Industrial Crops and Products 75 (2015) 206–211

Contents lists available at ScienceDirect

Industrial Crops and Products

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

Chemical composition and physical properties of dew- and water-retted hemp fibers

Zofija Jankauskienė [a,*], Bronislava Butkutė [b], Elvyra Gruzdevienė [a], Jurgita Cesevičienė [b], Ana Luisa Fernando [c]

[a] Upytė Experimental Station, Lithuanian Research Center for Agriculture and Forestry, Linininkų 3, Upytė, Panevėžys District, LT 38-294, Lithuania
[b] Institute of Agriculture, Lithuanian Research Center for Agriculture and Forestry, Instituto al. 1, Akademija, Kėdainiai District, LT-58344, Lithuania
[c] MEtRiCS, Departamento de Ciências e Tecnologia da Biomassa, Faculdade de Ciências e Tecnologia, FCT, Universidade Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal

ARTICLE INFO

Article history:
Received 14 January 2015
Received in revised form 12 June 2015
Accepted 15 June 2015
Available online 6 August 2015

Keywords:
Cannabis sativa L.
Cellulose
Cultivar
Dew- and water retting
Hemicellulose
Fiber
Flexibility
Lignin

ABSTRACT

Hemp (*Cannabis sativa* L.), as a multipurpose crop, is attracting more and more attention due to its economic importance allied with its renewable and sustainable character. In Lithuania, farmers have been allowed to grow industrial hemp only since 2014, yet, the declared area under this crop was over 1000 ha the same year. Hemp fibrous stem represent a source of industrial products but fiber extraction is limited to few methods, such as dew-retting (the most common) and water-retting. The aim of this study was to evaluate the effect of dew- and water-retting on the chemical composition and physical properties of hemp fibers obtained from different cultivars. Three replicated field experiment on eight hemp cultivars (Beniko (PL), Bialobrzeskie (PL), Epsilon 68 (FR), Fedora 17 (FR), Felina 32 (FR), Futura 75 (FR), Santhica 27 (FR), and USO 31 (FR)) were carried out during 2010–2011 in the Central Lowland of Lithuania. Hemp was sown (seeding rate 50 kg ha^{-1}) at the beginning of May, and harvested when the first mature seed appeared (in September or October, depending on the year and cultivar). Dew- and water-retting were applied for fiber loosening. Fiber flexibility and fiber strength and hemicellulose, cellulose, and Klason lignin were determined in the extracted fiber to characterize it, physically and chemically.

Three-way ANOVA was performed on the data to determine significance of the following factors: retting method, hemp cultivar, and harvest year. In all cases, the main constituent of hemp fiber was cellulose (the mean was close to 80% on dry matter of fiber basis). Lignin content consisted to an average of 11.2%. The *F*-test showed that retting method had a significant ($P < 0.01$) influence on fiber chemical composition. Water-retted stems provided higher content of cellulose (81.7%), hemicellulose (6.32%), but lower content of lignin (10.2%) in fiber when compared with that of dew-retted fiber (78.4, 5.9, and 13.1%, respectively). Cellulose content also depended on cultivar. The best fiber quality was for cv. Beniko (high cellulose and low ash content, high fiber content, and high fiber yield). The year also impacted cellulose and lignin content. In 2011, fiber had significantly higher lignin content (13.4%) and lower cellulose content (78.4%) than in 2010. Extracted fiber and fiber physical properties (flexibility, strength) dependency on retting method or other factors was inconsistent. Retting methods applied to extracted fiber had significant effect on fiber strength but not on flexibility. Fiber strength depended also on cultivar but not flexibility. The year affected both physical properties (flexibility and strength). In conclusion, the retting method is important to get a high quality fiber in hemp.

1. Introduction

Hemp is a valuable crop with economic importance representing a source for diverse uses and industrial applications (Amaducci and Gusovius, 2010; Salentijn et al., 2014). It is estimated that the global market for hemp consists of more than 25,000 products (Salentijn et al., 2014) and new applications of hemp products are continuously appearing. It is cultivated mainly for its fibrous

* Corresponding author.
E-mail addresses: soja@upyte.lzi.lt (Z. Jankauskienė), brone@lzi.lt (B. Butkutė), upyte@upyte.lzi.lt (E. Gruzdevienė), ala@fct.unl.pt (A.L. Fernando).

http://dx.doi.org/10.1016/j.indcrop.2015.06.044
0926-6690/

stem, being widely used in paper, textiles, construction, isolation, agriculture, composites, automotive, medicine, etc. (Delaney and Madigan, 2014; Fuqua et al., 2012; Salentijn et al., 2014). The long fiber can be spun into threads, made into twines, ropes, woven, canvas, etc. (Johnson, 2013; Karus and Vogt, 2004; Mwaikambo, 2006). A promising application for hemp fibers is the production of insulating products and fiber-reinforced composites due to the low density and good specific mechanical properties (Célino et al., 2014; Müssing et al., 2005; Zimniewska et al., 2011). Due to its high productivity and high cellulose content, hemp is also an interesting crop for energy and biofuel production (Prade et al., 2011, 2012; Satya and Maiti, 2013).

The physical and chemical properties of hemp fiber and the methods for its extraction are important to use it in different products. Several processes have been studied and applied for the hemp fiber extraction. Fiber bundles can be separated by using enzymatic, microbiological, chemical, and physical methods, but the developed methods, so far, do not show an economical viability for industrial upscaling (Amaducci and Gusovius, 2010; Reddy and Yang, 2005). The most widely used are dew- and water-retting methods, both carried out by pectic enzymes secreted by indigenous microflora (Bengtsson, 2009; Mwaikambo, 2006; Zhang et al., 2008). In dew retting, hemp stems are spread on the ground and the pectinolytic microorganisms, mainly aerobic fungi, disrupt pectins surrounding the fiber (Henriksson et al., 1997). In water retting, stems are soaked in water (nowadays in tanks, but in the past in ponds and rivers). The water, penetrating to the central stalk portion, breaks the outermost layer, thus, increasing absorption of both moisture and the developed pectinolytical bacterial community (Donaghy et al., 1990). Water retting produces, generally, higher quality fiber than those produced by dew-retting (Amaducci and Gusovius, 2010; Van Sumare, 1992; Xu, 2010). But the water retting process impacts the environment due to the consumption of large amounts of water (Van Dam and Bos, 2004) and energy (Van der Werf and Turunen, 2008).

The retting process is complex and depends on different factors (microbial content, duration of the degradation process which depends on environmental conditions). Under-retting makes separation difficult, and over-retting weakens the fiber (Preisner et al., 2014). The proportion of the main components in a fiber (cellulose, hemicellulose, and lignin), which defines its physical properties and applications, is also dependent on the retting process (Müssig and Martens, 2003). Fibers with high cellulose content, which provides strength and stability to the fiber (Reddy and Yang, 2005), are well suited to textile, paper, and other applications. Thus, fiber properties can be improved with the removal of lignin, pectin, and hemicellulose (Pickering et al., 2007) through processing. However, mechanical properties of pulp, composites, textile, and other end products are directly proportional not only to cellulose content but to its crystallinity also (Alemdar and Sain, 2008; John and Thomas, 2008; Mandolino and Carboni, 2004; Nadirah et al., 2012; Van der Werf et al., 1994). Mechanically, hemicellulose contributes little to the stiffness and strength of fibers or individual cells (Reddy and Yang, 2005). The mechanical properties of lignin are also lower than those of cellulose (Fuqua et al., 2012). The proportion of it impacts on the structure, properties, morphology, and flexibility of fibers as well as the rate of its hydrolysis (Reddy and Yang, 2005). Ash is a minor component but fibers containing more than 2% are poor feedstock for pulp, as that might accelerate mechanical depreciation of the processing equipment (Nadirah et al., 2012). Additionally, it can also affect the mechanical strength properties of the end products (Nadirah et al., 2012; Ververis et al., 2004).

The proportion of the main components in a fiber as well as fiber physical properties depend also on climatic conditions during hemp vegetative period, growing technology, cultivar, and growth/maturity stage at harvest (Amaducci et al., 2005; Célino et al., 2013; Keller et al., 2001; Mediavilla et al., 2001; Reddy and Yang, 2005). Chemical composition can also vary among plant parts (Keller et al., 2001).

Lithuania is more or less northerly, than countries from which hemp cultivars were originated, and because hemp is very sensitive to environmental conditions, such as day length and temperature, research on cultivars adaptation to Lithuanian environmental conditions is desirable. Therefore, the current study focuses on the aspects of variation in chemical composition and mechanical properties of extracted hemp fiber, depending on retting method and hemp cultivar grown under Lithuanian climatic conditions, in order to identify superior hemp genotypes. Moreover, this work will contribute to the missing data on fiber quality of the different hemp cultivars, once hemp genotypes have been mostly characterized by stem yield and fiber content.

2. Material and methods

The field experiment was conducted at the Upytė Research Station of the Lithuanian Research Centre for Agriculture and Forestry (the Central Lowland of Lithuania (55°39′11″N 24°13′59″E)) on an Eutri-Endohypogleyic Cambisol (FAO-UNESCO, 1997). In 2010–2011, a field experiment with 8 monoecious cultivars of hemp (in three replicates) was conducted. The examined cultivars were of different origin and different earliness. The hemp cultivar USO 31 (of Ukrainian origin) is known as very early cultivar. Polish cultivars Beniko and Bialobrzeskie are considered as medium-early in the country of their origin. The five other hemp cultivars were originated from France and they are classified as cultivars of different earliness: Fedora 17 is early-maturing, Felina 32 and Santhica 27 are medium-late maturing, Epsilon 68 is late maturing and Futura 75 is a very late-maturing cultivar.

Seeding rate of hemp was 50 kg ha^{-1}, inter-row spacing was 10 cm. Plot size was 20 m^2. Hemp was sown in randomized block at the beginning of May, and harvested when the first matured seed appeared (in September or October, depending on the year and cultivar). Hemp was harvested at selected time—when first matured seeds appeared, once field trials targeted the simultaneous production of fiber and seeds. In all years of experiment, the hemp cultivar USO 31 was harvested by 0.5–1 month earlier than others: 9 September in 2010 and 13 September in 2011. Remaining hemp cultivars were harvested 4 October in 2010 and 22–23 September in 2011.

Generally, weather conditions were favorable for hemp growing (Fig. 1). The amount of precipitation during hemp vegetation period was similar in both years (ca. 340–380 mm in 2010 and 320–350 mm in 2011), but the distribution of the precipitation was uneven: in 2010, in May and June, the amount of precipitation was twice as much than that in 2011 at the same period, while July and August of 2011 were more wet than the same period in 2010. In 2010, soil was richer in organic matter (2.33%), available phosphorus (245 mg kg^{-1}), and potassium (152 mg kg^{-1}), content than that in 2011, 1.89%; 137 mg kg^{-1}; and 139 mg kg^{-1}, respectively. Thus, in 2011, a higher rate of fertilizers was applied to hemp.

Dew- and water-retting was applied for fiber loosening before its extraction. Before starting dew or water retting, hemp stems was prepared by cutting away the top part of plant containing panicle and leaves. One part of hemp stem samples (0.5 kg per plot) was water retted (water temperature 37 °C) for 5 days, following the methodology presented by Arno et al. (1961). Hemp stalks were retted together with flax. In the big pool (ca. 6 × 3 × 2 m) all material for retting was spread, fixed by wooden deal on the top (to keep all material under the water), water was poured until the level above. Ratio stem mass to water was ca. 1 g of biomass per 7 L water. The other part (0.5 kg per plot) was dew retted on the grassland for