiérrez et al., 2006). For Klason lignin analysis, a crucible with filtrated solids (i.e., acid-insoluble ash + acid-insoluble lignin) was dried in an oven at 105 °C for 12 h. After cooling to room temperature in a desiccator, the crucibles were weighed (W1). Subsequently, the crucibles were placed in a muffle furnace at 550 °C for 3 h, then cooled in the desiccator and reweighed as W2 (i.e., acid-insoluble ash). The amount of acid-insoluble lignin (also termed Klason lignin) was determined as W1–W2.

Monosaccharide analyzes were performed by HPAEC-PAD analysis using an ICS-3000 system consisting of a gradient pump (model DP-1), an electrochemical detector/chromatography module (model DC-1) and autosampler (Dionex Corp., Sunnyvale, CA). Separation was achieved using a CarboPacTM PA20 (3 mm $\times$ 150 mm) analytical column following that described by Arnous and Meyer (2008). Roughly, it is considered that arabinose, galactose, galacturonic acid and rhamnose are specific to pectins, and glucose belongs to the cellulose moiety (Cr  nier et al., 2005; Vignon and Garcia-Jald  n, 1996). The concentration of polymeric sugars was calculated from the concentration of the corresponding monomeric sugars, using an anhydrous correction of 0.88 for C-5 sugars and a correction of 0.9 for C-6 sugar (Sluiter et al., 2011).

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