



Research Paper

Modification of hemp fibers (*Cannabis Sativa* L.) for composite applicationsTaneli Väisänen^{a,*}, Paolo Batello^{a,b}, Reijo Lappalainen^a, Laura Tomppo^a^a Department of Applied Physics, University of Eastern Finland, 70211 Kuopio, Finland^b School of Forestry, Wood Materials Science, University of Eastern Finland, 80101 Joensuu, Finland

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ABSTRACT

The use of bio-based constituents in polymer-based composites may provide multiple advantages in comparison to their synthetic counterparts in terms of performance, price, availability, and environmental impact. This study was set out to assess the effectiveness of different modification methods, i.e., alkali, enzymatic, steam, and wood distillate treatments, to enhance the characteristics of hemp fibers (*Cannabis Sativa* L.). Furthermore, the suitability of these fibers as reinforcements for epoxy resin was evaluated by preparing composites at approximately 30 wt% fiber contents. The results show that steam treated hemp fibers absorbed least water during 28 days at 65% relative humidity (RH) and 20.0 °C whereas the enzymatic treatment led to the lowest water absorption values at 85% RH and 20.0 °C. Mechanically treated hemp fibers were the strongest in terms of tensile properties. The modification of hemp fibers compromised the mechanical properties of epoxy-hemp composites but led to significantly lower water absorption values. Nevertheless, this study demonstrates that hemp fibers, either modified or unmodified, can act as an effective reinforcement in epoxy resin.

1. Introduction

Bio-based constituents are being increasingly utilized as fillers or reinforcements in polymer-based composites due to the ecological concerns related to the synthetic counterparts (Väisänen et al., 2017). The stringent regulations set by governments and regional councils together with the realization of finite nature of petroleum resources are highlighting the need to seek for new alternatives for materials derived from non-renewable resources (Das and Bhattacharyya, 2016; Väisänen et al., 2016a). The use of natural fibers for reinforcement in composites instead of synthetic ones provides multiple advantages, such as low density, ease of processing, abundance of raw materials, biodegradability, high toughness, and good specific mechanical properties.

Hemp fiber is one of the strongest and stiffest available natural fibers (Bledzki and Gassan, 1999; Faruk et al., 2012; Holbery and Houston, 2006; Ku et al., 2011; Lilholt and Lawther, 2000; Stokke et al., 2014; Zampaloni et al., 2007) and thus has a great potential as a composite reinforcement. The bast fibers in hemp are made up of approximately 74% cellulose, 14% hemicellulose, 5% lignin, 1% pectins, and 6% of other substances, such as waxes (Shahzad, 2012). The hemp stems are typically degraded to allow the fiber to be separated from the woody core; this is called retting. Sometimes the fibers are separated from the stalk through mechanical peeling called decortication or mechanical retting. Tahir et al. (2011) have divided the retting processes into dew retting, water retting, enzymatic retting, chemical retting, and

mechanical retting. After retting, the fibers can be separated from the other parts of the plant mechanically or through other extraction methods, such as steam explosion (Dhakal and Zhang, 2015).

Even though the use of natural fibers as a filler or reinforcement in composites provides multiple benefits, the inherent properties of natural fibers induce some undesired characteristics to the composites. Väisänen et al. (2017) identified four major challenges that can considerably limit the application of natural fibers in composites: limited mechanical properties, excess water absorption, poor fire resistance, and issues related to the processing and homogeneity. The parameters inducing variance especially in fiber properties are the time of harvest, the type of fiber processing, and the type of soil in which the plant is cultivated (Dhakal and Zhang, 2015). In order to at least partially solve the aforementioned challenges associated with natural fiber-polymer composites (NFPCs), several types of chemical and physical modification methods for polymer matrices and natural fibers have been developed (Adekunle, 2015; Dányádi et al., 2010; Moghadamzadeh et al., 2011; Mukhopadhyay and Figueiro, 2009; Oporto et al., 2007). However, more recently, industries and researchers have put more focus on the utilization of organic waste and residue materials as NFPC constituents, as the chemical and physical modification methods can be rather expensive and it is difficult to address all the problems associated with NFPCs with one single method (Das et al., 2015). For example, Das et al. (2016a,b,c) have shown that the addition of waste derived biochar has multiple beneficial effects on NFPCs without the need to include

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coupling agents, such as maleic anhydride-grafted polypropylene (MAPP), in the composites. Additionally, demonstrated that the inclusion of hardwood and softwood distillate has positive effects on the mechanical properties and water absorption of commercial wood-plastic composites (WPCs).

The aim of this study was to assess the effects of different treatments on the characteristics of hemp fibers and on the composites manufactured from them. The long (aligned) hemp fiber (*Cannabis Sativa* L.) reinforced epoxy composites were produced using vacuum assisted resin transfer molding (VARTM). These composites have potential in applications where directional reinforcement is required, such as building and constructions, automotives, and internal finishes. Hemp fibers were treated with alkali and enzymes as well as with hardwood and softwood distillate to enhance their properties. The mechanical properties of the fibers were studied using tensile tests. Fourier transform infrared spectroscopy (FTIR) was used to evaluate the effects of the treatments on the chemical properties of the fibers. The mechanical properties of the composites were assessed by tensile, flexural, and Charpy impact tests. Scanning electron microscopy (SEM) and optical microscopy were also used to study the morphology and dimensions of the fibers and composites.

2. Experimental

2.1. Materials

Analytical grade (98.6%) NaOH pellets were used for the alkali treatment of fibers. Araldite[®] LY 5052 low-viscosity epoxy resin and Aradur[®] 5052 epoxy hardener (Huntsman Advanced Materials, The Woodlands, TX) were used to prepare the matrix material for the composites. *Cannabis Sativa* L. (Futura 75) was sown in Juankoski, Finland, (63.0693889 N, 27.9991111 E) in the end of May 2016. The plants were harvested 127 days after sowing and the hemp stalks were immediately collected for further processing. The growing period of 127 days is close to the optimum growing period of 114 days observed by Pickering et al. (2007).

2.2. Methods

Before each treatment, unwanted pieces of woody core (hurd) were mechanically removed from the bast hemp fiber. Fibers were then dried in a forced-convection oven at 70 °C until the masses of the fibers remained constant (for at least 24 h). For mechanically treated fibers (also referred as unmodified fibers), only the removal of hurd and drying was performed before they were tested or used as composite reinforcements.

2.2.1. Alkali treatment

The procedure for the alkali treatment of the fibers was similar to the process described by Suardana et al. (2011). After the drying, fibers were placed into canisters of 5 L capacity. Pre-weighed NaOH solution (10 wt% concentration) was poured into the canisters, after which the canisters were placed into a forced-convection oven at 95 °C for 1 h. Fibers were then thoroughly washed with distilled water until the water dripping from the fibers had pH of approximately 7.0. After being washed, the fibers were dried in a forced-convection oven at 95 °C for at least 24 h. The mass losses of the dried fibers were determined in order to confirm successful treatments.

2.2.2. Enzymatic treatment

The treatment was performed by Arctic Fiber Company, Kiiminki, Finland. The activity of the enzyme preparation (incl. pectinase) was measured before starting the process, and it was fed into the system when necessary. Approximately 30 kg of hemp fibers were treated at 39–42 °C for 24–30 h depending on the conditions. The pH of the solution was 4.1–4.5 throughout the process. The treated fibers were

rinsed with water until the water dripping from the fibers reached pH of at least 6.0.

2.2.3. Steam treatment

Steam treatment is also an efficient method to modify the surfaces of natural fibers (Cao et al., 2012; Lavoie and Beauchet, 2012; Munawar et al., 2008). Steam treatment was performed at Natural Resources Institute Finland, Vantaa, by feeding steam (100 °C) through mechanically decorticated fibers at a speed of 400 mL/min for 60 min. The fibers were dried at 50 °C after the treatment.

2.2.4. Wood distillate treatment

Same wood distillates, i.e., hardwood and softwood distillate, with identical processing were used in the distillate treatments of the hemp fibers as for the commercial wood-plastic composites by . Wood distillates could act as efficient additives in NFPCs to enhance their properties. Before the fibers mats were coated with the distillates, mats with uniform masses were produced and dried in a forced-convection oven at 90–105 °C for at least 24 h, after which the fiber mats were weighed. The distillates were sprayed on the fiber mats with a spraying system so that the masses of the mats were continuously monitored. After the spraying, the distillate-treated mats were put in a forced-convection oven at 105 °C for at least 24 h to polymerize the distillates and to evaporate the solvents used in the distillates. Ultimately, the distillate content in the mats was approximately 10 wt%.

2.3. Testing and analyses of hemp fibers

The fibers were stored in a conditioned room at 20 °C and 65% relative humidity (RH) before testing. The tensile properties of the fiber bundles were characterized by means of a vertical column tester according to ASTM E3379-75. Casco[®] Strong Epoxy Professional (Sika Finland Ltd., Espoo, Finland) was used to glue and fix the fiber bundles on the cardboard tab. The resin was cured in a mildly ventilated oven at a temperature of 42 °C for a period of 20 h. The diameters of the fibers were measured using optical microscope and a caliper with accuracy of 10 µm. Ten fiber bundles of each fiber type were tested.

Water absorption of the fibers was determined in two different conditions, 65% RH/20.0 °C, and 85% RH/20.0 °C, at varying time intervals for 28 days. Three samples of each fiber type were put in the conditioning chamber and the results are reported as an average of three weighings.

ALPHA FTIR Spectrometer by Bruker was used for the FTIR characterizations. Before the measurements, the samples were dried in a forced-convection oven at 70 °C for at least 24 h. Five separate samples of each fiber type were characterized.

The morphology of the fibers was examined using SigmaHD VP field emission SEM (Carl Zeiss Microscopy Ltd., Cambridge, UK) with beam acceleration voltage between 6 kV and 10 kV and the variable chamber mode within low vacuum condition ($P < 40$ Pa). An optical microscope (Olympus SZ60) was also used to assess the morphology and dimensions of the fibers.

2.4. Fabrication of epoxy-hemp composites

Different types of the fibers were used for manufacturing the fiber mats, which were prepared using a fiber blending board for fibers and comb. A small amount (< 1 wt%) of Aerofix2 adhesive (Aerovac Systems Ltd., West Yorkshire, UK) was sprayed on the fiber mats to enable the handling of the mats. Fiber mats were then dried in a forced-convection oven at 105 °C before the composite preparation. The composites were prepared at Draw Ltd., Nilsiä, Finland, using VARTM so that each composite plate (250 mm × 250 mm × 3 mm) contained four fiber mat layers with altering orientation (0° and 90°) between each layer. The impregnation of the hemp fibers with the epoxy took approximately 4 min for each composite sample, and about 300 g of

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