# DELIVERABLES REPORT

MultiHemp

Multipurpose hemp for industrial bioproducts and biomass

(Ref n. 311849)

## WP5 – End use applications for hemp raw material

Task 5.1.2 “Mid-tech” biobased composite applications

**Deliverable 5.7 “Mid-tech” biobased composite applications**

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## Introduction

Within the consortium, we have discussed to use as many by-products as possible to follow the approach of the biorefinery concept. The idea was born to use fibres for the "Mid-Tech" composites from the losses of the hackling process ('Bulk hemp') (compare Fig. 1).

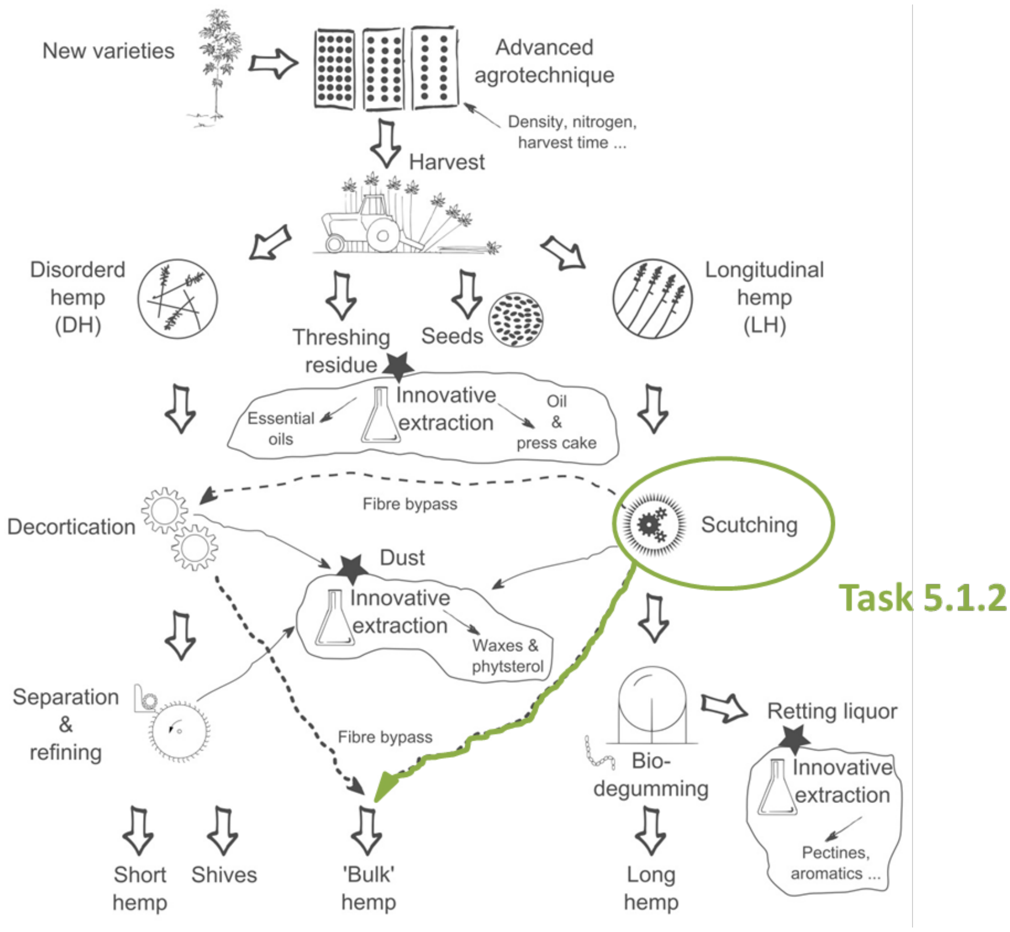


***Figure 1.*** Scheme of the MultiHemp biorefinery concept. Large arrows indicate raw material flow along the production chain; Stars highlight innovative processing to upgrade by-products into high added value end uses. The green line describes the process which was realised for the "Mid-tech" bio-based composites applications. Hackling loss was used for the compounding process to produce hemp fibre-reinforced PLA granules for the injection moulding process.

In the field of natural fibre reinforced composites (NFRC) thermoset processing techniques are available to produce high-quality composites. The same is valid for most thermoplastic processing techniques. But the conventional compounding processes are not optimised for NFRC leading to strong fibre damage during processing (Müssig, 2013). Therefore a new compounding concept for thermoplastic NFRC granules was used within the MultiHemp project: With the CMS-compounder it was possible to produce granules from long hemp fibre bundles (length > 40 mm) with fibre mass fractions of 20 % in a PLA-matrix. In Figure 2 some specific targets that have already been achieved under the development program of the continuous mixing system (CMS compounder) are shown.

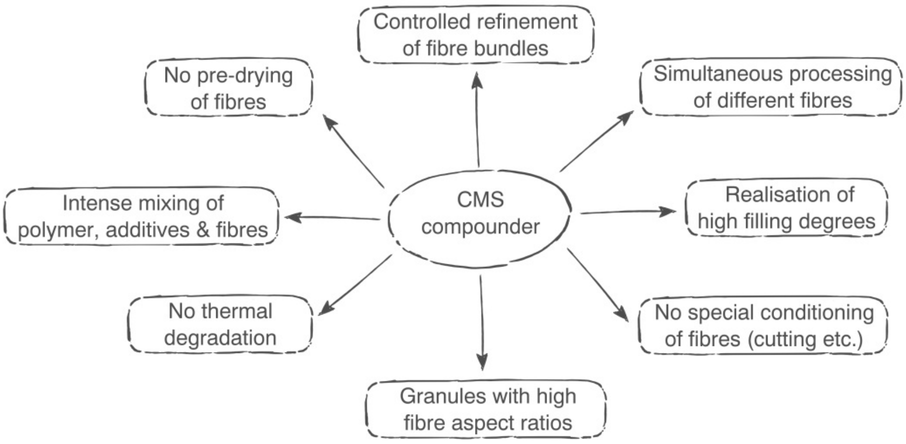


**Figure 2.** Examples of specific targets that have already been achieved under the development program of the CMS compounder (Müssig, 2013).

# Material & Methods

## *2.1. Fibres and matrix*

A NatureWorks™ (Minnetonka, USA) polylactide (PLA) 3251D injection moulding type was used for granule production.

Hemp samples from the variety trial in 2013 at Agritec (AGRITEC, Research, Breeding and Services Ltd. Czech Republic) (4 blocks: a, b, c and d) were scutched with the Agritec scutching device (AGRITEC, Research, Breeding and Services Ltd. Czech Republic). After scutching the long hemp was bio degummed at Gfibra (Gruppo Fibranova, Italy) with a bio degumming system which was internally developed by Gruppo Fibranova. After bio degumming and drying of the long hemp, the fibre bundles were manually hackled at Università Cattolica del Sacro Cuore, Piacenza, Italy. The following varieties were used in the trials:

- FNPC-251 / Ferimon
- IWNRZ-902 / Beniko
- FNPC-252 / Fedora17
- IWNRZ-901 / Bialobrzeskie
- AGM-703 / Tisza
- AGM-705 / Monoika
- FNPC-255 / Futura75
- VDS—303 / Markant
- AGM-702 / Tiborszallasi
- IWNRZ-903 / Tygra

## *2.2. Composite processing*

Samples were taken at two different harvest times (harvest 1 – full flowering and harvest 2 – seed maturity). Because the amount of the hackling loss for each block was very low, sometimes less than 100 g, the 4 blocks for each harvest time of one variety were mixed and were compounded with the CMS-compounder at 3N (3N Kompetenzzentrum Niedersachsen - Netzwerk Nachwachsende Rohstoffe & Bioökonomie e.V.), Werlte, Germany. For harvest 1 and harvest 2 all varieties were successfully compounded at process temperature between 170 and 180 °C (see table 1 and table 2).

**Table 1.** Produced compounds from hackling loss, *harvest 1*; 20 mass% hemp fibres in PLA (type 3251D).

| No. | Processing date | Sample number | Variety | Compound code | Mass of produced granules in g |
|---|---|---|---|---|---|
| 1 | 13.01. 2017 | FNPC-251 | Ferimon | CMS-HBMH-170113-1 | 1600 |
| 2 | 13.01. 2017 | IWNRZ-902 | Beniko | CMS-HBMH-170113-2 | 1200 |
| 3 | 13.01. 2017 | FNPC-252 | Fedora17 | CMS-HBMH-170113-3 | 1000 |
| 4 | 13.01. 2017 | IWNRZ-901 | Bialobrzeskie | CMS-HBMH-170113-4 | 1300 |
| 5 | 18.01. 2017 | AGM-703 | Tisza | CMS-HBMH-170118-1 | 1300 |
| 6 | 18.01. 2017 | AGM-705 | Monoika | CMS-HBMH-170118-2 | 980 |
| 7 | 18.01. 2017 | FNPC-255 | Futura75 | CMS-HBMH-170118-3 | 1030 |
| 8 | 18.01. 2017 | VDS-303 | Markant | CMS-HBMH-170118-4 | 680 |
| 9 | 18.01. 2017 | AGM-702 | Tiborszallasi | CMS-HBMH-170118-5 | 1300 |
| 10 | 18.01. 2017 | IWNRZ-903 | Tygra | CMS-HBMH-170118-6 | 1500 |

**Table 2.** Produced compounds from hackling loss, *harvest 2*; 20 mass% hemp fibres in PLA (type 3251D). The granules of the grey coloured blocks were mixed to one sample.

| No. | Processing date | Sample number | Variety | Compound code | Mass of produced granules in g |
|---|---|---|---|---|---|
| 1 | 24.10.2016 | AGM-702 | Tiborszallasi | CMS-HBMH-161024-1 | **1100** |
| 2 | 26.10.2016 | AGM-703 | Tisza | CMS-HBMH-161026-1 | **1000** |
| 3 | 26.10.2016 | FNPC-251 | Ferimon | CMS-HBMH-161026-2 | **1130** |
| 4 | 28.10.2016 | AGM-705 | Monoika | CMS-HBMH-161028-1 | **1000** |
| 5 | 28.10.2016 | IWNRZ-902 | Beniko | CMS-HBMH-161028-2 | **1100** |
| 6 | 28.10.2016 | VDS-303 | Markant | CMS-HBMH-161028-3 | **985** |
| 7 | 31.10.2016 | IWNRZ-903 | Tygra | CMS-HBMH-161031-1 | **1170** |
| 8 | 31.10.2016 | FNPC-255 | Futura75 | CMS-HBMH-161031-2 | **1080** |
| 9 | 31.10.2016 | IWNRZ-901 | Bialobrzeskie | CMS-HBMH-161031-3 | **1530** |
| 10 | 31.10.2016 | FNPC-252 | Fedora17 | CMS-HBMH-161031-4 | **1067** |
| 11 | 04.11.2016 | CRA-411 bis | CS_CRA_6 | CMS-HBMH-161104-1 | **1333** |
| 12 | 04.11.2016 | FNPC-253 | Felina32 | CMS-HBMH-161104-2 | **1054** |
| 13 | 04.11.2016 | AGM-704 | KC_Dora | CMS-HBMH-161104-3 | **900** |
| 14 | 04.11.2016 | FNPC-254 | Epsilon68 | CMS-HBMH-161104-4 | **430** |

The compounds of harvest 1 were each processed separately in the injection moulding process, harvest 2 compounds were mixed as shown in table 2. The injection moulding process was performed with the following settings: The temperatures in the screw were set to 150 °C (zone 1), 165 °C (zone 2), 175 °C (zone 3). The injection moulding machine nozzle was heated to 175 °C and the mould was cooled to 25 °C. The injection pressure was set to 1500 bar, injection speed was 24 $cm^3/s$ and the cooling time was 30 s in a standard test specimen tool (type 1 A according to DIN EN ISO 527-2).

### *2.3. Composite testing*

Prior to mechanical composite characterisation, test specimens were conditioned according to DIN EN ISO 291  for at least 18 hours at 23 °C and 50 % relative humidity.

#### *2.3.1. Density*

The density measurements were done on untested impact test specimens. The dimensions (length, width and thickness of the test specimen) were determined with an accuracy of 0.01 mm with a caliper (Mitutoyo Europe GmbH, Neuss, DE) and of the mass with an accuracy of 0.01 g with a scale (type Kern 440-35n; Kern & Sohn GmbH, Balingen-Frommern, DE). From these data the density ρ of the composite was calculated using the mass m and the volume V of each sample.

$$\rho = \frac{m}{V}$$

#### *2.3.2. Tensile properties*

5 test specimens (type 1A, DIN EN ISO 527-2) were tested with a universal testing machine type Zwick Z 020 (Zwick/Roell, Ulm, DE) working with a load cell of 20 kN and a Zwick/Roell pneumatic clamping system (clamping pressure: 1–2 bar). The gauge length was fixed to 100 mm. A preload of 50 N was used and the test was performed with a speed of 2 mm/min.

#### *2.3.3. Impact strength*

The unnotched Charpy impact strength was determined with a pendulum impact testing machine (type 5101, Zwick, Ulm, DE) operating with a pendulum hammer of 2 J according to DIN EN ISO 179. In deviation from the standard, 5 test specimens instead of the usual 10 with the dimensions of 80 × 10 × 4 $mm^3$ were investigated. The sample was hit on the flatwise impact direction.

### 2.4. Statistics

The statistical evaluation of the results was carried out using the open source R software (http://www.rproject.org/).

## Results & Discussion

### *3.1. Composite processing*

- All hemp samples - loss from the hackling process – were successfully compounded with PLA in cooperation with 3N (Werlte, Germany).

- The process was specially adapted to the hemp fibres and no thermal degradation took place during compounding (see figure 3).

- The injection moulding was adjusted to process natural fibres without thermal degradation. Injection moulding of all hemp –PLA compounds was succesfull.

- With the specially adapted injection moulding process aesthetically appealing materials of homogeneous quality could be produced.

***Figure 3.*** Hemp-reinforced PLA granules from hackling losses; samples show no thermal degradation and the yellowish colour of the hemp fibre bundles after bio-degumming is still present in the granules.

## *3.2. Composite properties*

In the following section the data of the different composite properties are illustrated as figures. For every measured property two separated figures will be shown for harvest 1 and harvest 2, an additional figure will be presented to directly compare the properties of the composites using fibres from both harvest times. In every figure the data for the neat unreinforced PLA processed with equivalent injection moulding and testing conditions is given as a reference value. At the end of each subsection the reader will find a short summary with the main findings and conclusions for each composite property.

### *3.2.1. Density / Harvest 1*

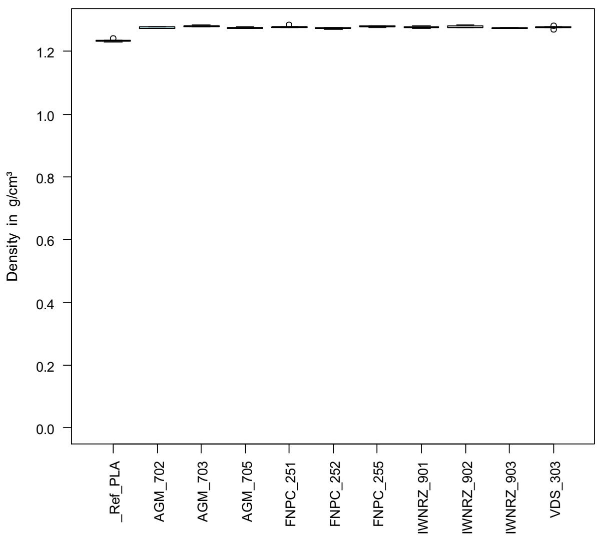


***Figure 4.*** Boxplots of the measured density of hemp/PLA composites from hackling loss from *harvest 1* compared to the PLA reference sample.

### *3.2.2. Density / Harvest 2*

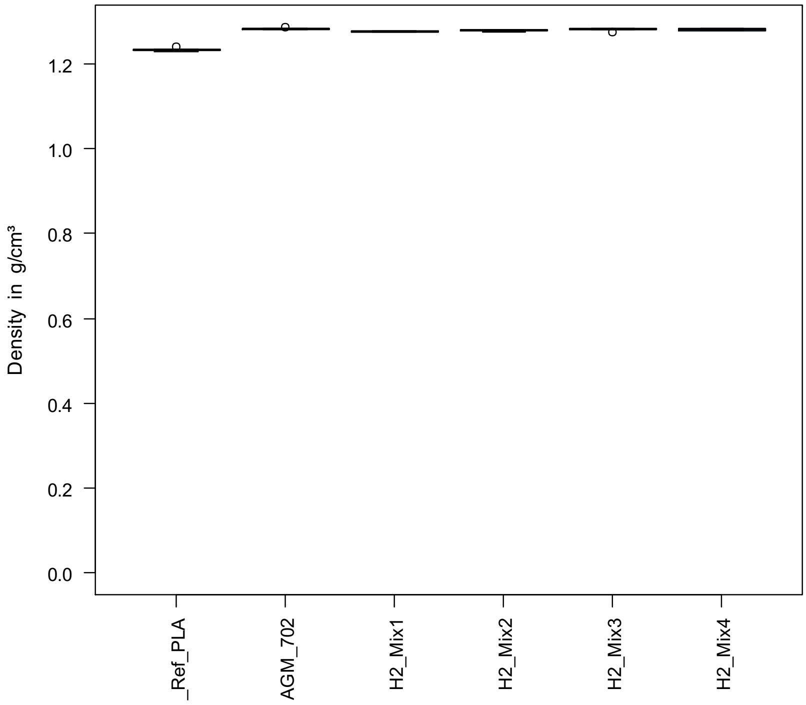


***Figure 5.*** Boxplots of the measured density of hemp/PLA composites from hackling loss from <u>*harvest 2*</u> compared to the PLA reference sample.

### *3.2.3. Density / Harvest 1 vs. Harvest 2*

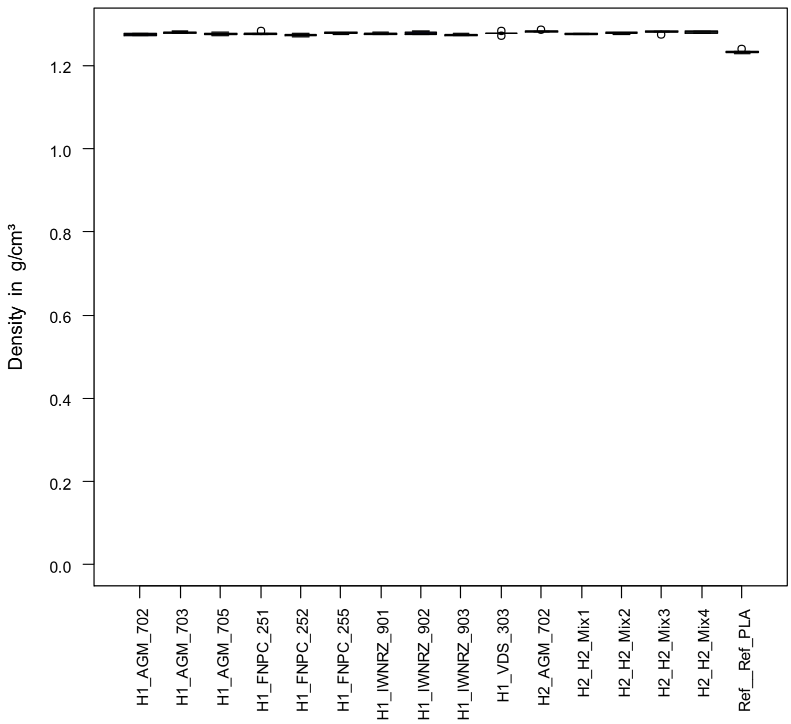

***Figure 6.*** Boxplots of the measured density of hemp/PLA composites from hackling loss – _harvest 1_ versus _harvest 2_ compared to the PLA reference sample.

**Table 3.** Density values and statistics of all tested composites; 20 mass% hemp fibres in PLA (type 3251D).

| ***Density in g/cm³*** | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Sample*** | ***n*** | ***mean*** | ***±*** | ***SD*** | ***Med*** | ***±*** | ***MAD*** | ***Min*** | ***Max*** | ***CI*** | ***W*** | ***p*** | ***Variety*** |
| *H1_AGM_702* | *5* | *1.27* | *±* | *0.00* | *1.27* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.884* | *0.328* | *Tiborszallasi* |
| *H1_AGM_703* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.28* | *0.000* | *0.876* | *0.292* | *Tisza* |
| *H1_AGM_705* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.973* | *0.892* | *Monoika* |
| *H1_FNPC_251* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.895* | *0.385* | *Ferimon* |
| *H1_FNPC_252* | *5* | *1.27* | *±* | *0.00* | *1.27* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.961* | *0.814* | *Fedora17* |
| *H1_FNPC_255* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.28* | *0.000* | *0.887* | *0.340* | *Futura75* |
| *H1_IWNRZ_901* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.985* | *0.960* | *Bialobrzeskie* |
| *H1_IWNRZ_902* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.875* | *0.289* | *Beniko* |
| *H1_IWNRZ_903* | *5* | *1.27* | *±* | *0.00* | *1.27* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.971* | *0.879* | *Tygra* |
| *H1_VDS_303* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.950* | *0.741* | *Markant* |
| *H2_AGM_702* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.29* | *0.000* | *0.810* | *0.097* | *Tiborszallasi* |
| *H2_H2_Mix1* | *5* | *1.28* | *±* | *0.00* | *1.27* | *±* | *0.00* | *1.27* | *1.28* | *0.000* | *0.866* | *0.251* | *Mix 1 (KC_Dora, CS_CRA_6, Felina32 , Epsilon68)* |
| *H2_H2_Mix2* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.28* | *0.000* | *0.881* | *0.313* | *Mix 2 (Fedora17, Futura75, Bialobrzeskie , Tygra)* |
| *H2_H2_Mix3* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.28* | *0.000* | *0.776* | *0.051* | *Mix 3 (Monoika, Beniko, Markant)* |
| *H2_H2_Mix4* | *5* | *1.28* | *±* | *0.00* | *1.28* | *±* | *0.00* | *1.28* | *1.28* | *0.000* | *0.840* | *0.164* | *Mix 4 (Tisza, Ferimon)* |
| *Ref__Ref_PLA* | *5* | *1.23* | *±* | *0.00* | *1.23* | *±* | *0.00* | *1.23* | *1.24* | *0.000* | *0.934* | *0.627* | *PLA 3251D* |

### ***Density***

- The density measurement of the pure PLA is in good agreement with data from literature, for example, 1.24 g/cm³published by Garlotta (2001).
- The compounding and injection moulding of hemp fibre-reinforced PLA increases the density of the composites compared to the PLA reference. The density measurement of the composites showed very little scattering and a very homogeneous quality. This can be interpreted as an indicator of homogeneous processing of the composites.
- No differences in the density values can be found between composites produced from hemp from harvest 1 and harvest 2.

### *3.2.4. Tensile strength / Harvest 1*

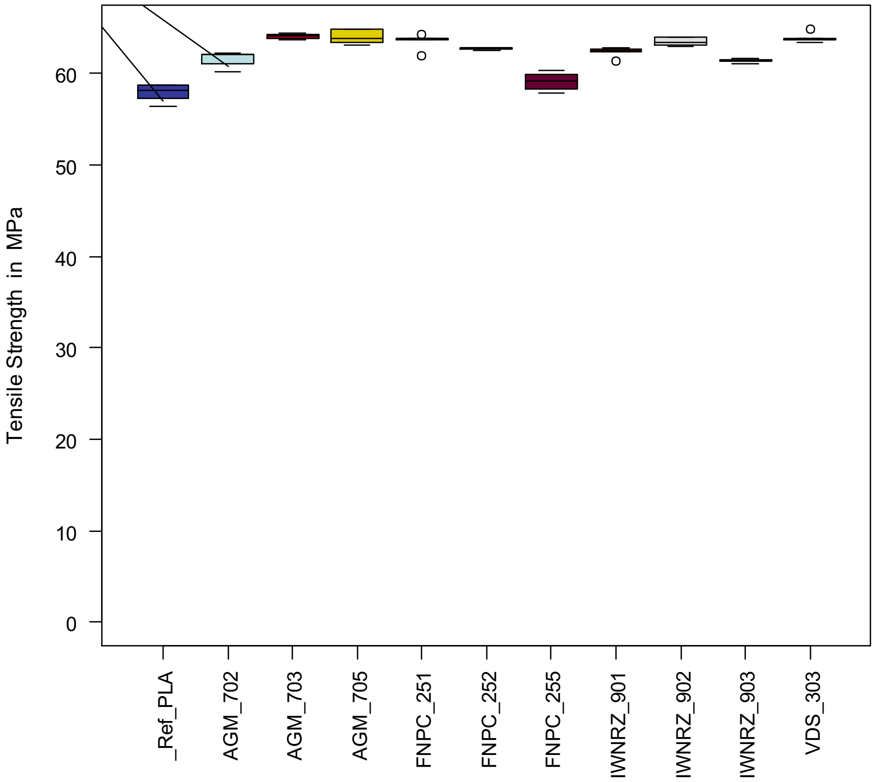


***Figure 7.*** Boxplots of the tensile strength of hemp/PLA composites from hackling loss – *harvest 1* compared to the PLA reference sample.

### *3.2.5. Tensile strength / Harvest 2*

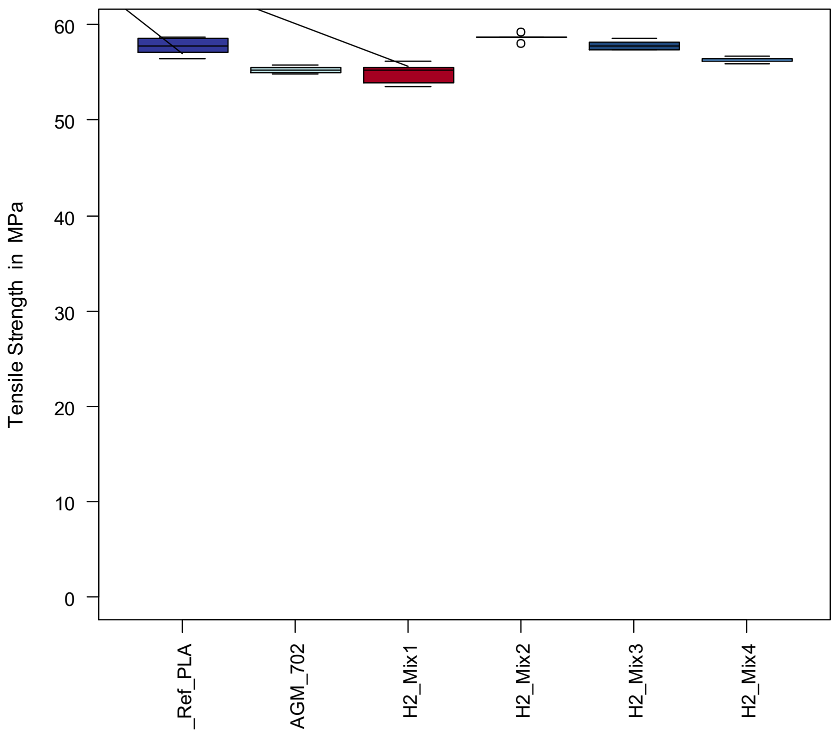


***Figure 8.*** Boxplots of the tensile strength of hemp/PLA composites from hackling loss – *harvest 2* compared to the PLA reference sample.

### 3.2.6. Tensile strength / Harvest 1 vs. Harvest 2

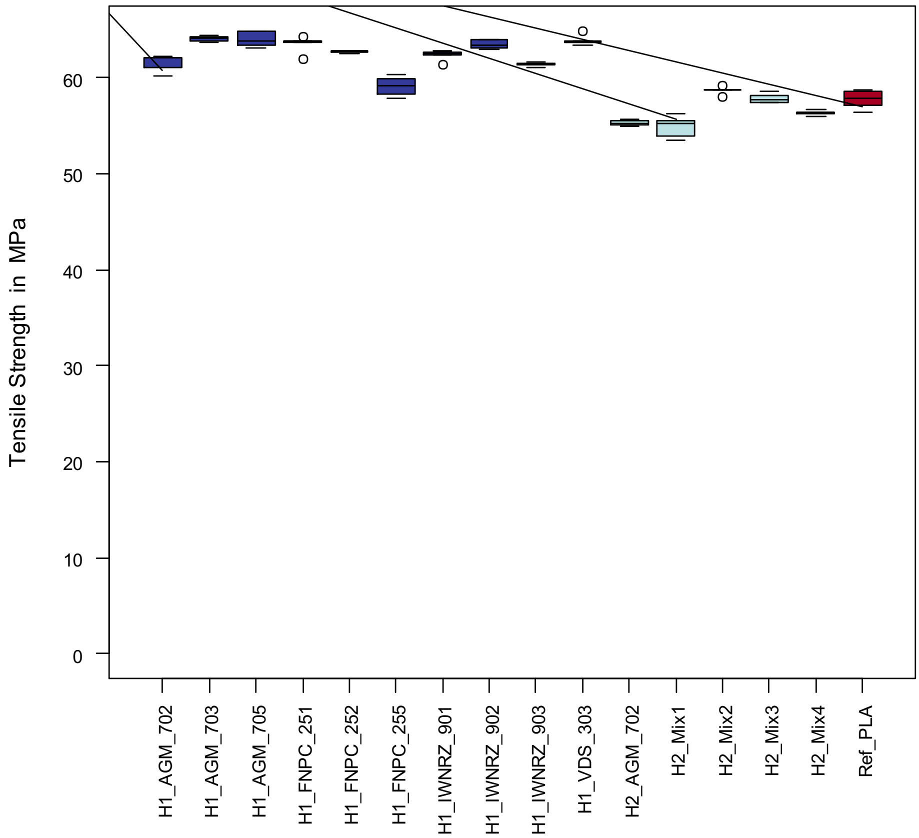


***Figure 9.*** Boxplots of the tensile strength of hemp/PLA composites from hackling loss – *harvest 1* versus *harvest 2* compared to the PLA reference sample; comparison of all samples.

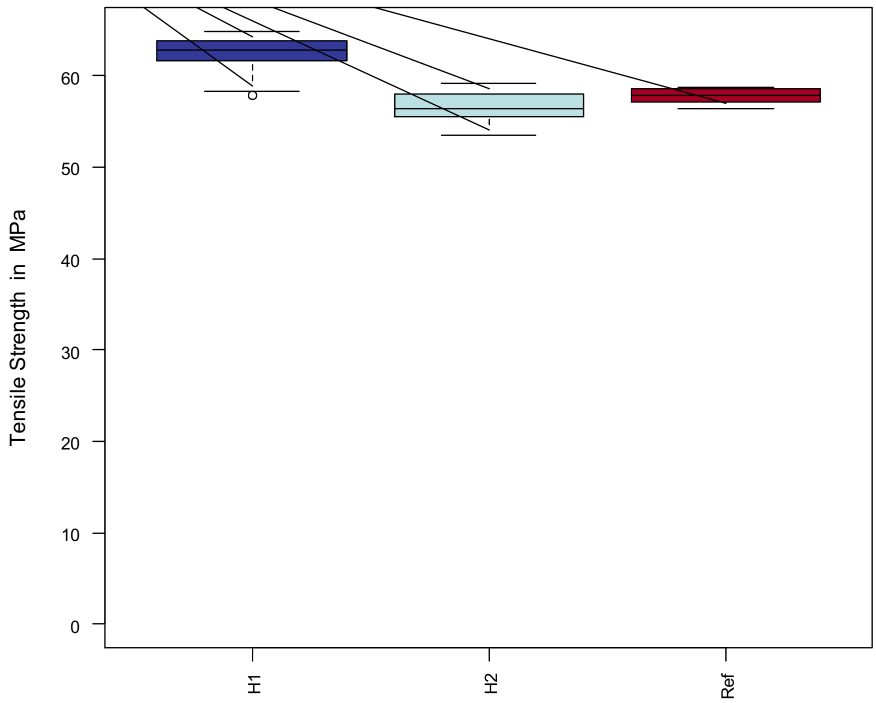


***Figure 10.*** Boxplots of the tensile strength of hemp/PLA composites from hackling loss – *harvest 1* versus *harvest 2* compared to the PLA reference sample; comparison of all measured values of harvest 1 and harvest 2, respectively, were grouped.

**Table 4.** Tensile strength values and statistics of all tested composites; 20 mass% hemp fibres in PLA (type 3251D).

| *Tensile strength in MPa* | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Sample*** | ***n*** | ***mean*** | ***±*** | ***SD*** | ***Med*** | ***±*** | ***MAD*** | ***Min*** | ***Max*** | ***CI*** | ***W*** | ***p*** | ***Variety*** |
| *H1_AGM_702* | *5* | *61.5* | *±* | *0.8* | *62.0* | *±* | *0.2* | *60.2* | *62.1* | *0.024* | *0.824* | *0.125* | *Tiborszallasi* |
| *H1_AGM_703* | *5* | *64.0* | *±* | *0.3* | *64.1* | *±* | *0.4* | *63.6* | *64.4* | *0.009* | *0.942* | *0.683* | *Tisza* |
| *H1_AGM_705* | *5* | *63.9* | *±* | *0.8* | *63.7* | *±* | *0.9* | *63.1* | *64.8* | *0.022* | *0.854* | *0.208* | *Monoika* |
| *H1_FNPC_251* | *5* | *63.5* | *±* | *0.9* | *63.7* | *±* | *0.2* | *61.9* | *64.2* | *0.026* | *0.758* | *0.035* | *Ferimon* |
| *H1_FNPC_252* | *5* | *62.7* | *±* | *0.1* | *62.8* | *±* | *0.1* | *62.5* | *62.8* | *0.004* | *0.851* | *0.198* | *Fedora17* |
| *H1_FNPC_255* | *5* | *59.1* | *±* | *1.1* | *59.2* | *±* | *1.3* | *57.8* | *60.3* | *0.030* | *0.960* | *0.809* | *Futura75* |
| *H1_IWNRZ_901* | *5* | *62.3* | *±* | *0.6* | *62.5* | *±* | *0.2* | *61.2* | *62.7* | *0.017* | *0.765* | *0.040* | *Bialobrzeskie* |
| *H1_IWNRZ_902* | *5* | *63.4* | *±* | *0.5* | *63.4* | *±* | *0.7* | *62.9* | *63.9* | *0.013* | *0.900* | *0.410* | *Beniko* |
| *H1_IWNRZ_903* | *5* | *61.3* | *±* | *0.2* | *61.3* | *±* | *0.2* | *61.0* | *61.5* | *0.006* | *0.898* | *0.401* | *Tygra* |
| *H1_VDS_303* | *5* | *63.8* | *±* | *0.5* | *63.7* | *±* | *0.2* | *63.4* | *64.8* | *0.015* | *0.797* | *0.077* | *Markant* |
| *H2_AGM_702* | *5* | *55.3* | *±* | *0.4* | *55.2* | *±* | *0.5* | *54.8* | *55.7* | *0.010* | *0.939* | *0.661* | *Tiborszallasi* |
| *H2_Mix1* | *5* | *54.9* | *±* | *1.1* | *55.2* | *±* | *1.5* | *53.5* | *56.2* | *0.031* | *0.941* | *0.672* | *Mix 1 (KC_Dora. CS_CRA_6. Felina32 . Epsilon68)* |
| *H2_Mix2* | *5* | *58.6* | *±* | *0.4* | *58.7* | *±* | *0.1* | *58.0* | *59.2* | *0.012* | *0.919* | *0.523* | *Mix 2 (Fedora17. Futura75. Bialobrzeskie . Tygra)* |
| *H2_Mix3* | *5* | *57.8* | *±* | *0.5* | *57.7* | *±* | *0.6* | *57.3* | *58.5* | *0.014* | *0.917* | *0.511* | *Mix 3 (Monoika. Beniko. Markant)* |
| *H2_Mix4* | *5* | *56.3* | *±* | *0.3* | *56.4* | *±* | *0.3* | *55.9* | *56.7* | *0.008* | *0.973* | *0.893* | *Mix 4 (Tisza. Ferimon)* |
| *Ref_PLA* | *6* | *57.7* | *±* | *0.9* | *57.7* | *±* | *1.1* | *56.4* | *58.7* | *0.024* | *0.937* | *0.638* | *PLA 3251D* |

### *Tensile strength*

- By adding 20 mass% of hackling loss of hemp fibre bundles from harvest 2 compounds are on the strength level of the unreinforced PLA injection moulded samples. A comparison between all harvest 2 samples and PLA shows no significant differences. It is worth to mention here that it is often reported in literature that a reinforcing effect is achieved only if more than 20 % of fibre mass is added to the PLA polymer.

- The use of hemp fibre bundles from harvest 1 shows significantly higher strength values compared to the pure PLA polymer. Hemp fibre bundles from harvest 1 show a better reinforcing potential than hemp fibre bundles from harvest 2.

- In general the produced compounds show very homogenous properties and, compared to the literature (see table 5), very promising properties for hemp as a by-product (loss) from the hackling process.

**Table 5:** Mechanical characteristics of composites produced from hemp/PLA via CMS compounding and injection moulding compared to results of injection moulded flax/PLA (results of neat PLA-matrix are given in brackets). Data based on a literature overview (Müssig. 2013).

| *Composite* | *Fibre mass fraction in %* | *Tensile strength in MPa* | *Young´s modulus in GPa* | *Unnotched Charpy impact strength in kJ/m²* | *Reference* |
|---|---|---|---|---|---|
| *Hemp KGE-02/PLA* | *20* | *56 (49)* | *5.4 (3.1)* | *9.4 (10.8)* | *Müssig and Graupner (2013)* |
| *Hemp Age/PLA* | *20* | *64 (49)* | *5.3 (3.1)* | *11.1 (10.8)* | *Müssig and Graupner (2013)* |
| *Flax/PLA* | *20* | *56 (60)* | *6.4 (3.6)* | | *Le Duigou et al. (2008)* |
| *Flax/PLA* | *20* | *49 (44)* | *5.1 (3.1)* | *10.5 (16)* | *Bax and Müssig (2008)* |
| *Hemp KGE-02/PLA* | *30* | *61 (49)* | *6.2 (3.1)* | *9.4 (10.8)* | *Müssig and Graupner (2013)* |
| *Hemp Age/PLA* | *30* | *70 (49)* | *6.6 (3.1)* | *11.6 (10.8)* | *Müssig and Graupner (2013)* |
| *Flax/PLA* | *30* | *53 (60)* | *7.3 (3.6)* | | *Le Duigou et al. (2008)* |
| *Flax/PLA* | *30* | *54 (44)* | *6.3 (3.1)* | *11.0 (16.0)* | *Bax and Müssig (2008)* |

## *3.2.7. Young's modulus / Harvest 1*

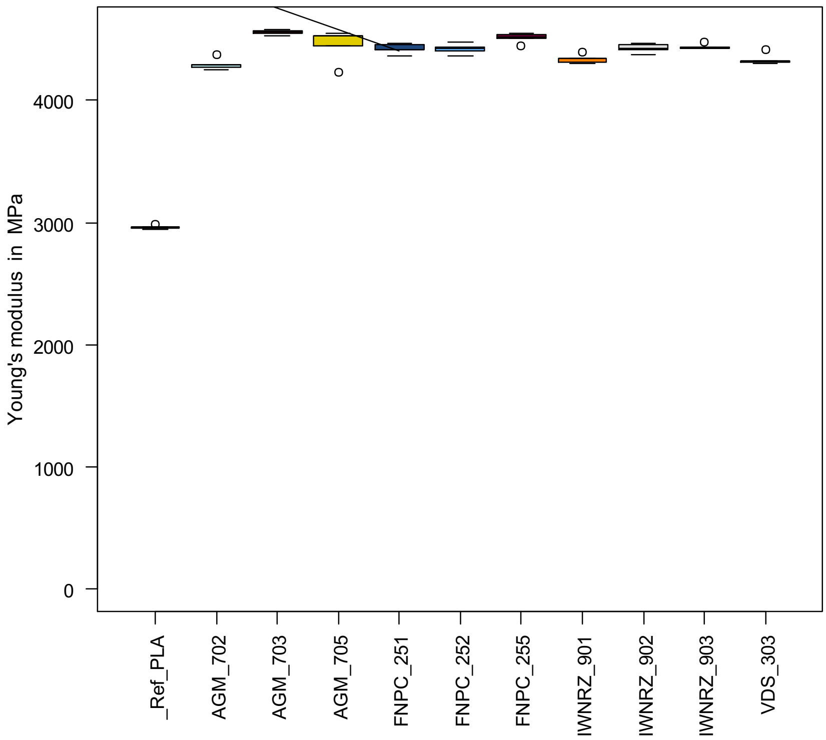


***Figure 11.*** Boxplots of the Young's modulus of hemp/PLA composites from hackling loss – <u>*harvest 1*</u> compared to the PLA reference sample; Young's modulus obtained by the crosshead-displacement of the universal testing machine

### *3.2.8. Young’s modulus / Harvest 2*

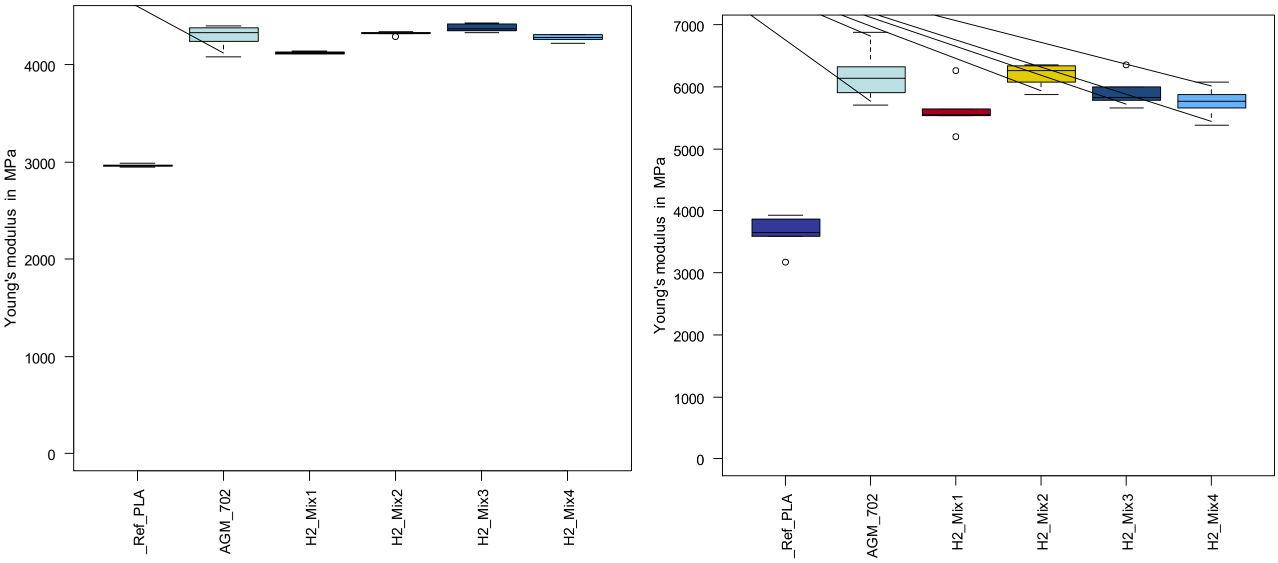


***Figure 12. Left.*** Boxplots of the Young’s modulus (obtained by the crosshead-displacement of the universal testing machine) of hemp/PLA composites from hackling loss – *harvest 2* compared to the PLA reference sample. ***Right.*** Boxplots of the Young’s modulus (based on extensiometer measurement) of hemp/PLA composites from hackling loss – *harvest 2* compared to the PLA reference sample.

### *3.2.9. Young’s modulus / Harvest 1 vs. Harvest 2*

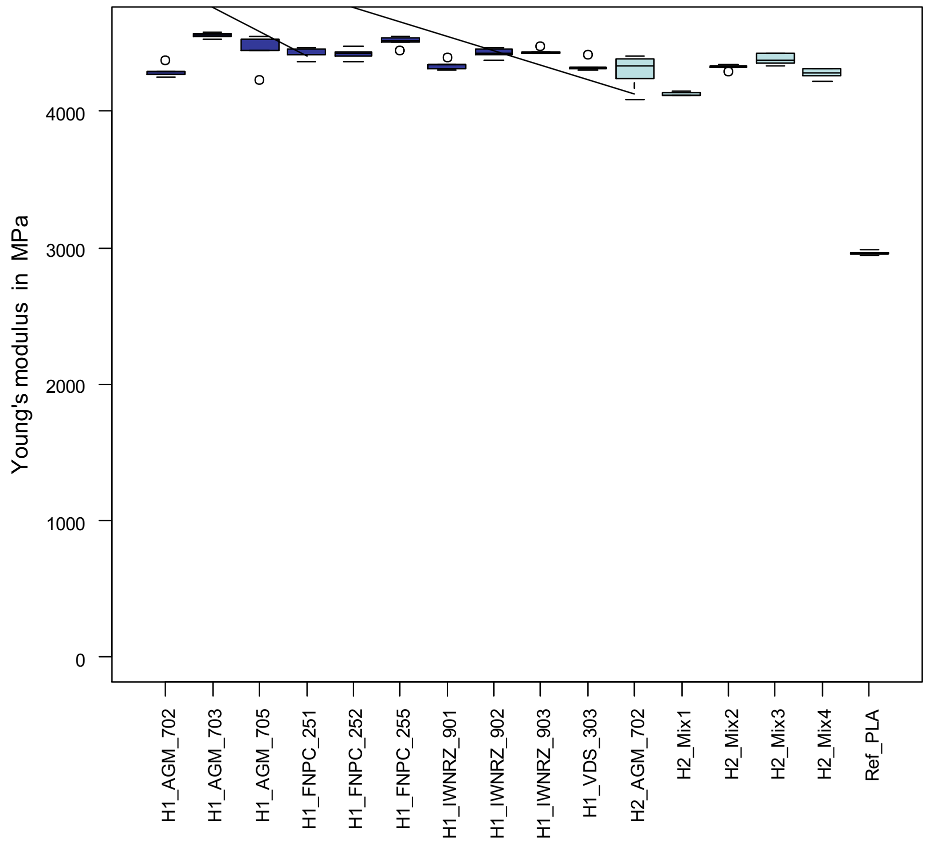


***Figure 13. Left:*** Boxplots of the Young’s modulus of hemp/PLA composites from hackling loss – *harvest 1* versus *harvest 2* compared to the PLA reference sample; comparison of all samples; Young’s modulus obtained by the crosshead-displacement of the universal testing machine.

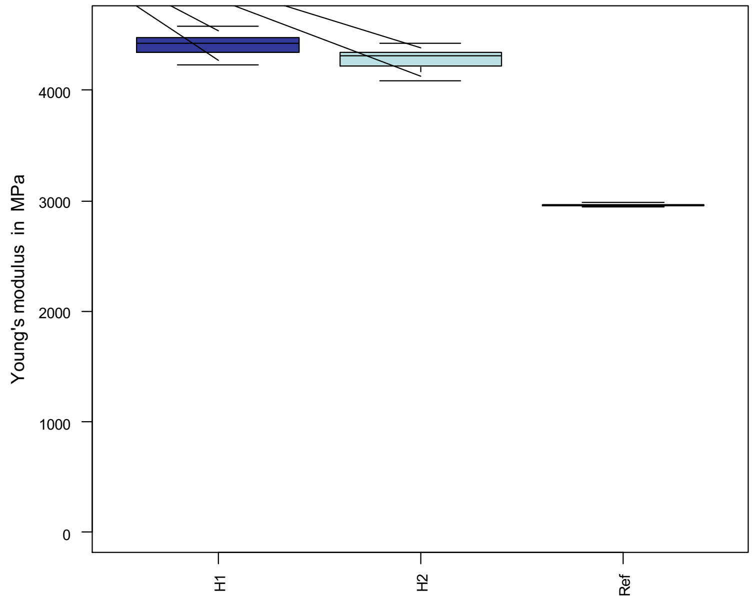


***Figure 14.*** Boxplots of the Young's modulus of hemp/PLA composites from hackling loss – *harvest 1* versus *harvest 2* compared to the PLA reference sample; comparison of all harvest 1 samples with all harvest 2 samples; Young's modulus obtained by the crosshead-displacement of the universal testing machine.

**Table 6.** Young's modulus values and statistics of all tested composites; 20 mass% hemp fibres in PLA (type 3251D). Young's modulus obtained by the crosshead-displacement of the universal testing machine.

| *Young's modulus in MPa* | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Sample*** | ***n*** | ***mean*** | ***±*** | ***SD*** | ***Med*** | ***±*** | ***MAD*** | ***Min*** | ***Max*** | ***CI*** | ***W*** | ***p*** | ***Variety*** |
| *H1_AGM_702* | *5* | *4295.0* | *±* | *48.3* | *4288.4* | *±* | *30.7* | *4248.5* | *4375.0* | *1.36* | *0.881* | *0.315* | *Tiborszallasi* |
| *H1_AGM_703* | *5* | *4554.7* | *±* | *18.8* | *4557.4* | *±* | *18.5* | *4528.4* | *4577.0* | *0.53* | *0.985* | *0.961* | *Tisza* |
| *H1_AGM_705* | *5* | *4456.6* | *±* | *130.4* | *4527.0* | *±* | *24.1* | *4233.0* | *4543.2* | *3.66* | *0.751* | *0.030* | *Monoika* |
| *H1_FNPC_251* | *5* | *4424.3* | *±* | *40.8* | *4415.6* | *±* | *65.7* | *4365.6* | *4466.1* | *1.14* | *0.913* | *0.483* | *Ferimon* |
| *H1_FNPC_252* | *5* | *4419.5* | *±* | *41.7* | *4419.0* | *±* | *23.3* | *4362.7* | *4476.7* | *1.17* | *0.992* | *0.987* | *Fedora17* |
| *H1_FNPC_255* | *5* | *4509.9* | *±* | *40.0* | *4518.1* | *±* | *26.6* | *4442.7* | *4543.7* | *1.12* | *0.846* | *0.183* | *Futura75* |
| *H1_IWNRZ_901* | *5* | *4339.2* | *±* | *36.7* | *4337.3* | *±* | *34.2* | *4303.7* | *4398.2* | *1.03* | *0.903* | *0.426* | *Bialobrzeskie* |
| *H1_IWNRZ_902* | *5* | *4426.5* | *±* | *34.4* | *4425.5* | *±* | *39.8* | *4375.8* | *4463.6* | *0.97* | *0.955* | *0.774* | *Beniko* |
| *H1_IWNRZ_903* | *5* | *4436.2* | *±* | *22.8* | *4424.5* | *±* | *5.2* | *4421.0* | *4475.2* | *0.64* | *0.758* | *0.035* | *Tygra* |
| *H1_VDS_303* | *5* | *4334.5* | *±* | *45.0* | *4323.3* | *±* | *17.7* | *4301.0* | *4413.2* | *1.26* | *0.742* | *0.025* | *Markant* |
| *H2_AGM_702* | *5* | *4285.5* | *±* | *129.8* | *4328.0* | *±* | *106.7* | *4081.5* | *4400.0* | *3.64* | *0.894* | *0.375* | *Tiborszallasi* |
| *H2_Mix1* | *5* | *4123.8* | *±* | *12.6* | *4118.5* | *±* | *10.7* | *4111.3* | *4141.8* | *0.35* | *0.915* | *0.501* | *Mix 1 (KC_Dora. CS_CRA_6. Felina32 . Epsilon68)* |
| *H2_Mix2* | *5* | *4325.9* | *±* | *18.2* | *4333.3* | *±* | *11.9* | *4294.9* | *4341.4* | *0.51* | *0.825* | *0.128* | *Mix 2 (Fedora17. Futura75. Bialobrzeskie . Tygra)* |
| *H2_Mix3* | *5* | *4379.0* | *±* | *43.9* | *4368.1* | *±* | *56.3* | *4330.1* | *4428.6* | *1.23* | *0.898* | *0.399* | *Mix 3 (Monoika. Beniko. Markant)* |
| *H2_Mix4* | *5* | *4274.9* | *±* | *37.6* | *4278.9* | *±* | *41.9* | *4221.0* | *4311.3* | *1.05* | *0.928* | *0.583* | *Mix 4 (Tisza. Ferimon)* |
| *Ref_PLA* | *6* | *2961.6* | *±* | *13.4* | *2958.5* | *±* | *10.0* | *2944.9* | *2984.2* | *0.34* | *0.948* | *0.727* | *PLA 3251D* |

***Young's modulus***

- By adding 20 mass% of hackling loss of hemp fibre bundles from harvest 1 and 2 to the PLA, the stiffness of the resulting composites is more than 1000 MPa higher compared to the unreinforced injection moulded PLA samples. This corresponds to a stiffness increase of > 20 %.
- The use of hemp fibre bundles from harvest 1 significantly increases the Young's modulus values compared to the pure PLA polymer. The reinforcing potential is slightly better than hemp fibre bundles from harvest 2.
- Young's modulus values obtained by the crosshead-displacement of the universal testing machine are approximately 2000 MPa lower compared to Young's modulus data based on extensiometer measurement (compare figure 12). Bearing this in mind all hemp/PLA composites from harvest 1 and harvest 2 show excellent stiffness values compared to already published data from other authors (compare table 5).

### *3.2.10. Charpy impact strength / Harvest 1*

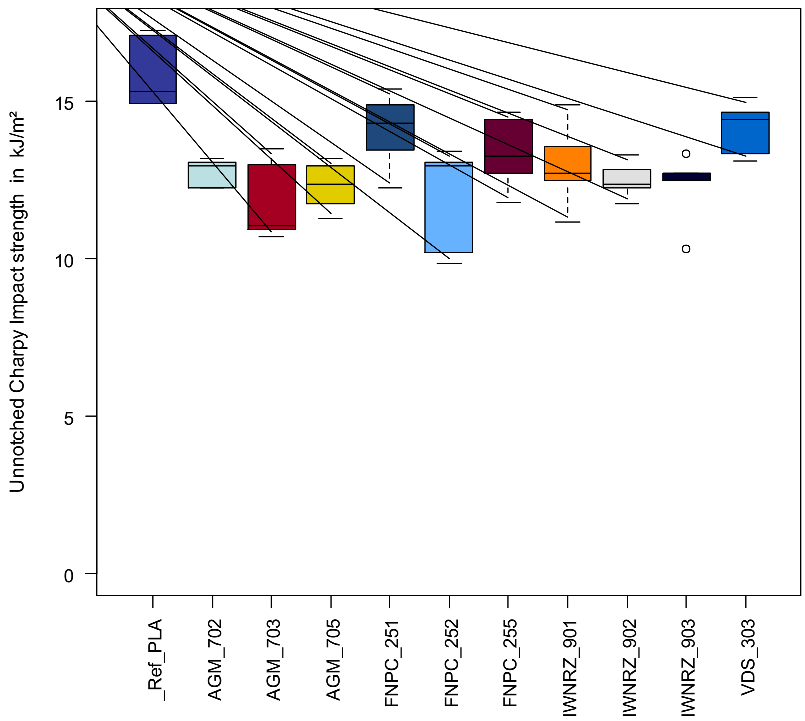


***Figure 15.*** Boxplots of the Charpy impact strength of hemp/PLA composites from hackling loss – *harvest 1* compared to the PLA reference sample.

### 3.2.11. Charpy impact strength / Harvest 2

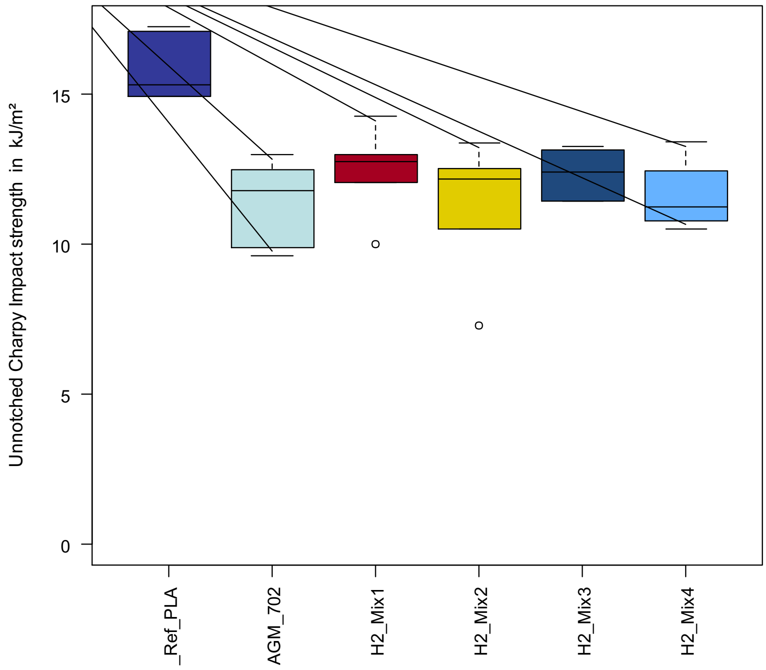


***Figure 16.*** Boxplots of the Charpy impact strength of hemp/PLA composites from hackling loss – _harvest 2_ compared to the PLA reference sample.

### 3.2.12. Charpy impact strength / Harvest 1 vs. Harvest 2

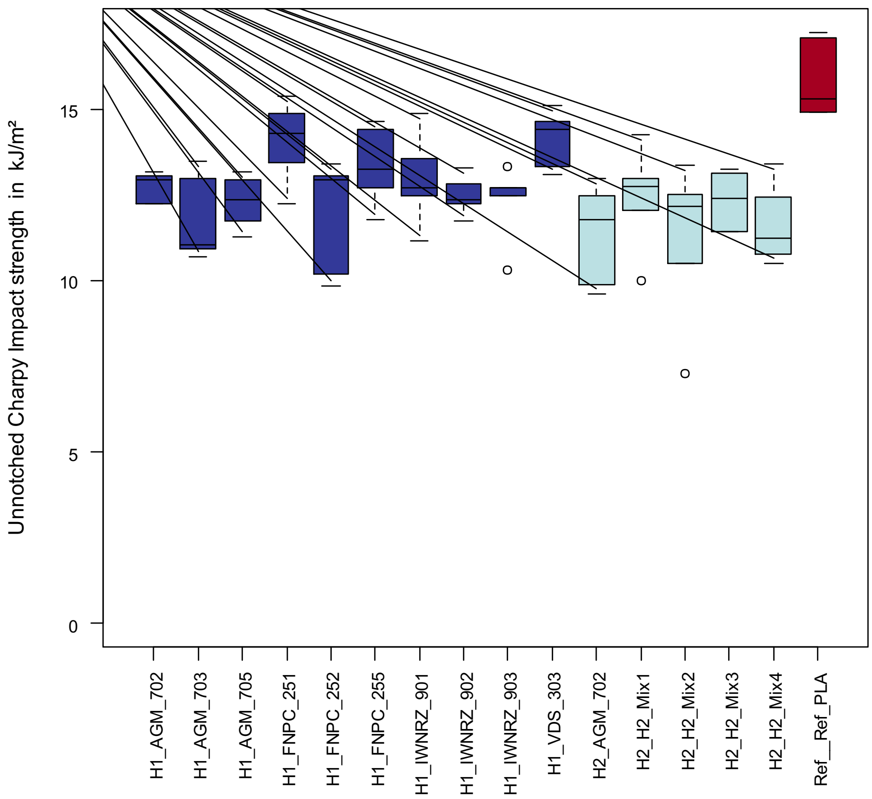


***Figure 17.*** Boxplots of the Charpy impact strength of hemp/PLA composites from hackling loss – _harvest 1_ versus _harvest 2_ compared to the PLA reference sample; comparison of all samples.

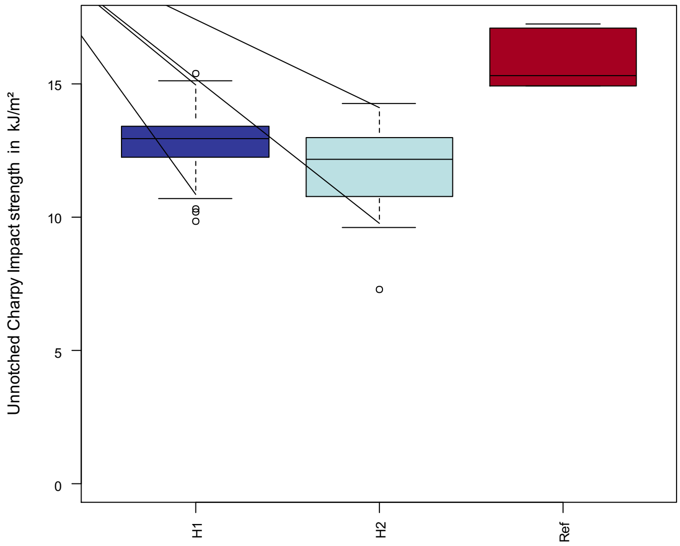


***Figure 18.*** Boxplots of the Charpy impact strength of hemp/PLA composites from hackling loss – harvest 1 versus harvest 2 compared to the PLA reference sample; comparison of all harvest 1 samples with all harvest 2 samples.

**Table 7.** Charpy impact strength values and statistics of all tested composites; 20 mass% hemp fibres in PLA (type 3251D).

| *Unnotched Charpy Impact strength in kJ/m²* | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Sample*** | ***n*** | ***mean*** | ***±*** | ***SD*** | ***Med*** | ***±*** | ***MAD*** | ***Min*** | ***Max*** | ***CI*** | ***W*** | ***p*** | ***Variety*** |
| *H1_AGM_702* | *5* | *12.7* | *±* | *0.5* | *13.0* | *±* | *0.3* | *12.2* | *13.2* | *0.01* | *0.808* | *0.095* | *Tiborszallasi* |
| *H1_AGM_703* | *5* | *11.8* | *±* | *1.3* | *11.1* | *±* | *0.5* | *10.7* | *13.5* | *0.04* | *0.818* | *0.112* | *Tisza* |
| *H1_AGM_705* | *5* | *12.3* | *±* | *0.8* | *12.3* | *±* | *0.9* | *11.3* | *13.2* | *0.02* | *0.949* | *0.728* | *Monoika* |
| *H1_FNPC_251* | *5* | *14.0* | *±* | *1.2* | *14.3* | *±* | *1.3* | *12.3* | *15.4* | *0.03* | *0.963* | *0.826* | *Ferimon* |
| *H1_FNPC_252* | *5* | *11.9* | *±* | *1.7* | *13.0* | *±* | *0.7* | *9.8* | *13.4* | *0.05* | *0.790* | *0.068* | *Fedora17* |
| *H1_FNPC_255* | *5* | *13.3* | *±* | *1.2* | *13.2* | *±* | *1.7* | *11.8* | *14.6* | *0.03* | *0.939* | *0.655* | *Futura75* |
| *H1_IWNRZ_901* | *5* | *13.0* | *±* | *1.4* | *12.7* | *±* | *1.2* | *11.2* | *14.9* | *0.04* | *0.986* | *0.964* | *Bialobrzeskie* |
| *H1_IWNRZ_902* | *5* | *12.5* | *±* | *0.6* | *12.4* | *±* | *0.7* | *11.7* | *13.3* | *0.02* | *0.983* | *0.951* | *Beniko* |
| *H1_IWNRZ_903* | *5* | *12.3* | *±* | *1.2* | *12.7* | *±* | *0.3* | *10.3* | *13.3* | *0.03* | *0.793* | *0.071* | *Tygra* |
| *H1_VDS_303* | *5* | *14.1* | *±* | *0.9* | *14.4* | *±* | *1.0* | *13.1* | *15.1* | *0.02* | *0.906* | *0.446* | *Markant* |
| *H2_AGM_702* | *5* | *11.3* | *±* | *1.5* | *11.8* | *±* | *1.8* | *9.6* | *13.0* | *0.04* | *0.883* | *0.322* | *Tiborszallasi* |
| *H2_H2_Mix1* | *5* | *12.4* | *±* | *1.6* | *12.8* | *±* | *1.1* | *10.0* | *14.3* | *0.04* | *0.954* | *0.763* | *Mix 1 (KC_Dora, CS_CRA_6, Felina32 , Epsilon68)* |
| *H2_H2_Mix2* | *5* | *11.2* | *±* | *2.4* | *12.1* | *±* | *1.8* | *7.3* | *13.3* | *0.07* | *0.886* | *0.336* | *Mix 2 (Fedora17, Futura75, Bialobrzeskie , Tygra)* |
| *H2_H2_Mix3* | *5* | *12.3* | *±* | *0.9* | *12.4* | *±* | *1.2* | *11.4* | *13.2* | *0.02* | *0.845* | *0.178* | *Mix 3 (Monoika, Beniko, Markant)* |
| *H2_H2_Mix4* | *5* | *11.7* | *±* | *1.2* | *11.2* | *±* | *1.0* | *10.5* | *13.4* | *0.03* | *0.907* | *0.449* | *Mix 4 (Tisza, Ferimon)* |
| *Ref__Ref_PLA* | *5* | *15.9* | *±* | *1.2* | *15.3* | *±* | *0.5* | *14.9* | *17.2* | *0.03* | *0.776* | *0.051* | *PLA 3251D* |

***Charpy impact strength***

- In general, adding stiff bast fibres to a polymer like PLA decreases the unnotched Charpy impact strength compared to the unreinforced polymer (compare literature data in table 5). This is the case for the data shown with 20 mass-% reinforced PLA, too.
- By adding 20 mass% of hackling loss of hemp fibre bundles from harvest 1 and 2 to the PLA the impact value drop from a mean value of 16 kJ/m² for the unreinforced injection moulded PLA sample to values between 11 kJ/m² and 14 kJ/m² for hemp/PLA.
- Even though the differences are not significant, there is a tendency towards higher Charpy impact strength values with hemp from harvest 1 than with hemp from harvest 2.

# Conclusions

- Within the MultiHemp project a production concept for losses of the hackling process could be built up. The use of this hackling loss for “Mid-Tech” composites represents an interesting and promising solution as part of the bio-refinery concept of MultiHemp.
- All hemp samples were successfully compounded with PLA and afterwards injection moulded. With the specially adapted compounding and injection moulding process aesthetically appealing materials of homogeneous quality could be produced.
- The use of only 20 mass% hemp fibre bundles from harvest 1 already significantly increased the strength values compared to the pure PLA polymer and shows a better reinforcing potential than hemp fibre bundles from harvest 2.
- The use of hemp fibre bundles from harvest 1 significantly increases the Young’s modulus values compared to the pure PLA polymer and shows a slightly better reinforcing potential than hemp fibre bundles from harvest 2.
- In general, adding stiff bast fibres to a polymer like PLA leads to a lower unnotched Charpy impact strength compared to the unreinforced polymer. The impact of the hemp/PLA samples is still on an acceptable level.
- On the basis of the existing data there are some varieties behaving better than others. For example, FNPC 251 and VDS 303 show both in strength and impact the highest values for harvest 1 samples.
- We see a significant influence of harvest time which is stronger than the variety influence on the composite properties.

## Acknowledgement

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