Industrial Crops & Products 125 (2018) 105–113

Contents lists available at ScienceDirect

Industrial Crops & Products

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

Effect of wheat husk surface pre-treatment on the properties of husk-based composite materials

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ARTICLE INFO

Keywords:
Wheat husk
Renewable resources
Composite material
Particleboard
Pre-treatment

ABSTRACT

The effect of different types of winter wheat husks treatments (alkaline, hydrothermal and plasma treatment) on the properties of the particle material, as well as on composite materials made from such pre-treated husks, was studied. After pre-treatment, the surface energy, equilibrium moisture content, surface morphology using SEM, elemental content on the surface and the fluorescence life time (FLIM) of husks were estimated, as well as the IR spectra (FTIR) of pre-treated husks. Boards with target density of 450 kg/m^3 were produced using urea-formaldehyde resin (resination 9%) and the equilibrium moisture content, thickness swelling, vertical density profile, internal bonding, bending strength and insulation properties of boards were examined. Modification in 2% sodium hydroxide solution caused noticeable erosion of husks resulting in higher lignin content, higher equilibrium moisture of husks and consequently higher thickness swelling of boards made from pre-treated husks and lower mechanical properties of those boards. Both hydrothermal (boiling in water) and plasma treatment (air plasma at atmospheric pressure, jet system of application) resulted in the desired effect in terms of an increase in the surface energy of husks, decrease of husk equilibrium moisture content and increase of mechanical properties of boards. All of the manufactured boards exhibited low thermal conductivity that ranged from 0.0714 W/(mK) to 0.0783 W/(mK).

1. Introduction

At present, the main raw material for particle board production is low quality wood, and its share in the production of particle board is about 70%. Nevertheless, thanks to improved woodworking technologies, this wood is increasingly being used in the production of glued laminated timber or cross-laminated timber, where its value increases (Klímek and Wimmer, 2017). However, the lack of lower quality logs is not only caused by the above-mentioned competition, as the potential mass for the production of chips is also used by the paper, energy and chemical industries. The requirements for the supply of this wood material are increasing and have already resulted in its shortage, which is reflected, for example, by a reduction in the profits of woodworking enterprises (Seintsch, 2011; Lauri et al., 2012; Sujová et al., 2017), and the increased demand for this raw material necessarily increases wood prices. This increase in the price of the main input raw material can cause supply outages or reduce the competitiveness of the particle board, and growth of wood in Europe do not cover this growing demand (Bostedt et al., 2016). A possible solution may be partial replacement of wood by post-harvest crop residues in the production of composite materials, and this solution seems appropriate both economically and politically (Klímek and Wimmer, 2017). In recent years, research focusing on the use of alternative raw materials in wood-based composites has already been carried out. Research has been conducted concerning the use of oil palm trunk (Lee et al., 2018), rapeseed straw (Dukarska et al., 2017; Hýsek et al., 2018b), soybean and wheat straw (Sitz et al., 2017; Hýsek et al., 2018a; Boquillon et al., 2004), durian peel and coconut coir (Khedari et al., 2004), hemp fibres and rice husks (Battegazzore et al., 2017, 2018), castor stalks (Grigoriou and Ntalos, 2001), eggplant stalk (Guntekin and Karakus, 2008), sunflower, topinambour and cup-plant (Klímek et al., 2016a,b). Using these materials, both the physical and mechanical properties of the produced boards were improved. However, in most cases the amount of adhesive used was higher than the amount of adhesive used for industrial production of particleboards from wood.

The question of the use of alternative raw materials for the production of composite materials must be dealt within terms of production technology. It is well known from an extensive amount of research that wax and siliceous substances occur on their surfaces in the stems of annual plants, which cause difficulties when they are glued (Bekhta

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https://doi.org/10.1016/j.indcrop.2018.08.035
Received 29 March 2018; Received in revised form 24 July 2018; Accepted 11 August 2018
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et al., 2013; Mamun and Bledzki, 2013; Le Moigne et al., 2014; Qian et al., 2015; Částková et al., 2018). In order to disrupt the waxy layer that inhibits high-quality bonding between the particle and the adhesive, particle pre-treatment is typically required in the production of composite materials from these stems (Gajdačová et al., 2018; Hýsek et al., 2018b). Instead of particle pre-treatment, post-treatment of produced boards is also possible. (Lee et al., 2018) enhanced physical properties of urea formaldehyde-bonded oil palm trunk particleboard via post heat-treatments in palm oil.

In this research, weak bonding between particle and adhesive was also observed when gluing wheat husks. The husks are a by-product in the production of grain and constitute an available source of cellulose and lignin in large quantities. Wheat husks contain 36% cellulose, 18% hemicellulose, 16% lignin, 9% starch, 6% protein and 5% fat (Bledzki et al., 2010). Due to their high cellulose content and fibrous structure, the husks have the potential to be used in cellulose-based composites. Based on the weight ratio between wheat and husk production of about 5:1 (McCartney et al., 2006; Bledzki et al., 2010; Đorđević and Antov, 2016), we are able to estimate that approximately 30×10^6 t of wheat husk (Eurostat, 2017) are produced annually in the European Union, which, in terms of weight, represents 1.5 times the amount of wood needed for particle board production in the European Union (Klímek and Wimmer 2017). The husks are currently used for feeding livestock or biofuel production (Bledzki et al., 2010). Research activities are also evident in the area of bio-composites (Mavani et al., 2006; Sobhy and Tammam, 2010; Mamun and Bledzki, 2013) or heavy metals absorption materials (Sud et al., 2008). Loosely laid wheat husks exhibit excellent thermal insulation properties (Pavelek et al., 2018). In this research, we would like to partly preserve this natural property of husks, and therefore, boards with lower average density than that of commercially produced particleboards were produced.

This paper describes the effect of different types of winter wheat husks surface pre-treatments on the properties of the particle material, as well as on composite materials made from such pre-treated raw materials. Specifically, this concerns the chemical, hydrothermal and plasma pre-treatment of winter wheat husks, namely crops with large sowing areas in the Czech Republic and the European Union, and whose post-harvest residues are available in large quantities. It is hypothesized that through husk pre-treatment, the adhesion between husk and adhesive can be significantly improved. It is anticipated that the composite properties can be improved by adjusting effective husk pre-treatment, and therefore, three pre-treatment methods were chosen based on a literature search. Chemical, hydrothermal and plasma pre-treatment are expected to erode the waxy surface of wheat husks, which will subsequently lead to better adhesion between the husk and the adhesive.

2. Materials and methods

2.1. Wheat husks

Wheat husks (Fig. 1) were supplied by a local supplier from the Central Bohemia Region in the Czech Republic. Digital image analysis was used in order to characterize distribution of particle dimensions. The proportion of individual fractions was defined per 100 g of material sample using a CAMSIZER analyser (Retsch Technology GmbH, Haan, Germany).

Fig. 1. Wheat husks for board production.

2.2. Wheat husk pre-treatment

Three types of wheat husk pre-treatments were selected; hydrothermal pre-treatment (marking H), alkaline pre-treatment (marking A) and plasma pre-treatment (marking P). Untreated husks were also used as a reference (marking R). The hydrothermal pre-treatment was carried out by boiling in water for 45 min and the chemical pre-treatment was executed by soaking the husks in 2% sodium hydroxide solution (NaOH) at 25 °C for 45 min. The husk to sodium hydroxide solution ratio was 1:5. After both treatments, the husks were flushed with water and then oven-dried to 6% moisture content. Plasma pre-treatment was carried out using jet plasma generator. Cold air plasma was used. The parameters of plasma jet of voltage 26.9 V and current 6.9 A were set. The application of plasma on the husks was performed using circulation of 80 g husks in an application bowl for 3 min. The principle of the application is further described in Hýsek et al. (2018a).

2.3. Characterization of modified material

After the wheat husk pre-treatment, several analyses were used in order to characterize the modified particle material. Scanning electron microscopy (SEM) and elemental analysis of wheat husks were performed using a MIRA 3 electron microscope (Tescan Orsay Holding, Brno, Czech Republic) with a secondary electron detector operated at 15 kV acceleration voltage. The elemental compositions of the husk surface were examined by an energy dispersive spectroscopy system (Bruker XFlash X-ray detector, Karlsruhe, Germany, and ESPRIT 2 software). The surface tension of pre-treated husks was estimated using the test ink method; non-toxic test inks Arcotest (Arcotest GmbH, Moensheim, Germany) were used. Test inks were applied to particles using a small brush, and the reaction of test ink was observed using the DTX 90 digital microscope (Levenhuk, Tampa, USA). The equilibrium moisture content of the modified husks was estimated using moisture analyser Ultra X 3011 (A&P instruments, Detmold, Germany) after conditioning of husks in an environment of 65% relative humidity and 20 °C. In order to characterize chemical changes in the mass, samples for FLIM (Fluorescence-lifetime imaging microscopy) and FTIR (Fourier transform infrared spectroscopy) analyses were disintegrated and homogenized using laboratory ball mill cooled with liquid nitrogen. In order to perform FTIR, powder from husks was placed directly on the ATR (attenuated total reflection) crystal and pressed with reproducible pressure. IR spectroscopy was performed using a Paragon 1000 FTIR spectrometer (PerkinElmer, Waltham, Massachusetts, USA). The spectral range was recorded from 3800 cm^{-1} to 500 cm^{-1} with a count of 10 scans each. For the FLIM analysis, the Leica SP8 module with TCSPC (Time-Correlated Single Photon Counting) capability was employed. A pulsing laser at 405 nm with pulsing frequency of 40 MHz was used for specimen excitation. The expose period was 1.5 ns and the signal was taken from square of 581 × 581 μm in dimension. A five-component multi-exponential decay model was fitted to the fluorescence decay data, which was found to provide the optimum fit measured by minimising the χ^2 value.

2.4. Particleboard manufacturing

Urea-formaldehyde resin K350S was used as an adhesive to produce boards made from the wheat husks. The composition of the adhesive mixture is specified in Table 1.

Adhesive mixture was applied on husks using a laboratory blender, and after adhesive mixture application, the husks were dried to 7%

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