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Adhesive distribution related to mechanical performance of high density wood fibre board

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ABSTRACT

In a full-scale mill experiment two groups of high density wood fibre boards were produced. While one group was bonded with a standard melamine reinforced urea-formaldehyde adhesive, a second group was bonded with a modified adhesive yielding systematically improved board properties at identical amounts of adhesive applied. By means of a novel fluorescence-microscopic method using the dye Acriflavine to colour the cured adhesive after board production, adhesive distribution within the industrial produced boards was evaluated and quantified. Very clear differences in the size distribution of the two adhesives were found, leading to the conclusion that a relationship exists between adhesive distribution and mechanical board performance.

1. Introduction

The disintegration of wood into particles and fibres, followed by re-assembly and adhesive bonding, leads to wood composite products with high structural homogeneity, dimensional stability, and standardised processing characteristics within very narrow limits of variability. Besides the nature of wood particles or fibres used, and processing parameters such as assembly, pressure and duration of hot-pressing, the type of adhesive applied plays a crucial role in the economic production and the technological performance of wood composite panels. Wood fibre boards may be produced with very little or no adhesive, relying on intrinsic fibre-fibre bonds built-up in wet processing. However, modern state of the art wood fibre panels such as medium density fibre board (MDF) or high density fibre board (HDF) rely on efficient adhesive bonding. Besides a minor fraction of phenolic adhesive bonded boards, amino resins are the adhesive of choice due to its highly competitive price, high reactivity, and good bonding performance. Since the amount of adhesive spent contributes significantly to the overall product cost, any optimisation of adhesive use is welcome. In order to do so, the relationships between adhesive mechanics, interfacial adhesive-wood fibre interactions, and adhesive distribution on the one hand, and fibre board performance on the other hand need to be studied. With regard to adhesive distribution in Urea-Formaldehyde resin (UF)-bonded boards, the lack of natural colour contrast between UF and wood poses a challenge for microscopic studies.

X-ray photoelectron spectroscopy (XPS) technique was used for high resolution imaging of elemental concentrations [1,2]. For comparison, the same authors used confocal laser scanning microscopy (CLSM) for adhesive detection. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used in combination with energy-dispersive X-ray spectroscopy (EDXS) [3,4] as a tool for the detection of UF resin penetration into wood cell. Thumm et al. [5] used SEM to investigate the penetration of urea melamine formaldehyde adhesive in wood fibres. With the same intention Cyr et al. [6] used CLSM. Various researchers reported the adhesive coverage ratio of MDF fibres by using CLSM and fluorescence labelling [5,7–13]. Electron energy loss spectroscopy (EELS) in combination with TEM was used to determine the distribution of melamine-formaldehyde adhesive in wooden cell walls [14]. Gierlinger et al. [15] studied the melamine-formaldehyde adhesive content within cell walls of impregnated spruce wood by using UV microscopy and confocal Raman microscopy. UV microscopy was also used by Gindl et al. [16,17]. Finally, UF resin distribution in wood composites has also been determined by light and fluorescence microscopy [8,18–20].

An analytical method was developed based on our method, involving a combination of fluorescent and visible (Vis) stains recently applied with success to UF-bonded particle board [19,20]. Instead of using Brilliant Sulphaflavine to colour the adhesive, the dye Acriflavin was

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used. This dye has been proven to be adequate for colouring UF resins by incorporating it during resin synthesis [12,13]. In this work, the dye is used to colour the cured resin *after* boards have been produced. Thus, a method is obtained that is capable of delivering valid information on adhesive distribution in industrial produced HDF boards. Furthermore, adhesive distribution data will be discussed with respect to potential relationships with mechanical board performance.

2. Experimental details

HDF boards were produced in a full-scale industrial mill trial using a standard melamine-reinforced UF (mUF) termed adhesive (A), and a modified optimised mUF termed adhesive (B) under exactly the same conditions. Costs for the modification of adhesive (B) are in a range that allows industrial implementation. The viscosity of both adhesives (A) and (B) was the same. The resin loading in the HDF boards was set in the common industrial range of 10–13% solid resin on dry wood, and was equal for boards produced with (A) and (B).

Mechanical characterisation was carried out in three-point bending according to EN 310 [21]. Internal bond strength was evaluated according to EN 319 [22], and thickness swelling after 24 h immersion in water was measured according to EN 317 [23].

The sample preparation for microscopic analysis of adhesive distribution described in Fig. 1 starts with cutting 8 small prisms with an edge length of 3 mm from the core of board series of each adhesive group. These blocks were then impregnated with epoxy resin after all air was removed during a vacuum step. After curing the epoxy at 60 °C the blocks were trimmed and sectioned with an ultramicrotome equipped with a diamond knife. The thin slides have dimensions of approx. 2 × 3.0 mm, with the thickness of the thin sections set to 2 µm. For a better contrast between adhesive and fibres the sections were stained twice. Firstly, Acriflavine, an orange fluorescent dye, was used to stain the adhesive. Secondly, Gentian Violet was applied to stain cell wall material. Between and after the staining steps, the specimens were repeatedly washed with deionised water, and finally mounted to glass slides. With a fluorescence microscope (Zeiss Axioplane 2 Imaging) images were taken in incident light mode using an ultraviolet lamp Fluo Arc HBO 300 in order to capture fluorescent areas originating from stained adhesive. A 450–490 nm excitation filter and a 515 nm emission filter were used. All images were taken with a magnification factor of 200 × and a resolution of 0.528 µm/pixel. In order to cover a large

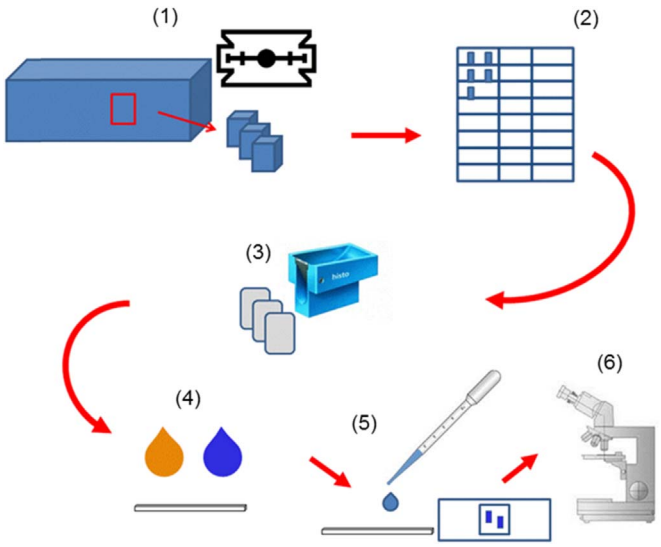

Fig. 1. Schematic of the sample preparation process. Small cubes are prepared from the HDF boards (1), embedded in epoxy resin (2) and cut into thin Section (3). The thin sections are deposited onto glass slides where they are stained (4) and washed (5), before being observed in the light microscope (6).

Table 1
Comparison of important physical characteristics of series of HDF boards bonded with two different mUF resins (average values of samples taken during the industrial trials).

Adhesive type	Density (g cm^{-3})	Flexural strength (MPa)	Flexural modulus (GPa)	Internal bond strength (MPa)	24 h thickness swelling (%)
mUF A	0.89	50	4.9	1.68	10.4
mUF B	0.90	53	5.1	2,03	9.6

sample area several overlapping images were taken. The images were matched with correspondent neighbouring images using the image processing software MosaicJ, a plug in from ImageJ. Further analysis was done with Photoshop and ImageJ. In order to improve the contrast between wood fibres and adhesive, respectively, and to decrease background fluorescence a threshold was set in the Lab colour space at L 110/255, a 0/255 and b 0/255. With binary images it was possible to quantify the size and size distribution of adhesive areas within the HDF boards.

3. Results and discussion

3.1. Mechanical board properties

A summary of the most relevant physical parameters for evaluating HDF board quality is given in Table 1. In order to determine these properties, the quality control department analysed numerous board samples taken during the large scale industrial trials. According to the experience of this department and the industrial partners involved, the difference between two industrial trials with adhesive (A) and two industrial trials with adhesive (B), is regarded significant.

Since typically, board properties vary significantly with density [24,25], comparable density is prerequisite when possible causes for mechanical variability of boards are being discussed. In case of the two variants of HDF examined in the present study, the industrial line produced without deviations and no obvious difference in density was observed, with an average density of 0.9 g cm^{-3} for both variants. Despite roughly identical densities, mechanical board properties varied clearly. Consistently, improved performance was observed for the variant bonded with modified mUF adhesive, compared to the unmodified reference. While the trends towards improved properties in 3-point bending are weak, a strong improvement in internal bond strength of 21% on average was seen. In parallel, an 8% reduction of thickness swelling after 24 h immersion in water was measured. Since identical amounts of adhesive were applied and no differences in density were found, the reason for improved board properties is to be found in other board characteristics. As the modification of the resin was made with the goal to alter the adhesive distribution, this parameter was examined to help to identify possible causes for the variability in physical properties observed.

3.2. Adhesive distribution

Representative images captured in Vis and fluorescence mode demonstrate the very significant improvement in optical contrast between wood and adhesive achieved by means of fluorescence staining. In Vis mode (Fig. 2a) the general structure of HDF board, consisting of individualised fibres and fibre-bundles, is easily discernible due the very efficient staining of wood cell walls by means of Gentian violet. However, even at close inspection, adhesive cannot be easily identified in Vis mode due to lack of contrast. When switching to fluorescence mode in the same specimen region (Fig. 2b), cell walls appear in bright red, whereas small spots of adhesive may be easily discerned due to their greenish fluorescence. At higher magnification (Fig. 2c-d), adhesive is predominantly found in inter-fibre spaces, whereas internal fibre