

## Validity limits of Kubelka–Munk theory for DRIFT spectra of photodegraded solid wood

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**Abstract** The validity of the Kubelka–Munk (K-M) theory was investigated to determine the IR absorption spectra of wood based on diffuse reflection infrared Fourier transform (DRIFT) measurements taken on photodegraded samples. After analysing plenty of DRIFT spectra of wood and examining the shape of the K-M equation, it can be concluded that the measured K-M function can be used as an absorption spectrum if the values of the function are below 14 K-M units. Above this limit, the K-M theory, which was created for poorly absorbing materials, does not give the absorption of wood properly. If a matt aluminium plate is used as a background material and the values are between 14 and 40, absorption changes can be calculated after normalisation of the spectra. This normalising manipulation is only successful if there is an absorption peak close to the examined one which does not change its absorption during the photodegradation.

### Introduction

Wood is a good light absorber for infrared (IR), visible and ultraviolet (UV) light. If the wood sample is lighted by UV light, it is totally absorbed in a 75- $\mu\text{m}$ -thick layer (Hon and Ifju 1978). The depth profile and penetration of light into wood was investigated by Kataoka et al. (2004, 2005, 2007). The penetration depth depends on the wavelength of the light. The secondary effects of UV light irradiation can be

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detected in 400–500  $\mu\text{m}$  depth for the aromatic skeletal vibration ( $1,510\text{ cm}^{-1}$ ) and in 600–700  $\mu\text{m}$  depth for carbonyl ( $1,730\text{ cm}^{-1}$ ) in a long-term treatment (e.g. 1,500 h; Kataoka and Kiguchi 2001). This means wood is not transparent for light, and the usual transmission method is not suitable to measure the absorption properties of wood. For this purpose, powdered wood is usually mixed with light transparent material. This mixture is pressed to pellet, and the pellet is used to measure the wooden absorption using the transmission method. For measurements in the IR region, potassium bromide is a widely applied matrix material. This method was an excellent analytical tool to discover the chemical constitution of wood by IR spectroscopy. Since the change caused by irradiation appears in a thin surface layer, it is not easy to remove the proper thin layer for pellet preparation. One important example is the UV photodegradation of wood. It is difficult to remove a layer thinner than 80  $\mu\text{m}$  which has suffered change during the treatment. Concerning this difficulty, many researchers apply the pellet method without indicating the thickness of the wood layer used for powder making (Hon and Feist 1986; Kosikova and Tolvaj 1998; Müller et al. 2003; Pandey 2005; Umemura et al. 2008).

The powerful Fourier transform IR technique gives the possibility to measure the diffusely reflected (DRIFT) intensity of a thin layer. This is a perfect technique to investigate the photodegradation because the information is given from the same layer which suffered photodegradation. The Kubelka–Munk (K-M) theory (Kubelka and Munk 1931; Kubelka 1948) was created to calculate the absorption properties of the surface layer using the reflected light. This theory was created for poorly absorbing materials. Since wood is a good light absorber, the K-M theory is widely applied to determine its light absorption (Owen and Thomas 1989; Horn et al. 1994; Tolvaj and Faix 1995; Craciun and Kamdem 1997; Takei et al. 1997; Zanuttini et al. 1998; Pandey 1999; Moore and Owen 2001; Freer et al. 2003; Weiland and Guyonnet 2003; Mitsui et al. 2003; Cui et al. 2004; Kishino and Nakano 2004; Tolvaj and Mitsui 2005). The situation is the same for pulp and paper research (Michell et al. 1989; Ristolainen et al. 2002). In many cases, even the K-M intensity data are not presented in the articles (Owen and Pawlak 1989; Zavarin et al. 1990; Pandey and Theagarajan 1997; Takei et al. 1997; Pandey and Khali 1998; Zhang and Kamdem 2000).

In many papers, milled wood or milled wood diluted with KBr powder is used to determine the absorption properties of wood by DRIFT technique (Berben et al. 1987; Faix and Böttcher 1992; Bouchard and Douek 1993; Stewart et al. 1995; Chang et al. 1998; Holmgren et al. 1999; Ferraz et al. 2000; Baldock and Smernik 2002; Vane 2003; Nuopponen et al. 2006; Toivanen and Alen 2007). Pandey and Theagarajan (1997) found that specular reflection causes distortion in the intensities of the bands in the range  $950\text{--}1,150\text{ cm}^{-1}$ , which can be minimised by diluting sample powder with KBr. Faix and Böttcher (1992) investigated DRIFT spectra of wood meal of increasing concentrations in KBr as well as increasing particle sizes and also that of solid wood surfaces. They concluded that in order to obtain reproducible FTIR spectra from wood surfaces, the roughness and the anatomical directions of the cut (tangential, radial and cross-cut) must be constant. It is difficult

to compare the FTIR spectra of different solid wood surfaces because the natural roughness of the wood species varies widely.

Only some publications consider that the results calculated by K-M theory are questionable. Jones and Heitner (1973) found that the light scattering and absorption properties of radial wood sections using 456 and 495 nm light could be expressed mathematically by using K-M equations. The radial sections contain both earlywood and latewood, hence the photometric results would be similar for different samples from the same tree. However, every tangential section cut from the same tree would show different proportions of earlywood and latewood resulting in different photometric properties for different samples. From that finding, it was concluded that the tangential wood sections do not follow the K-M equations. These results only indicate that the diffuse reflection properties of earlywood and latewood are different because of their different porosity and chemical structure and not because of the invalidity of the K-M equations. Shen et al. (2000) reported that the scattering intensity distribution in the direction across the wood fibres is very close to perfect diffuse reflection, while the distribution in the direction along the fibres is a combination of diffuse and specular reflection at 632.8 nm wavelength. Hembree and Smyrl (1989) reported on the distortions of the K-M spectrum around intensive absorption bands of calcium carbonate and caffeine. In the case of wood sample, Faix and Németh (1988) pointed out that the DRIFT spectra evaluation of photodegraded wood is very difficult because the surface properties strongly influence the quality of the DRIFT spectra. Michell (1991) observed anomalous effects in the IR 950–1,200  $\text{cm}^{-1}$  wavenumber region. Zavarin et al. (1990) reported on deviation in the intensity of the band at 1,166  $\text{cm}^{-1}$  measured in directions parallel and perpendicular to the grain. Tolvaj and Mitsui (2004) found that the intensity of the peak around 1,173  $\text{cm}^{-1}$  changed depending on the angle between the fibre direction and the incident IR light. The 950–1,200  $\text{cm}^{-1}$  IR region is where the wood has the greatest absorption. Here, anomalous effects of DRIFT spectra were found as well. These experiences focused the attention of the authors on examining the validity of K-M theory in the investigated range.

## Materials and methods

The investigated wood species were Japanese beech (*Fagus crenata*), poplar (*Populus caulescens*) and Japanese cypress (*Chamaecyparis obtuse*). Two samples were cut with a size of 50 × 10 × 2  $\text{mm}^3$  (long. × tang. × rad.). Planed surface of tangential cut was prepared (by industrial planer machine) containing only earlywood. After preparation, the air-dried samples were stored in an air-conditioned (20°C) laboratory (except irradiation). A strong UV light emitter, a mercury lamp, was applied. The total light emission of the mercury lamp was 320 W, and the samples were located 64 cm away from the lamp. (HAL 800NL, installed into a KBP.659 Nippon Denchi Co. Ltd. chamber.) The emission spectrum of the mercury lamp (supplied by the producer of the lamp) is located mainly in the UV region (80%). It contains 31% UV-A (380–315 nm), 24% UV-B (315–280 nm) and 25% UV-C (<280 nm) radiation. The irradiation was interrupted after 1, 3, 6,

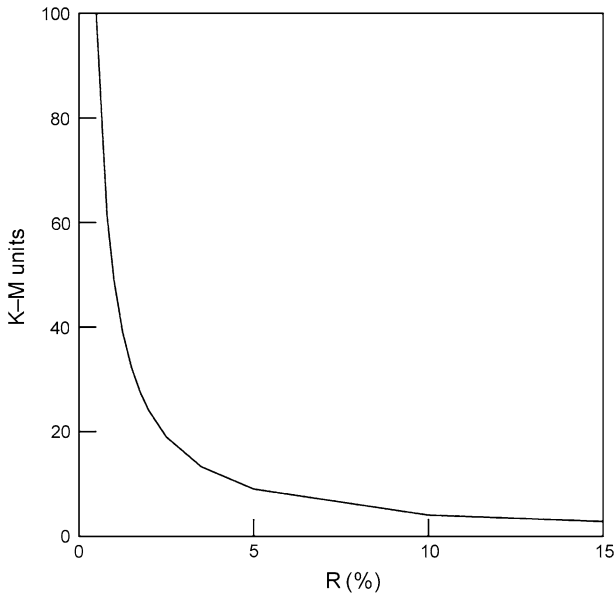
12, 20, 24 and 36 h of irradiation in order to determine any changes. Diffuse reflectance Fourier-transformed infrared (DRIFT) spectroscopy was applied to determine the changes caused by the light irradiation. The 4,000–400  $\text{cm}^{-1}$  wavenumber range was monitored. The IR measurements were repeated after each surface modification. The IR measurements were taken using a JASCO FTIR  $\mu$ -8,900 double beam spectrometer equipped with a diffuse reflectance unit (JASCO: DR-81). The resolution was 4  $\text{cm}^{-1}$ , and 64 scans were averaged. Five different places were identified for measurement on each sample, and two samples were irradiated and measured parallel. Average spectra were calculated from ten individual spectra at all irradiation times and used for further analysis. The background spectra were obtained against a matt aluminium plate, because its reflection properties were stable during the 10-month examination period. The spectral intensities were calculated in K-M units. A new series of beech and Japanese cypress samples were irradiated for up to 36 h. After 36 h of irradiation, the surface of these samples was pressed between two iron plates at a pressure of  $2.5 \times 10^6$  Pa for 10 s to reduce the roughness of the surface. For the difference spectra, the calculated K-M values were normalised to the peak at about 1,380  $\text{cm}^{-1}$ , and the normalised spectra were baseline corrected at 3,800 and 1,900  $\text{cm}^{-1}$  with straight line method. The difference spectra were calculated as the spectrum after irradiation minus the bulk (non-irradiated sample) spectrum. The whole experiment was repeated once to confirm the results.

## Results and discussion

The DRIFT spectrum gives only information about a thin surface layer. Using this method, the diffusely reflected light is collected, and the absorption is calculated by the Kubelka–Munk equation

$$\frac{k}{s} = \frac{(1 - R)^2}{2R},$$

where  $k$ ,  $s$  and  $R$  are the absorption coefficient, the scattering coefficients and the reflectance of a thick enough layer, respectively. Fortunately, the scattering coefficient hardly depends on the wavenumber of the IR light, so the shape of K-M function is similar to the wavenumber dependence of the absorption coefficient. This K-M function is usually used as the IR absorption spectrum. The K-M function is shown in Fig. 1. From IR spectrum of wood, the measured K-M values are usually higher than five units. This means that less than 10% of the incident light can be collected from the surface of the wood by the detector. The collected energy quantity is highly important in the case of background spectrum as well. The light source—sample—detector geometry is always designed, such that only the diffusely reflected light can reach the detector. For background spectrum, the proper material has bad mirror reflection and excellent diffuse reflectance properties. Metal mirror is never used for this purpose. Rough surface is recommended with particles as small as possible. Therefore, KBr powder is often used for this purpose. The K-M function

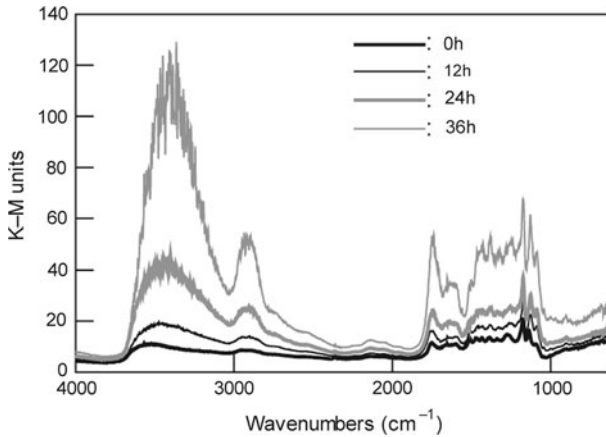


**Fig. 1** The Kubelka–Munk function

is almost linear below 14 K-M units, while between 14 and 40 units, it is a curve. Above 40 units, the function is highly sheer, so a small decrease in the reflection causes a large change in the  $k/s$  values. In this region, the change of K-M units is not proportionate to the absorption change. This happens if the surface roughness increases by roughening the surface with sandpaper. The rough surface elevates the light scattering, and the collected light intensity decreases. The DRIFT spectrum increases enormously not because of the absorption increase. (Details are written in Tolvaj and Mitsui 2004). This finding indicates that the DRIFT spectra cannot be used automatically as absorption spectra. The K-M units of a DRIFT spectrum are calculated by comparison of the reflected intensity data of the background material with that of the sample. High intensities in background spectrum generate high intensities in DRIFT spectrum of the sample too. That is why a proper, good quality background spectrum is important for getting high-quality DRIFT spectrum. The limits of K-M intensities defined in this study are only valid if the background is a matt aluminium plate.

Similar phenomenon occurs if the wooden surface is exposed to strong UV light for a long time. The DRIFT technique is usually applied for monitoring the chemical changes in this case using the K-M equation. The phenomenon is well demonstrated in Fig. 2. This figure represents the K-M functions of beech wood after 0, 12, 24 and 36 h of irradiation using a mercury lamp. The band assignment can be found in a previous work (Tolvaj and Faix 1995).

The main change is the overall intensity increase caused by UV irradiation. This increase is not proportionate with the irradiation time. This change cannot represent the absorption increase because the absorption increase is due to the creation of new

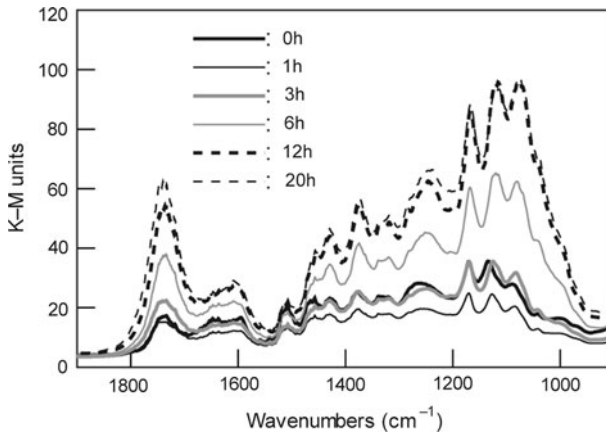


**Fig. 2** IR spectra (K-M function) of a beech sample irradiated with a mercury lamp for 0, 12, 24 and 36 h

chemical groups. The creation of new absorbing groups may happen only after degradation of other chemical compounds. This degradation should be visible as an absorption decrease; however, there is no absorption decrease visible in Fig. 2. The UV treatment increases the roughness of the sample surface and therefore changes the scattering properties. In consequence, less light reaches the detector, and the K-M values increase according to the K-M function (Fig. 1). This increase is enormously fast if the K-M units exceed 40. This phenomenon is visible in Fig. 2, especially in 3,100–3,600  $\text{cm}^{-1}$  region.

The poor reflection creates the automatic signal amplification of the spectrophotometer, which amplifies the noises as well. This is well visible in Fig. 2 in 3,100–3,600  $\text{cm}^{-1}$  region. This example represents clearly that the K-M theory cannot be applied automatically to determine light absorption of wood.

To define the limits of the K-M theory, more detailed treatments are needed. For this purpose, the treatment was interrupted after 1, 3, 6, 12 and 20 h of UV irradiation. The changes of a poplar sample are presented in Fig. 3. These are the measured data without baseline correction. Poplar was chosen because its K-M function was the most intensive one in the important fingerprint (900–1,900  $\text{cm}^{-1}$ ) region. The intensity increase is similar here to the one in Fig. 2. The change of K-M functions should represent both changes caused by alteration of scattering and absorption. Unfortunately, the change of absorption is not visible at all. The great changes by roughness alteration overlap the smaller changes of absorption. Fortunately, the scattering is not wavenumber dependent in a small wavenumber region in contrast to the absorption, which is characteristically wavenumber dependent. This fact gives the possibility to diminish the effect of scattering mathematically. For this purpose, an internal wavenumber point is needed where almost no absorption change is caused by the examined treatment. If the spectra are normalised to the unit at this wavenumber, this mathematical process can minimise the effect of scattering. Certainly, the change here is determined only by the

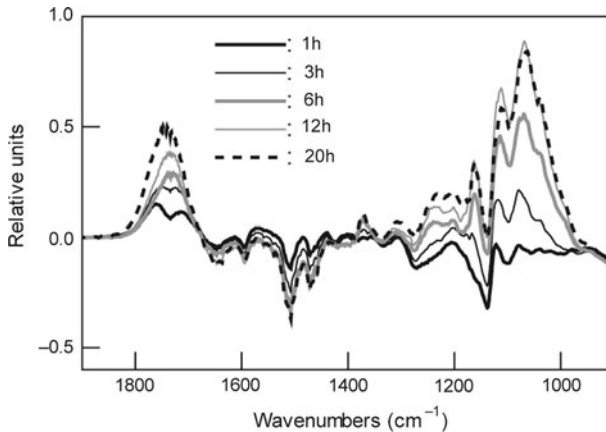


**Fig. 3** IR spectra (K-M functions) of a poplar sample irradiated with a mercury lamp for 0, 1, 3, 6, 12 and 20 h

scattering because there is no absorption change. Experiments on photodegradation confirm that the CH deformation in polysaccharides absorbing around  $1,380\text{ cm}^{-1}$  is intact during photodegradation (Tolvaj and Faix 1995; Forsskåhl and Janson 1995; Kataoka and Kiguchi 2001). So this peak intensity can be used as an internal standard in case of photodegradation. The mathematical process of normalising is that all K-M data are divided by the value at  $1,380\text{ cm}^{-1}$ , and the results are expressed in relative units and not in K-M units anymore.

The difference spectra were calculated after baseline correction and normalising the spectra at  $1,380\text{ cm}^{-1}$  (Tolvaj and Faix 1995). These difference spectra are presented in Fig. 4. After irradiation, the carbonyl band between  $1,680$  and  $1,850\text{ cm}^{-1}$  increased, and the peak of the aromatic skeletal vibration originating from lignin ( $1,510\text{ cm}^{-1}$ ) decreased together with the guaiacyl vibrations at  $1,275\text{ cm}^{-1}$  as has been noted in previous studies (Kosikova and Tolvaj 1998; Müller et al. 2003; Horn et al. 1994; Pandey and Theagarajan 1997; Ohkoshi 2002; Sudiyani et al. 2003; Mitsui and Tsuchikawa 2005). Usually two peaks develop in  $1,680$ – $1,850\text{ cm}^{-1}$  region during the exposure of wood to UV radiation. After irradiation for 1 h using a mercury lamp, the two bands are well separated; however, after 6 h, they have already merged to create one peak. The absorption decrease of guaiacyl vibrations at  $1,275\text{ cm}^{-1}$  is well demonstrated after 1 h of irradiation. However, this negative peak is lifted up because of the overall increase in  $950$ – $1,300\text{ cm}^{-1}$  region. More pronounced is the behaviour of the absorption decrease at  $1,140\text{ cm}^{-1}$ . This decrease originates from the splitting of ether bonds caused by UV irradiation. This large negative peak is also lifted so high that after 12 h of irradiation, no one can expect that this is a negative peak. It seems like a minimum between two positive peaks. However, it should be a negative peak if the abnormal overall increase has not lifted that part of the spectrum.

As shown in Fig. 3, the intensity of K-M function exceeds the 40 units level between  $1,040$  and  $1,280\text{ cm}^{-1}$  at 6 h of irradiation. At 12- and 20-h irradiations,

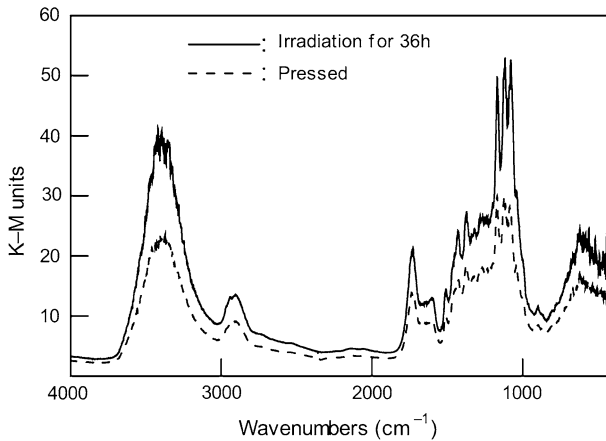


**Fig. 4** Difference IR spectra (K-M functions) of a poplar sample irradiated with a mercury lamp for 1, 3, 6, 12 and 20 h

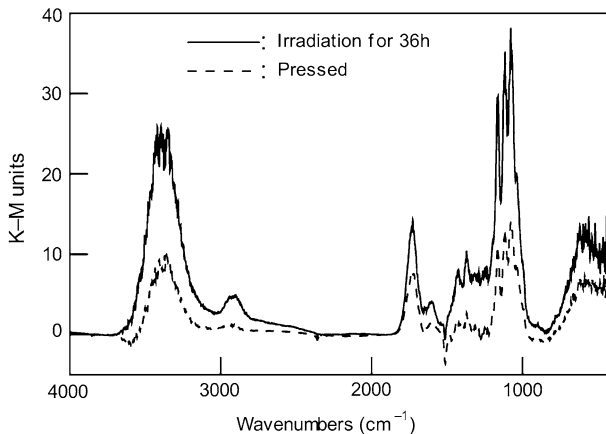
this range is between  $990$  and  $1,400\text{ cm}^{-1}$ , and the peak at  $1,740\text{ cm}^{-1}$  also overhangs this limit. The difference spectra (Fig. 4) do not show distortion in the  $1,300$ – $1,700\text{ cm}^{-1}$  interval, where the K-M intensities are below (or close to) 40 units. Here, only the changes caused by the photodegradation are visible. But if the K-M value considerably exceeds 40 units ( $1,000$ – $1,300\text{ cm}^{-1}$  interval), normalising, as a mathematical manipulation, is unable to correct the distortion caused by the K-M function (Fig. 4). Comparison of Fig. 3 with Fig. 4 demonstrates that normalising the spectra at an internal wavenumber can only partly reduce the distortion of the K-M function.

The surface roughness can be diminished by pressing the surface with a flat metal plate. While pressure reduces the roughness of the surface, this manipulation hardly changes the chemical bond system. Small alterations in the chemical bond system are not visible in the IR spectrum, but the decrease of roughness reduces the scattering. Thus, values of the K-M function can be diminished by this method, and these reduced values are able to satisfy the limits of the K-M theory. The effect of pressing is visible in Fig. 5. The treated Japanese cypress sample (after irradiation for 36 h with a mercury lamp) was pressed at a pressure of  $2.5 \times 10^6\text{ Pa}$  for 10 s with a metal plate in a pressure apparatus, and the sample was measured before and after pressing. It is visible that the pressure reduces the intensity values of the K-M function considerably. Figure 6 presents the difference spectra (created from baseline corrected spectra, normalisation was not applied) of cypress sample after 36 h of irradiation and after pressing this irradiated surface. Figure 6 demonstrates that pressing makes visible the absorption decrease of aromatic ring splitting at  $1,510\text{ cm}^{-1}$  and the decrease of the guaiacyl vibration at  $1,275\text{ cm}^{-1}$ , which are not visible on the difference spectrum before pressing. Moreover, the absorption decrease of the OH groups around  $3,620\text{ cm}^{-1}$  will be visible by pressing, as reported in previous works (Tolvaj and Faix 1995; Papp et al. 2005; Barta et al. 2005). Based on Figs. 5 and 6, it can be stated that pressing can reduce the





**Fig. 5** IR spectra (K-M functions) of a Japanese cypress sample (earlywood of sapwood) caused by 36 h of mercury lamp irradiation and the same after pressing at a pressure of  $2.5 \times 10^6$  Pa for 10 s



**Fig. 6** Difference IR spectra (K-M functions) of a Japanese cypress sample (earlywood of sapwood) caused by 36 h of mercury lamp irradiation and the same after pressing at a pressure of  $2.5 \times 10^6$  Pa for 10 s

scattering effect of the UV light irradiated surface of wood, but the changes caused by the photodegradation remain visible.

## Conclusion

After analysing plenty of DRIFT spectra of wood and examining the shape of the K-M equation (Fig. 1), it can be concluded that the measured K-M function can be used as an absorption spectrum if the values of the function are below 14 K-M units. Above this limit, the K-M theory, which was created for poorly absorbing materials,

does not indicate the absorption of wood properly. If a matt aluminium plate is used as a background material and the values are between 14 and 40, the absorption changes can be calculated after normalisation of the spectra. This normalising manipulation is only successful if there is an absorption peak close to the examined one which does not change its absorption during the examined treatment.

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