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CHARACTERISTICS OF AGING OF WOOD-FIBERBOARD FROM THE POSITION OF IR SPECTROSCOPY

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Fiber boards, like other wood-polymer composites, are widely used in construction. However, their service life is often less than that declared by the manufacturer, which is due to insufficient knowledge of the processes of degradation of polymer components of resin and wood filler under the influence of aggressive environmental factors. In this regard, the task is to reveal, using reflective IR spectroscopy, structural changes in the molecular structure of polymeric substances included in a wood fiber composite after heat aging, artificial UV radiation and exposure to direct sunlight. The results of the study showed that the IR spectra of all samples are identical, but differ in the intensity of individual absorption bands. This suggests that under the influence of aging factors, a free-radical rupture of hydrogen, hydrocarbon and ether bonds occurs in various functional groups of cellulose, hemicellulose, lignin and resin. At the same time, heat aging causes structural changes throughout the entire volume of the slab, and artificial UV irradiation destroys the surface layer about one millimeter thick. Sunlight during the summer season destroys the surface layer less than 0.5 mm thick and contributes to additional structuring of the polymer components of the resin and wood of the inner layers as a result of heating the board.

Key words: fibreboards, heat aging, UV irradiation, IR spectroscopy

INTRODUCTION

Currently, in low-rise construction, there is an increase in the consumption of wood-based panels based on polymer binders. Their relatively low cost, light weight and manufacturability can significantly reduce material and time costs in the construction of such buildings. Unfortunately, the service life of wood composites declared by manufacturers is often overestimated, which leads to a premature loss of performance of structures [1] to [3]. This is due to the fact that when assessing their service life, the susceptibility of the polymer binder to aging under the influence of external non-mechanical factors, which lead to irreversible structural changes, is not fully taken into account [4] to [6].

IR spectroscopy is a unique method for establishing the characteristics of the structure and properties of molecular compounds, determined by the nature and system of intra- and intermolecular interactions. The method is informative for the study of wood and materials based on it [7] to [9], therefore, the goal is to assess the effect of aging factors on the structural changes of an aged wood fiber composite using IR spectroscopy.

MATERIALS AND METHODS

As an object of research, we chose hard fiber boards (Fibreboard) with a thickness of 3.2 mm and a density of 950 kg/m^3, made by hot pressing in accordance with GOST 4598-86.

The experimental material accumulated to date and its analysis made it possible to identify the most aggressive types of action for polymer composite materials: temperature and solar radiation, especially its UV part [10], [11]. In this regard, in the work as the main factors of accelerated aging, UV irradiation created by a DRT1000 lamp with a radiant flux of 128 W in the wavelength range of 240-320 nm and an increased temperature of 80 ºC created in a thermal aging installation are taken. The duration of the factors was 500 hours.

To compare the results of laboratory tests with real operating conditions, a series of fiberboard samples were subjected to natural climatic aging in a temperate climate (under direct sunlight) during the months of June and August.

IR spectra were recorded using the ATR (disturbed total internal reflection) method on a JascoFT/IR-6200typeA FT-IR spectrometer with a spectral resolution of 4 cm^{-1} and a reflection angle of 45 degrees.

To assess the depth of development of degradation processes along the thickness of the slabs, the samples were removed layer by layer. The thickness of the layers was taken to be about 0.4 mm.

RESULTS

Today, most of the fibreboard is produced by hot pressing, in which stable chemical nodes (methylene and dimethylether bonds) and unstable physical nodes (hydrogen bonds between oxygen-containing groups - phenolic

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hydroxyls, methylol hydroxide groups and dimethylene ether bonds) are formed in the composite [12] to [14].

Wood fibers are characterized by an unfolded surface and have a significant amount of hydroxyl, carbonyl and aldehyde groups, which during hot pressing are combined with the same groups of neighboring wood fibers [15] to [17]. Thus, the interaction of wood fibers is carried out not only due to physical interweaving, but also at the chemical level [12].

At the same time, the phenol-formaldehyde binder interacts with the functional groups of wood fibers and hardens, forming hydrogen, simple and complex ether bonds [12].

Under the influence of elevated temperature and the UV part of sunlight, irreversible physicochemical transformations take place in the wood composite, which affects its operational properties

Thermal and photooxidation leads to disruption of the continuity of the surface layers of the material, increasing its roughness and porosity. The latter is also large enough in the internal volume of wood composites as a result of evaporation of moisture and volatile organic compounds when using the hot pressing method [10].

Air oxygen penetrates through the outer pores into the inner ones, thereby oxidizing the components of the wood composite from the inside. At the same time, atmospheric moisture contributes to the intensification of oxidation processes, increasing the mobility of macromolecular chains. In addition, an uneven distribution of stresses over the volume of the material is created, which leads to a violation of adhesion between wood fibers and a polymer binder [10].

Thermal and photooxidation occurs by a free radical mechanism. Under the influence of elevated temperature or high energy of the UV part of sunlight, a chain of chemical bond ruptures in the polymer composite is initiated. As a rule, ruptures begin near free hydrocarbon radicals, since the strength of the C–H bond is 7 times less than the strength of the skeletal C–C bonds in the polymer [10].

It should be noted that the short-wavelength limit of sunlight on the earth's surface ranges from 290 to 300 nm and has sufficient energy to destroy most chemical bonds (Table 1) [11].

Further, the formed free hydrocarbon radical is oxidized by atmospheric oxygen, turning into an unstable peroxide radical, which immediately abstracts a hydrogen atom from a neighboring polymer molecule by a radical mechanism [10], [11].

Thus, a hydroperoxide radical and a new hydrocarbon free radical are formed, which is again oxidized by oxygen and detaches a hydrogen atom from a neighboring polymer molecule, thereby continuing the chain of breaking bonds. At this time, the hydroperoxide radical also decomposes into two new free radicals [10], [11].

All reactions leading to the formation of free radicals are reversible, and many primary macroalkyl radicals are reunited intra- or intermolecularly, leading to branching and crosslinking. Sometimes this leads to a short-term improvement in the properties of the composite, but as a result, there is a decrease in molecular weight and complete destruction of polymer macromolecules [10], [11].

For example, the destruction of lignin always occurs through its condensation. First, new carbon is formed - carbon bonds with a sparse spatial network. Then, due to hydrogen bonds and peripheral groups of macromolecules, a three-dimensional structure is formed, which is subsequently destroyed by oxidized free radicals of neighboring molecular chains [10], [18].

Let us trace with the help of IR spectroscopy what changes in the structure occur in fibreboard under the influence of aging factors.

In the work of I. Kotlyarova described the IR spectrum characteristic of wood, in which several regions can be distinguished: the region of 3700–3100cm^{-1} characterizes the stretching vibrations of various types of hydroxyl groups involved in hydrogen bonds; area 3100-2750cm^{-1} – the region of symmetric and asymmetric stretching vibrations of CH groups of methyl, methylene, methine groups of wood composite components; area 1800-1000cm^{-1} (area of "fin

Table 1: The relationship between the wavelength of UV radiation from the Sun, photon energy and binding energy [11]

Communication	Communication energy, kJ/mol	Photon energy, kJ/mol	Wavelength, nm
C-C	955	1197	100
C=O	730,8	798	150
C=C	609	798	150
C=C (aromatic)	520,8	598,5	200
O-H	462	478	250
C-H (CH4)	411,8	478	250
C-O	365,4	478	250
C-O (ether)	331,8	399	300

gerprints") - the area of stretching vibrations of groups with multiple bonds (C=O, C=C, C_{Ar}-C_{Ar}), stretching vibrations of C-C and C-O bonds, as well as deformation vibrations of C-H bonds and OH groups of lignin and cellulose and hemicelluloses; area 1000-650cm^{-1} – region of out-of-plane deformation vibrations of C-H bonds of the aromatic ring of lignin, vibrations of the glucopyranose ring of cellulose and hemicelluloses, out-of-plane deformation vibrations of OH groups of cellulose and hemicelluloses [19] to [21]. Based on the above description, let us consider the IR spectra of the surface of plates that are not subject to aging (Figure 1).

The IR spectrum of a wood-fiber composite is rather complicated, since it is a set of absorption bands not only for wood fiber, but also for a phenol-formaldehyde binder, despite the fact that its content in the board is low [22] to [24].

The situation is further complicated by the fact that the wood fiber itself is a complex ligno-carbohydrate complex; therefore, absorption bands of cellulose, hemicellulose and lignin are superimposed in the IR spectra. At the same time, domestic and foreign scientists consider the following stripes characteristic of wood plastics 3750, 3330, 2920, 2850, 1727, 1595, 1509, 1267, 1160, 1105, 1053, 1025cm^{-1} [25] to [27].

In the range 3800-3100cm^{-1} with a maximum in a strip of 3330cm^{-1} there is a broad band of stretching vibrations of free and bound hydroxyl OH-groups of cellulose involved in intermolecular and intramolecular hydrogen bonds (Figure 1) [21], [28], [29].

As a rule, after hot pressing in the surface layer of the boards, the content of free OH groups is insignificant in comparison with the inner layers, since wood particles are coated with resin [15]. This is evidenced by the absence of a pronounced band at 3750 cm^{-1} for the initial composite. The action of aging factors leads to the destruction of the surface resin film, exposure of wood particles and the release of hydrogen-bonded OH-groups of cellulose, as a result of which a whole series of rather intense bands is observed in the wavenumber region of 3750cm^{-1}. In this case, a decrease in the intensity of the band at 3330cm^{-1} (Figure 1).

In the interval 3000-2600cm^{-1} two bands of medium intensity are observed with maxima at 2920cm^{-1} and 2850 cm^{-1}, corresponding to stretching vibrations of methine and methylene –CH2 and –CH groups of cellulose, lignin and phenol-formaldehyde resin [30] to [32].

Under the influence of aging factors, the maximums of these bands do not shift, but their intensity decreases as a result of the breaking of hydrocarbon CH bonds (Figure 1). In this case, photooxidation has a more negative effect.

In the range of 1800-1580cm^{-1} For the initial material, bands are observed that are characteristic of stretching vibrations of C=O bonds in carbonyl and C-O bonds in the carboxyl groups of the ligno-carbohydrate complex of the wood filler (Figure 1). There is also a 1595cm^{-1} strip, corresponding to stretching vibrations of double C=C benzene and aromatic bonds of lignin [18], [28], [30].

As a result of thermal and photooxidative processes,

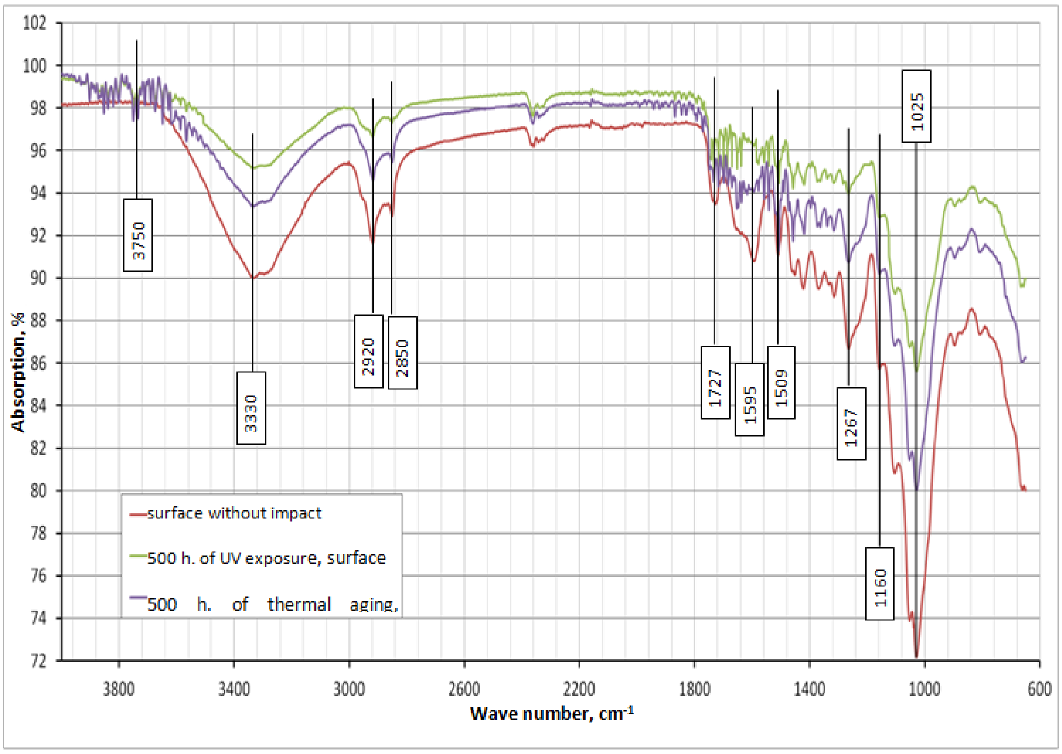

Figure 1: IR spectra of the surface of fiberboard samples, initial and exposed to UV irradiation and thermal aging for 500 hours

an increase in the number of carbonyl groups occurs, which leads to the appearance of a series of bands of low intensity (Figure 1). Decrease in the intensity of the 1727cm^{-1} band indicates the degradation of chemical C-O-C bonds in the molecular structure of cellulose and hemicellulose. At the same time, the band 1595cm^{-1}, indicates the degradation of chemical C-O-C bonds in the molecular structure of cellulose and hemicellulose. At the same time, the band 1595^{-1} for all samples. This is the interval of deformation vibrations of groups CH_3, CH_2 и O-H and aromatic skeletal vibrations of lignin (1509cm^{-1}) [28], [31], [32]. It is worth paying attention to the stripe 1267cm^{-1}, the intensity of which under the influence of UV irradiation decreases more strongly than after heat aging. This band characterizes the skeletal vibrations of the guaiacyl ring of lignin, which is part of wood fibers [23]. The decrease in the intensity of the band is apparently associated with the rupture of the C-O bonds in the ring as a result of oxidation.

In the range 1200-1000cm^{-1} for all samples can be see “fingerprinted”; i.e the most typical for cellulosic materials four intense bands with maxima at 1160, 1105.1053 and 1025cm^{-1}. These bands are due to the presence of acetal C–O–C and C–O bonds in alcohols, ethers, and polysaccharides [19], [21], [32].

Decrease in the intensity of the defining band 1025cm^{-1} under the influence of aging factors is associated with the weakening of carbon bonds in the C-C and C-H groups as a result of the oxidation of lignin and hemicellulose, which are more reactive than cellulose and therefore more susceptible to degradation [10], [20], [28].

To assess the depth of development of degradation processes along the thickness of the slabs, the samples were removed layer by layer. The thickness of the layers turned out to be about 0.4 mm. The IR spectra of individual layers of boards subject to aging are shown in Figure 2.

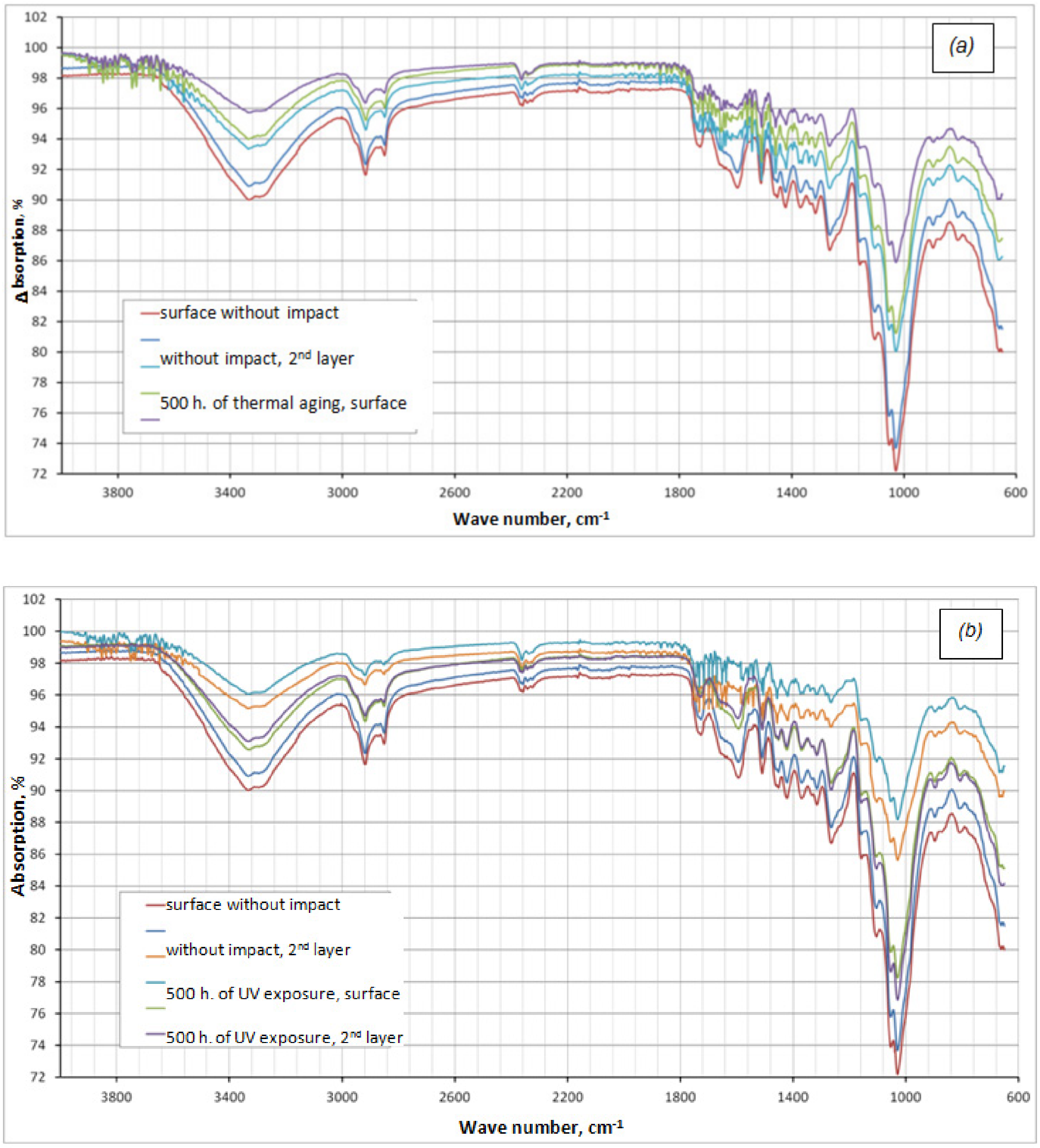

Figure 2: IR spectra of the surface and inner layers of fiberboard, initial and subject to thermal aging (a) and UV irradiation with a DRT1000 lamp (b)

It is important to note that UV irradiation and thermal aging do not cause the absorption bands of the inner layers of the composite to shift either to the region of low or high vibration frequencies. The difference is that heat aging initiates destructive processes in the composite at a depth of more than 1mm (Figure 2a), and it can be safely assumed that the destruction of bonds by a free radical mechanism occurs throughout the entire volume of the slab.

UV irradiation from an artificial light source affects the composite layer with a thickness of less than 0.8mm, since the behavior of the IR spectra for layers 3 and 4 completely coincides with the behavior of the spectrum of the composite that is not subject to aging (Figure 2b).

Aging in direct sunlight causes similar changes in the structure of the composite (Figure 3). The IR spectrum of the irradiated surface generally coincides with the rest of the spectra, but differs in the degree of change in the intensity of the bands.

It can be seen that the UV part of sunlight promotes the release of hydroxyl groups as a result of breaking hydrogen bonds, which leads to the appearance of a band at 3750cm^{-1} and a decrease in the intensity of the band 3330cm^{-1}.

As in the case of an artificial source of UV radiation for the surface layer of fiberboard, a significant decrease in the intensity of two bands at 2920cm^{-1} and 2850cm^{-1} as a result of the destruction of CH bonds connection.

The low resistance of lignin to solar radiation leads to the destruction of aromatic skeletal bonds and skeletal bonds of the guaiacyl ring in its polymer macromolecules, which leads to the disappearance of the absorption bands at 1509 and 1267cm^{-1} respectively. Almost complete absence of the 1462cm^{-1} band indicates a violation of bonds in the etheric functional group OCH3, which is present in hemicelluloses [21].

The destruction of chemical bonds in the hydrocarbon part of wood fibers under the action of solar radiation is confirmed by a decrease in the intensity of the characteristic band at 1025cm^{-1}.

An increase in the number of carboxyl groups as a result of photooxidation of polymer resin, lignin and hemicellulose leads to a decrease in the intensity of bands in the range 1800-1580cm^{-1}. However, in contrast to an artificial radiation source, in this case, the appearance of an additional large number of low-intensity bands is not observed, which is possibly associated with the occurrence of condensation processes from heating the composite under mild conditions under sunlight.

If the composite is irradiated by sunlight, the thickness of the layer damaged by photooxidative processes is less than 0.4mm. The observed increase in the intensity of the bands under consideration for layer 2, i.e. at a depth of more than 0.4 mm, apparently, is associated with additional structuring of polymer macromolecules of the resin and components of wood fibers as a result of heating the composite.

CONCLUSIONS

The study shows that IR spectroscopy is an informative method that allows you to identify changes in the molecular structure of fiberboards caused by thermal and photooxidation processes. Prolonged action of elevated temperature and UV irradiation initiates free-radical reactions of breaking hydrogen and hydrocarbon bonds in

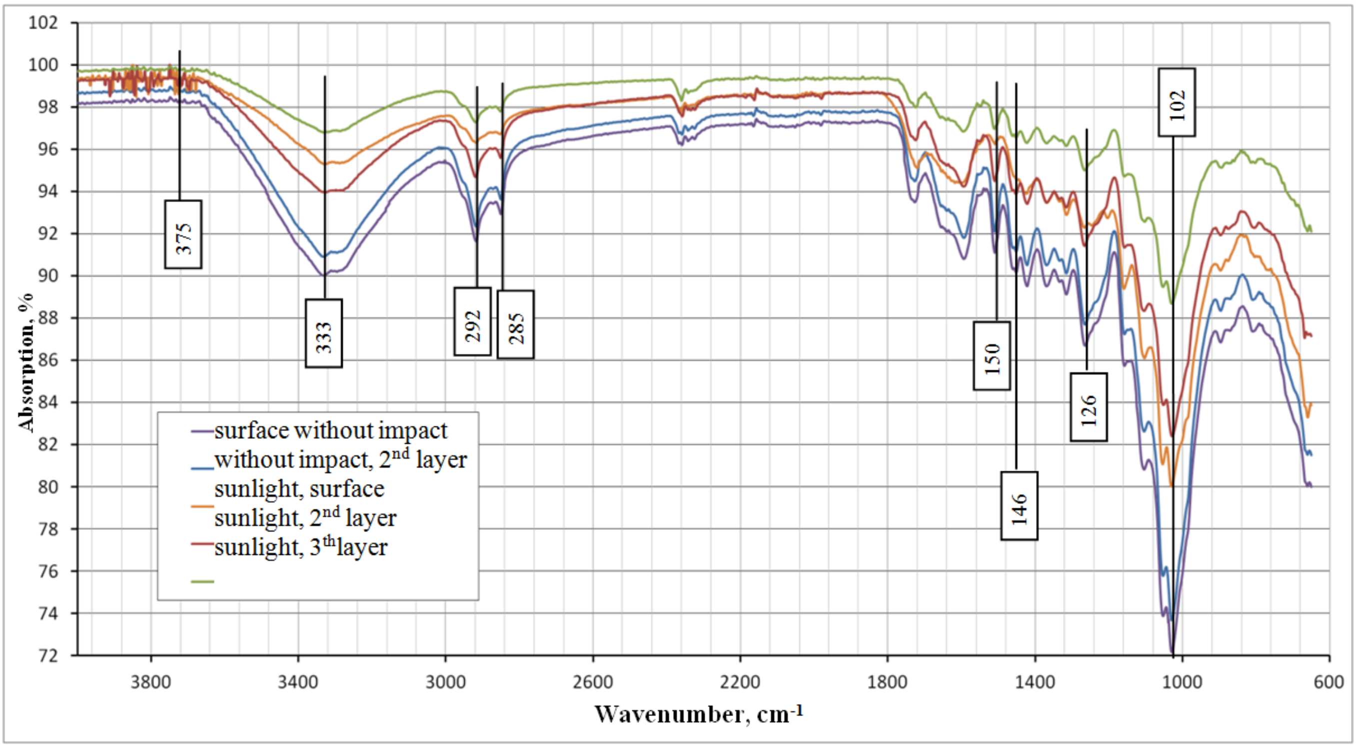

Figure 3: IR spectra of the surface and inner layers of fiberboard exposed to sunlight during June-August

various functional groups of macromolecules, which is confirmed by a change in the intensity of the corresponding absorption bands.

Artificial UV irradiation degrades the material at a depth of about one millimeter. Under the influence of sunlight, not only the destruction of the surface layer less than 0.5mm thick occurs, but also the heating of the material, which contributes to the additional structuring of the polymer components of the inner layers. In the case of heat aging, destructive changes are observed throughout the entire volume of the composite.

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