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DETERMINATION OF SHARE OF ADHESIVE ON PARTICLES WITH FT–IR SPECTROSCOPY

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ABSTRACT

It is well known, that particle size influences the surface of adhesive applied on the particles during the particleboard production, and it is also well known that "only" 10 % of adhesive (mass based) is used for particleboard production. Since most adhesives that are used in particleboard industry are colourless it is difficult to determine its share on the particle or even the surface of adhesive on particles. The purpose of this research was to determine weather it is possible to determine the share of adhesive on particles with FT-IR spectroscopy. Beech (*Fagus silvatica* L) particles five different fractions were blended with urea-formaldehyde adhesive. According to the wave number significant for presence of urea-formaldehyde adhesive and the reference the share of adhesive was quantified. As comparison the results obtained by image analysis were used. The results show that the surface covered with adhesive is increasing by decreasing the specific surface area of particles. We also determined that there is a difference in the spectra between unblended and blended particles.

KEY WORDS: particles, share of adhesive, FT-IR spectroscopy, wave number

INTRODUCTION

Despite the fact that „only“ 10 % of adhesive (mass based) is used for particleboard production it presents an important impact on the board properties. At the beginning, the general idea was that adhesive impacts on properties is related mainly to the type of it and its share, but later it was determined that surface covered with adhesive, its distribution on the surface of particle and between particle and also the penetration of adhesive plays important role on the quality of produced boards.

To achieve a good bond between particles, adhesive have to wet the surface of particle and also have to spread over the surface. Important factor for optimal adhesion/cohesion strength of bond is also the penetration of adhesive.

If we focus only on the wetting ability of adhesive, then wood surface need's to be adequately prepared. The surface needs to be smooth, clean and without stains that would negatively influence the wetting of surface and penetration of adhesive. Such clean, smooth surface is hard almost impossible to achieve, because at manufacturing of particles they are thorn, crushed, etc.

The creation of adhesive bond between particles is also influenced by the drying of particles to the moisture content appropriate for further processing (1...4 %). At drying, when particles are subjected to high temperature (up to 400 °C), we change the surface of particles; extraction components of wood emigrate to the surface and are bonded to the free radical on the surface hence surface is irreversible closed. Due to drying chemical deactivation of surface could occur, caused by chemical contamination by the hydrophobic and chemically active extraction components and due to the oxidation of possible bonding places on particles. Those processes that occur at drying could also slower the curing of adhesive and hence also on the cohesion and adhesion strength of the bond. Wetting of the surface is at particleboard production diminished additionally by hot pressing, where temperature at urea-formaldehyde adhesive could be between 170 and 200 °C. Due to high temperature volatile compounds of wood emigrates on the surface during curing of adhesive, what slows the process of wetting of the surface and penetration of adhesive (Vick 1999).

Due to difficulties at determination the surface of particles covered with adhesive Lehmann (1965) proposed that prior to spreading (blending) proper staining technique should be used, what would enhance the contrast between particles and adhesive. Author used Rhodamin B (0,6 % with regard to the mass of adhesive). Lehmann determined that the best board properties are obtained when adhesive is uniformly spread over the particle surface and also between particles.

Lehmann (1965) researched the distribution of adhesive on particle under ultra violet microscope. Author determined that at spraying, small droplets of adhesive occur on particles (radius of droplet between 32 and 56 μm), which later, due to the mixing in the blender and high pressure and high temperature (at pressing), joins into almost continuous glue line.

For the determination of the surface covered with adhesive Wilson and Krahmer (1976) successfully used scanning electron microscope. (SEM).

Dunky (1988) made a research on the relation between particle size and surface covered with adhesive. Author determined that in the particle mixture (according to size), the adhesive is not uniformly distributed between particles, due to the size of droplets. Dunky determined that in the particle mixture more adhesive (higher surface covered with adhesive) could be found on the bigger particles (lower specific surface area).

Niemz et al. (1990) and Körner et al. (1992) reported about the possibility of usage of FT-IR (Fourier Transform Infrared) spectroscopy. They determined the correlation between the peeks significant for adhesive, reference peek and share of adhesive. Due to the change of the share of adhesive mentioned authors determined the changes in peeks between 1540...1560 cm^{-1}, 1655...1665 and 1511...1512 cm^{-1}. With increasing share of adhesive also relation between adhesive peek and reference peek increased.

As we can see from previous researches the determination of the surface cover with adhesive at particles is difficult, due to relatively small size of particles and also because of the small share of added adhesive and its colour (especially UF adhesive). The most appropriate method, according to above mentioned authors is to dye the adhesive prior to its spraying on particles. They have also expressed that uniform spraying of adhesive n particles plays important role at achieving good quality of boards.

The aim of this article is to present the possibility of usage of the FT-IR spectroscopy for the determine weather it is possible to detect the presence on adhesive on particles and hence its share on particles.

MATERIAL AND METHODS

For the purpose of this research beech particles and UF resin were used. Particles were produced from beech chips in laboratory conditions (in laboratory chipper). Blended and unblended particles were separated into 5 different fractions that are most common in surface layer (Tab. 1).

Tab. 1: Fraction used and its share in surface layer

Fraction	Share
	%
0,237	53,73
0,600	34,45
1,000	9,60
1,270	2,00
1,500	0,22

Since some authors (Lehmann 1965, Lehmann 1968) reported that determination of surface covered with adhesive is difficult, especially when colourless UF resin is used, we have decided that we will dye adhesive prior to blending. For staining Rhodamin B was used. We added 0,5 % of Rhodamin B, (according to mass of adhesive). The share of adhesive was similar as it is in industry for surface layer particles - 11,5 %. Blended particles were exposed to temperature 180 °C. Exposure time was 4 minutes.

Fourier Transform Infrared (FT-IR) analysis of the share of adhesive on particles

For the determination of the share of adhesive on particles Fourier Transform Infrared (FT-IR) spectroscopy was used. FT-IR spectroscopy is a measurement technique where IR radiation from coherent source (IR light) passes through a sample. Some of the infrared radiation is absorbed by the sample, while some of them passes through; are transmitted. The resulting spectrum represents the molecular absorption and transmission, what creates a molecular fingerprint of measured sample.

The measurement was done by Diffuse Reflectance Infrared Furier Transform (DRIFT) technique, which is the most common technique, used for measurement of particles, glued or coloured surfaces. At DRIFT analysis technique the beam of IR light is focused on the surface of a sample and the diffusely reflected light is collected by the integrating sphere and analyzed. Because the surface of the sample is rough, the beam is deflected in any direction.

At DRIFT spectra some distortion of the band, shifts of maxima and the appearance of spurious band can occur. This deviation can be minimized by the dispersion of sample in a relatively low concentration in a non-absorbing material of high reflectance such as KBr or KCl. Spectra obtained by such technique is expressed in Kubelka-Munk (KM) units:

$$F(R) = \frac{(1-R)^2}{2+R}$$

where

R	the ratio of the diffuse reflectance of the sample at the “infinite depth” to that of the non-absorbing material in which the sample is dispersed
F(R)	the Kubelka-Munk function

As we mentioned before, we need to disperse the sample in the non-absorbing material. In our research we used KBr. Each fraction of blended and unblended particles was grind in a laboratory grinder to maximum size 50 µm. In the mortar 0,05 g of grinded particles (blended and unblended) were mixed with 5 g of KBr powder. As a reference the spectra of clean KBr was used. For each fraction 10 measurements were made.

At obtained spectra's for blended particles we identified the peak's that are different from those at unblended particles. We also looked for the peaks that are significant for the presence of UF adhesive. Such peaks are, in accordance to Köroner et all (1992) at wave number 1540...1550 cm^{-1} and 1655...1665 cm^{-1}.

RESULTS AND DISCUSSION

As we know, the wetting and penetration of adhesive is dependant also from the particle size - fraction used. At testing 258 g of adhesive was sprayed on the 980 g of particles. If we calculate the amount of adhesive per surface of particle, with regard to the fraction, than we can see that it is the biggest at the fraction 1,500 (Tab. 2).

Tab. 2: Amount of adhesive per surface of particle with regard to the fraction used

Fraction	Particle surface	Amount of adhesive
	m^2	$g.m^{-2}$
0,237	44,77	5,67
0,600	25,46	10,13
1,000	13,70	18,83
1,270	13,79	18,71
1,500	8,72	29,57

The surface is calculated to the 980 g of particles

At fraction 0,237 the amount of adhesive is the smallest (only 5,67 $g.m^{-2}$), while at fraction 1,500 the amount of adhesive per particle surface is almost 5×higher. On the basis of the calculation presented in Tab. 2, we can conclude that small amount of adhesive per particle surface, at particles with high specific surface area (smaller particles), could lead to smaller penetration or smaller surface covered with adhesive - small share of surface covered with adhesive.

At Fig. 1 to 5, where red colour presents the adhesive, we can see that the biggest surface covered with adhesive is at biggest fractions.

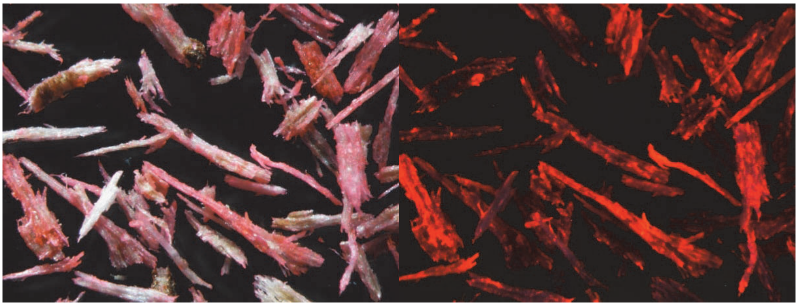

Fig. 1: Blended beech particles fraction 0,237 (magnification 20×)

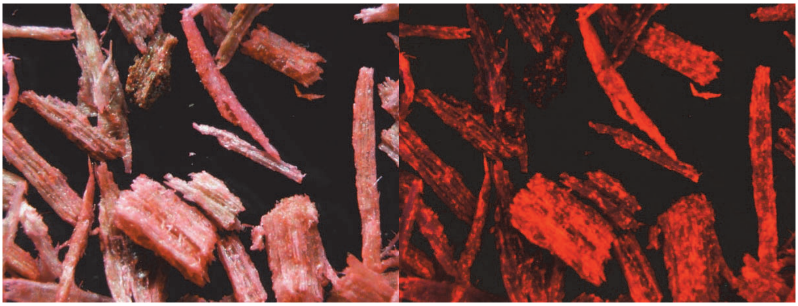

Fig. 2: Blended beech particles fraction 0,600 (magnification 20×)

Fig.3: Blended beech particles fraction 1,000 (magnification 20×)

Fig. 4: Blended beech particles fraction 1,270 (magnification 20×)

Fig.5: Blended beech particles fraction 1,500 (magnification 20×)

The left image on Fig. 1 to 5 was taken at normal and right under fluorescence light; hence the contrast between adhesive applied and particles was bigger (due to staining of adhesive with dye).

Dunky (1988) already reported about the influence of particle size on the surface covered with adhesive. Author discovered that adhesive in the mixture of particles (regarding the size of particles) is not uniformly distributed between particles. Dunky determined that the biggest share of adhesive was on the biggest particles, due to the size of the adhesive droplet, which was bigger that the width of particles (smaller fraction).

At our experiment the diameter of droplets was between 5 and 25 µm. The probability that particle will be "hit" by adhesive is bigger at bigger particles than it is at smaller particles. And if the adhesive "hits" particles than it will also spread over particle surface and penetrate. Even if we presume that at least one droplet of adhesive will "hit" the particle (with regard to the size of droplet justified presumption), than more droplets will be on the particles with higher surface area (fraction 1,500). In both cases more adhesive will most probably be on bigger particles.

FT-IR analysis of the share of adhesive on particles

Since in Tab. 2 and also in Fig. 1 to 5, we could saw that at bigger particles more surface was covered with adhesive we wanted to see weather this difference is also visible in the spectra obtained by FT-IR analysis (Fig. 6).

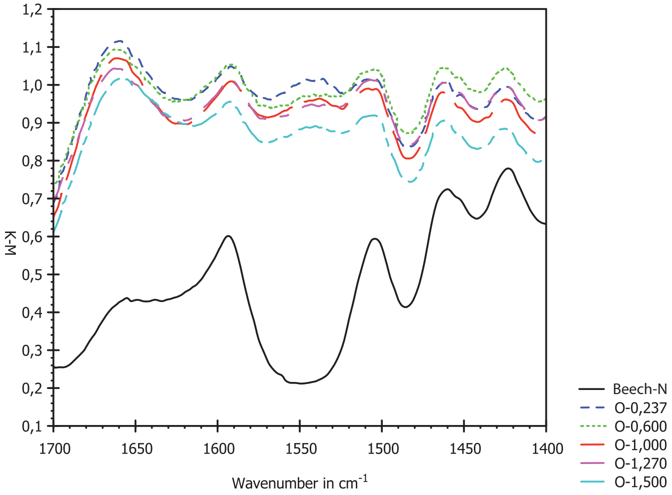

Fig. 6: DRIFT spectra of unblended (Bukev–N) and blended (O–0,237; O–0,600; O–1,000; O–1,270 in O–1,500) beech particles

In Tab. 3, the comparison between K-M values of unblended and blended particles is presented with regard to the values at chosen peaks.

Tab. 3: Comparison of K-M values for chosen peaks for unblended (Bukev-N) and blended (O-0,237; 0-0,600; 0-1,000; 0-1270 and O-1500) particles

	Wave number	K-M	Wave number	K-M	Wave number	K-M	Wave number	K-M
	cm^{-1}		cm^{-1}		cm^{-1}		cm^{-1}	
Bukev-N	3338	0,34	1655	0,43	1549	0,21	1111	1,28
O-0,237	3327	1,00	1659	1,11	1545	1,01	1109	1,39
O-0,600	3332	1,02	1663	1,09	1541	0,97	1109	1,40
O-1,000	3323	1,00	1662	1,07	1538	0,96	1111	1,37
O-1,270	3329	1,01	1663	1,04	1543	0,94	1115	1,37
O-1,500	3346	1,05	1659	0,99	1540	0,87	1110	1,36

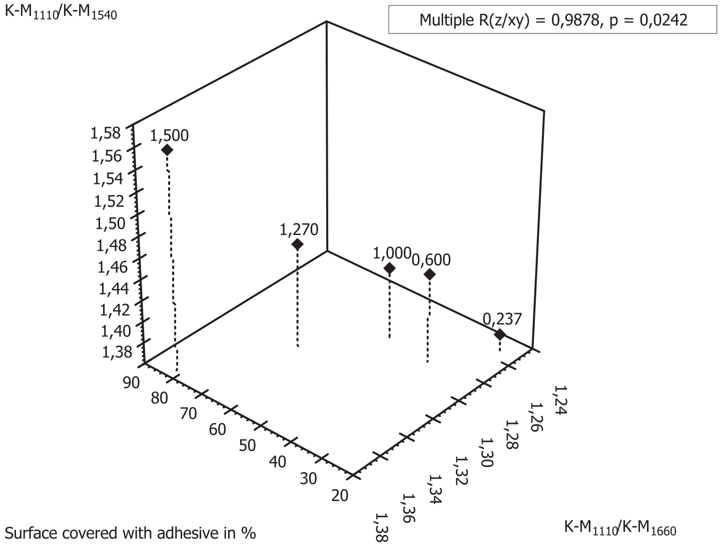

Fig. 7: Ratio between surface of reference band (K–M $_{1737}$) and surface of band significant for blended particles (K–M $_{1660}$ and K–M $_{1540}$) with regard to the surface covered with adhesive

As we can see from Fig. 6 and Tab. 3, there is a difference between unblended (Bukev-N) and blended particles (O-0,237; 0-0,600; 0-1,000; 0-1270 and O-1500). The difference is also visible between blended particles from different fraction.

Like Köroner et al. (1992), we also discovered a difference between blended and unblended particles at wave number between 3320…3350 cm^{-1}, 1655...1665 cm^{-1} and 1535...1550 cm^{-1}.

The peak between 3320…3350 cm^{-1} is significant for presence of urea-formaldehyde resin and is assigned to O-H and N-H vibrations. Also the peaks at the wave number between 1655...1665 cm^{-1} and 1535...1550 cm^{-1} are significant for urea-formaldehyde resin (Niemz et all. 1990; IR-Wizard results..., 2003). The peak between 1535...1550 cm^{-1} is, according to Köroner et al. (1992) and Müller et al. (2009), caused by secondary amides, resulting as a modification of pure urea as a component of UF resin The peak at wave number between 1105 and 1115 cm^{-1} is significant for cellulose and hemicelluloses OH group. The differences in K-M values at wave number 1105 and 1115 cm^{-1} were minimal, therefore we decided to use those values as a reference value.

At unblended particles K-M values at peaks between 1655 to 1665 cm^{-1} and 1535 to 1550 cm^{-1} were low, while at blended particles values at mentioned range were high due to the presence of urea-formaldehyde adhesive. As we can se from table 2 K-M values are dependant on the share of adhesive on particle. With increasing size of particles K-M values decreases.

If we compare data from Tab. 3 with the data reported by Medved and Resnik (2004) we can see that with increasing surface covered with adhesive the K-M value (ratio between reference values and values at significant peaks) increases (Fig. 7).

As we mentioned before there is a correlation between the share of adhesive on particles and the K-M values. With increasing particle size the surface covered with adhesive decreases as well as the ratio between K-M values.

CONCLUSIONS

With regard to the results presented in paper we can draw following conclusions:

- FT-IR spectroscopy can be used for estimation of the share of adhesive on particles
- There is a difference in spectra of unblended and blended particles and also between the particles from different fractions
- At blended particles peaks between 1655 to 1665 cm^{-1} and 1535 to 1550 cm^{-1} were determined (not present at unblended particles)
- With increasing particle size the surface covered with adhesive decreases as well as the ratio between K-M values

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