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Analysis of plant materials pre-treated by steam explosion technology for their usability as insulating materials

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Abstract Raw materials of annual plants fibres are not easily usable for industrial production applications. Pre-treatment of the annual plant fibres is necessary to increase the homogeneity of the material and to improve the material properties. This study deals with the influence of steam explosion treatment on the quality of annual plant fibres used as insulating material.
Maize and wheat straw were selected for production of insulating panels. To clarify the changes within the structure of the plants due to the pre-treatment process material analysis was carried out using FT-IR spectroscopy and raster electron microscope. Furthermore, the bulk density and the thermal conductivity were analysed as important values for insulating materials.
The results showed that the pre-treatment process homogenizes the materials and the processes could be used for the production of bulk insulation.

Key words: thermal-hydro treatment, thermal conductivity, maize straw, miscanthus.

INTRODUCTION

Crops material used as insulating material has a long tradition. Buildings with straw bales are energy efficient, durable and attractive (Ashour et al., 2001). Beside these advantages also considerable drawbacks like the thickness of the building wall and the anisotropic material properties exist for many applications. Pre-treatments of these plant fibres have potential to increase their homogeneity and to improve the material properties (Nagl et al., 2015a, b). In this study, the effect of steam explosion treatment of different straw material for insulating material was examined.

The steam explosion treatment modifies the chemical composition, the sorption behaviour and the mechanical properties of wheat straw fibres (Han et al., 2009). A degradation of lignin and hemicellulose (polyose) was determined. The structural changes due to the steam explosion treatment were analysed by Han et al. (2010). They concluded that higher steam temperatures and longer retention times results in more homogeneous fibre materials. Moreover, the opportunity of glue less fibreboards made from steam exploded *Miscanthus sinensis* was analysed by Velásquez et al. (2002). It could be shown that the mechanical properties have increased due to the treatment process.

However, the insulation properties of steam pressurized straw materials have not been analysed in detail. This study deals with possible applications of treated plant materials for the application of bulk insulating material.

MATERIALS AND METHODS

Straw Materials

Straw of maize (*Zea mays* L.) and miscanthus (*Miscanthus sinensis Giganteus*) was used for the analysis of insulating properties. The air-dried materials were shredded by using a customary shredder system for the further process.

Steam Explosion Process

The straw was treated under steam explosion conditions using a pilot plant of the Upper Austria University of Applied Sciences. The pilot plant was detailed described by Eisenhuber et al. (2013). Steam temperature was 200 °C and the retention time was 20 minutes. Each batch of about 900 g of straw was put inside the steam chamber. The treated straw was dried to a moisture content between 6 and 12%.

Determination of the particle size distribution

The particle size distribution was analysed according to the DIN CEN/TS 15149-2 (2010). The vibratory sieve shaker AS 200 digit from Retsch was applied. For all materials the time for vibration was 15 min with an amplitude of 50. The weight for each sieve was used to calculate the percentage of the different particle sizes.

Determination of the bulk density

The bulk density of the various materials was determined according to the ÖNORM EN 15103 (2009).

Analysis of thermal conductivity

The thermal conductivity was measured according to EN 12667 (2001) using the lambda-meter EP500 of the Lambda Measurement Technologies Corporation. All various material was stored at 20 °C temperature and 65% relative humidity. The bulk densities in the measuring field of 25 x 25 x 2 cm^3 for the measurement were varying depending on the material properties and due to the requirements of the standard used (e.g. completely material filling of the test area). The thermal conductivity measurements were taken at three temperatures, at 10°, 25° and 40 °C, respectively.

FT-IR Spectroscopy

For the FT-IR measurements each raw material was milled with a cutting mill (Retsch) to pass a mesh of 500 µm and the fractions between 250 and 63 µm were separated with a sieving apparatus (Retsch). The spectra of the milled materials were recorded between 4,000 and 600 cm^{-1} with 32 scans at a resolution of 4 cm^{-1} using a Frontier FT-IR spectrometer (PerkinElmer) equipped with a Miracle diamond ATR accessory with a 1.8 mm round crystal surface. The average of three spectra was baseline-corrected and used for the further analysis.

RESULTS AND DISCUSSION

Chemical and structural changes in materials

The chemical changes of the different materials due to the stream explosion process were analysed by using FT-IR spectroscopy (Fig. 1).

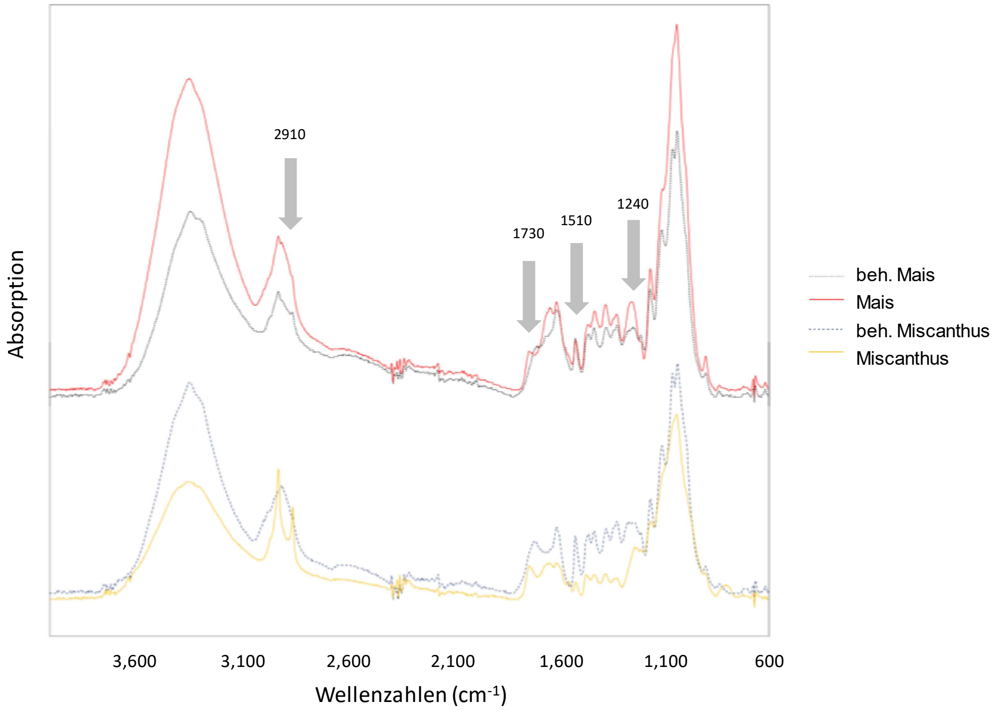

Figure 1. FT-IR spectra of treated and untreated maize as well as miscanthus, respectively.

The chemical changes of the plant components due to the steam explosion occurred mainly in holocelluloses and in lignin (Han et al., 2010a, b). This was also the first impression of the chemical changes caused by steamed pressurized treatments. A difference between the IR spectra of treated and untreated miscanthus and maize samples were observed at the wavenumbers in the area about 2910 cm^{-1}. The peaks obtained at 2,930 cm^{-1} and 2,890 cm^{-1} are an indication of stretching vibration form CH, CH_2 and CH_3 (Pretsch et al., 2010). These functional groups are corresponding with the wax on the raw materials. Miscanthus samples showed a clear form of the two bands. After the steam pressurised treatments, the heights of the flanks were decreased and clear form of the peaks cannot be observed anymore. The peak at around 1,730 cm^{-1} is an initiation of carbonyl groups or COOH-groups, which may correspond with the hemicellulose and fatty acids of the natural wax. At around 1,510 cm^{-1} a peak can be observed, which are corresponding to the aromatic molecules (Pretsch et al., 2010). This peak can be increased due to the treatment based on the exploration process of the miscanthus materials. The band around 1,240 cm^{-1} was confirmed to the stretching vibration of C = O and COOH groups of aromatic compounds and hemicellulose. Based on these results, it can be assumed that the hemicellulose was degraded due to the steam explosion process. Besides the chemical modification the changes in structure are important for the application for the use as insulating materials.

Fig. 2 shows the raw material of maize before and after the steam explosion process at the macro and micro levels. A high portion of leaves can be determined in Fig. 2, a, whereas the material from the stem is negligible. It can be observed that this process frayed out the cell complex of the lignocellulosic materials. However, the after the drying process some conglomerates of the material could be seen for maize and miscanthus, respectively.

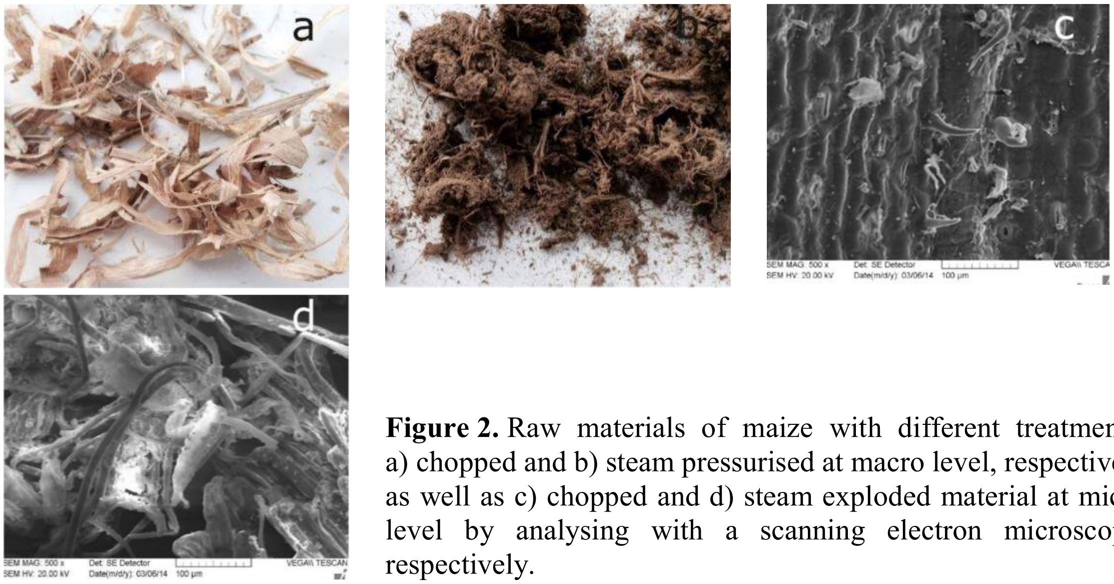

Figure 2. Raw materials of maize with different treatments: a) chopped and b) steam pressurised at macro level, respectively as well as c) chopped and d) steam exploded material at micro level by analysing with a scanning electron microscope, respectively.

The steam explosion influences on the material on fibre and fibrils levels were shown in Fig. 3 for miscanthus materials. Some intact parts of the miscanthus stem can be observed in Fig. 3, b. The retention time of the steam explosion process was too short for defibrating these big particles. Nevertheless, the wax surface of the various straw species was also destroyed due to the steam explosion process, which is in line with the results from the FT-IR spectroscopy. The analysis at the macro level depicted that the particle size distributions between the natural and untreated materials are different.

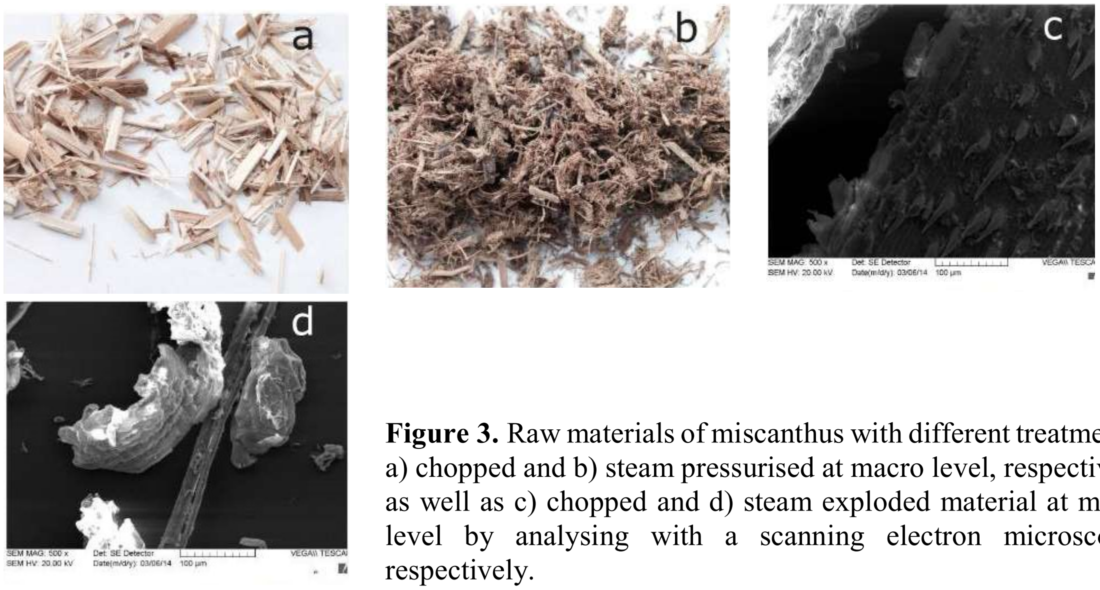

Figure 3. Raw materials of miscanthus with different treatments: a) chopped and b) steam pressurised at macro level, respectively as well as c) chopped and d) steam exploded material at micro level by analysing with a scanning electron microscope, respectively.

The results of the shift in particle size distribution of the raw material and different treatments are shown in Fig. 4. The portion of material fractions below 250 μm were almost zero for the chopped raw materials. By contrast the treated materials show an amount of 8%. Most of this fraction is dust and cannot be used for the development of insulating materials as well as this material is lost for the insulation material. However, this fraction has a high impact on the bulk density. In the range between 250 μm and below 1.0 mm the amount of particles was also lower for the chopped samples than for the steam pressurised samples.

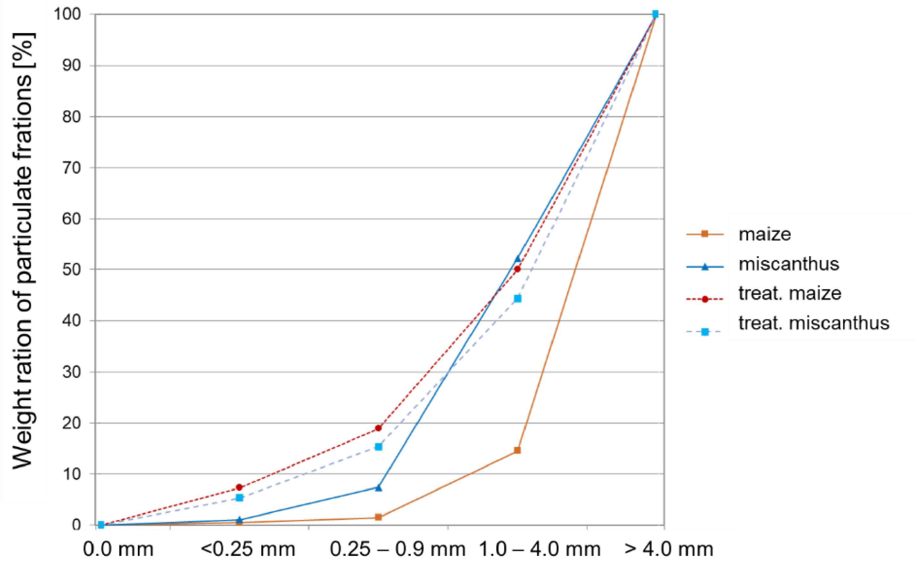

Figure 4. Particle size distribution regarding the different raw materials and treatments.

The sieve results for the particle fraction between 1.0 and 4.0 mm depict that the relative amount of copped miscanthus, steam treated maize and miscanthus have a range of 29 to 45%. Only the chopped maize samples have very low portion in this class, which the relative amount laid around 13%. This behaviour can be explained by the structure of the chopped maize straw. The samples from the maize had a higher portion of leaves than stem material, whereas the miscanthus samples presented a high amount of stem material (cf. Figs 2, a and 3, a). The relative amounts of the sieve fraction larger than 4 mm particle size showed different results. The values from the copped maize material showed the highest amount about 86%, which is followed by the steam pressurised miscanthus samples with 56% and treated maize material with 50%. The lowest values presented the chopped miscanthus materials with 48% of the relative portion.

Physical changes in materials properties

Table 1 shows that the bulk densities of the raw materials and stream pressurised materials are different. Due to the treatment the bulk density of the maize samples increased. This phenomenon results due to a relative high amount of dust in the materials compared to the untreated maize material (c.f. Fig. 4). For the miscanthus material a contrary change can be observed. The bulk density decreases due to the effect of the steam explosion process through the increase of material fraction in the range between 1.0 to 4.0 mm.

Table 1. Overview of different material properties of chopped and stream pressurised materials at 200 °C

Crops	Temperature (°C)	Retention time (min.)	Bulk density (kg m^{-3})	Moisture content# (%)
Maize	0	0	48.6	9.5
Maize	200	20	82.7	6.1
Miscanthus	0	0	137.6	10.1
Miscanthus	200	20	93.4	5.7

material storage at 23 °C temperature and 50% relative humidity.

Measurements of the thermal conductivity of chopped and steam treated maize showed that there is only a small difference between both materials, as bulk densities are very similar (Table 2). Separating the raw materials into different fractions show different results of the thermal conductivity. The larger the particle size the higher are the values of the thermal conductivity. The measurement area can compactly fill in with small particles. This phenomenon can be seen if the bulk density in the measurement area is compared. Higher densities represented higher amount of material and less air inside the measurement field.

Table 2. Results from the thermal conductivity measurements of maize samples at 10 °C temperature

Crops	Temperature (°C)	Retention time (min.)	Particle size (mm)	Bulk density (kg m^{-3})	Thermal conductivity (W (mK)$^{-1}$)
Maize	0	0	org.#	98.0	0.04600
Maize	200	20	org.#	100.8	0.04573
Maize	200	20	> 4	80.0	0.04626
Maize	200	20	< 4–2	86.2	0.04661
Maize	200	20	< 2–1	94.1	0.04362
Maize	200	20	< 1–0.25	106.6	0.04277

original material samples without fractionation.

Table 3. Results from the thermal conductivity measurements of miscanthus samples at 10 °C temperature

Crops	Temperature (°C)	Retention time (min.)	Particle size (mm)	Bulk density (kg m^{-3})	Thermal conductivity (W (mK)$^{-1}$)
Miscanthus	0	0	org.#	151.9	0.05147
Miscanthus	200	20	org.#	120.0	0.04726
Miscanthus	200	20	org. #	100.3	0.04546
Miscanthus	200	20	< 4–2	98.1	0.04725
Miscanthus	200	20	< 2–1	79.1	0.04484
Miscanthus	200	20	< 1–0.25	96.0	0.04286

original material samples without fractionation.

Table 3 shows the thermal conductivity of the miscanthus samples. However, the high bulk density in the measurement area of chopped miscanthus material of 151.9 kg m^{-3}, which could not obtain with the steam pressurised materials. The results show that treated material samples have a lower thermal conductivity compared to the chopped miscanthus material. At a bulk density of 100.3 kg m^{-3} a thermal conductivity value of 0.04546 (W $(mK)^{-1}$ was determined and this value was lower than the measured value from the treated miscanthus materials with 120.0 kg m^{-3} bulk density. The low values in thermal conductivity and bulk densities are affected from the low moisture content of the steam pressurised material samples.

Measurements of the thermal conductivity of the fractionated treated materials showed a clear trend. With decreasing particle size, the values of thermal conductivity are getting lower to 0.04286 (W $(mK)^{-1}$). However, this behaviour is not related with the bulk density as measuring with the maize material. Thermal conductivity measurements with larger particle size than 4 mm could not be conducted due to the insufficient coverage of the test area.

CONCLUSIONS

Steam explosion treatment were used to defibrillate the straw materials of maize and miscanthus. These particles and fibres were analysed for the applicability as insulating material (e.g. bulk insulation). Within the limitation of this study, the main conclusion can be drawn as follows:

- The chemical composition of treated material is changing.
- The particle size of the steam pressurised samples decreases compared to the reference samples due to destroying of natural fibre composite.
- Lower material moisture contents were measured for the treated samples.
- The use of steam pressurised materials has positive effects on the bulk densities and thermal conductivity by using fractionated materials.

According to these results the possible application of steam pressurised materials as bulk insulating material is shown. These findings provide a basis for help in transfer from laboratory to industrial conditions for consumer applications.

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Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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