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ORIGINAL ARTICLE – Forest Products Science and Technology

Research of the Properties of Plywood Based on Urea-Formaldehyde Binder with the Added Multi-Wall Carbon Nanotubes

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Abstract

The work proposes the use of multi-walled carbon nanotubes in various concentrations treated in nitric acid (f-MWCNT) as a filler for urea-formaldehyde resin to produce birch plywood with improved physical-mechanical, water-resistant and thermal properties. The MWCNTs were characterized using transmission electron microscopy, IR and Raman spectroscopy, and the zeta potential was determined. The thermal stability of plywood on the UFR/f-MWCNT adhesive composition was assessed and a higher thermal stability of plywood was revealed when a nanofiller was added to the resin. The modulus of rupture increased by 62.7% and the modulus of elasticity by 113% with the addition of 1.5% f-MWCNT. A decrease in water absorption was established by 43%. A steady decrease in the emission of free formaldehyde from plywood was determined. The possibility of intermolecular interaction between the functional groups of f-MWCNT and urea-formaldehyde resins has been established.

Keywords: multi-walled carbon nanotubes, plywood, urea-formaldehyde resin, strength, heat resistance, formaldehyde emissions.

1. INTRODUCTION

Wood is a material with a complex anisotropic structure due to the structural characteristics of its cell walls. The cell wall layers in wood differ depending on the direction of the cellulose microfibrils. The peculiarities of wood's structure determine the different nature of wear on components and structures made from natural wood, and have a significant impact on limiting wood operating conditions. (Zhao et al., 2023). An alternative to natural wood can be wood panel materials, characterized by optimal physical and mechanical properties and lower anisotropy (Castanié et al., 2024; Pramreiter et al., 2023; Wang, Y. et al., 2023), as well as economic attractiveness, availability of raw materials associated with the production of fast-growing species (Rohumaa et al., 2021; Wang, T. et al., 2022) or waste from wood processing industries from inexpensive wood (Mirski et al., 2020; Pędzik et al., 2021)

Plywood is one of the most common panel materials with an annual production volume of 170 million m^3 in the world, 3% of which is manufactured in the Russian Federation (Zykov & Dayneko, 2022). The use of plywood as a structural material is justified by the possibility of changes in its manufacturing technology, allowing one to predict the necessary properties of the resulting material.

The properties of the binder play a primary role in the quality of the adhesive joint of plywood and, as a result, largely determine the physical and mechanical characteristics of the resulting product. A number of studies have been conducted aimed at comprehensively improving the binder through the introduction of modifying additives or its physical treatments (Gonçalves et al., 2022; Sanghvi et al., 2022; Xu et al., 2023).

To obtain plywood with improved physical and mechanical properties, researchers have proposed the use of an alternative plastic-based binder, for example, virgin or recycled polyethylene films (Ashori et al., 2023), as well as polypropylene (Arya et al., 2022), heat-treated veneer (Hsu et al., 2023), and reinforcement of plywood with copper (Sankar & Sajan, 2021), carbon (Auriga et al., 2020), and basalt fibers (Mayer et al., 2024).

The use of urea-formaldehyde resins, although widespread in the wood panel industry, release of formaldehyde from the final products is one of the most important health and environmental concerns in this industry. This appears to be primarily due to the emission of free formaldehyde as a result of the cleavage of ether bonds and terminal groups of methylol.

Currently, special attention is being paid to studying the possibility of using nanomaterials as binder modifiers due to their unique properties: high resistance to deformation, high functional density, lack of point defects, and catalytic activity, which helps to increase the reactivity of nanoparticles with their environment. (Iftikhar et al., 2023; Karkush et al., 2023). Research has been conducted on the use of graphene nanoplatelets as a resin modifier, resulting in a heat-stable and fire-resistant plywood with increased electrical conductivity (Li, J. et al., 2023; Łukawski et al., 2024).

In some studies (Dorieh Selakjani et al., 2022), the authors reported the use of multi-walled carbon nanotubes to improve the properties of binders in wood-polymer composites. For example, MWCNTs have been used to modify urea-formaldehyde resin and produce fiberboards (Gul et al., 2020; Kumar et al., 2015a, 2017), with increased strength and thermal stability and reduced free formaldehyde emissions (Li, C. et al., 2020; Mazaheri et al., 2022).

An increase in the strength characteristics of wood composites was established when a small amount of MWCNT was added to the binder. (ŁUKAWSKI et al., 2019). As a rule, MWCNTs obtained industrially have insignificant reactivity. In most cases, an increase in the chemical activity of the MWCNT surface occurs due to the preliminary functionalization of carbon nanotubes (Chen & Cheng, 2022). One of the effective methods for modifying the surface of MWCNTs is treatment with concentrated acids to form chemically active functional groups such as carboxyl and carbonyl groups on the surface. (Lykidis, 2023)

This study investigates a complex binder based on urea-formaldehyde resin (UFR) with the addition of nitric acid-activated multi-walled carbon nanotubes (f-MWCNTs) as a modifier for the production of plywood composite with improved physical, mechanical and environmental performance.

Thus, the purpose of this work was to study the role of nanoadditives in the form of functionalized multi-walled carbon nanotubes introduced into a urea-formaldehyde binder on the physical, mechanical, thermal and environmental properties of the resulting plywood composite.

2. MATERIALS AND METHODS

2.1. Materials

For the experiments, multi-walled carbon nanotubes (MWCNT) were used, which were purchased from MST-Nano (Riga, Latvia). Urea-formaldehyde resin (UFR) with a molar ratio of carbamide to formaldehyde of 1:1.8 was purchased from RossPolymer LLC (Moscow, Russia). The mass fraction of dry matter in the resin is 68.2%. The pH is 7.2 and the viscosity is 170 sP. Nitric acid with a concentration of 57% was used for acid modification. Distilled double-purified water (Milli-Q) was used in all experiments. NH_4Cl was used as a hardener.

2.2. Analysis and characterization of MWCNTs

The dimensional characteristics and morphology of MWCNTs were obtained using a transmission electron microscope with an Auger electron accelerating voltage of 2 kV (Carl Zeiss Libra 120, Germany). To determine the size and stability of MWCNTs in an aqueous solution, studies were carried out on dispersed phase particles directly in the solution using the zeta potential (ζ) method. The study was conducted on a Zetasizer Nano ZSP particle size analyser (Great Britain, Malvern Instruments Ltd.), using the Doppler frequency shift method for scattered light, when an external electric field was applied to the sample. The solutions were analyzed after settling for 20 days at a temperature of 25 °C, the sample volume was 50 μl using a helium-neon laser with a wavelength of 633 nm and a power of 10 mW. The sizes of the nanotubes were determined by repeating each test three times and averaging the results. Fourier transform IR spectroscopic analysis was carried out on a VERTEX 70 spectrometer (Bruker, Germany) using the attenuated total internal reflection method using a diamond prism in the frequency range of 400 to 4000 cm^{-1} with a resolution of 2 cm^{-1} in transmission mode. The Raman spectrum was obtained using the INTEGRA Spectra scanning probe micro-Raman spectroscopic complex (NT-MDT, Russia), at an exciting laser wavelength of 532 nm. The samples for IR spectroscopy were in liquid form, pre-treated under ultrasound as described above.

To study the changes in mass and thermal effects, a thermogravimetric analysis was conducted in nitrogen gas using an analyzer (Netzsch STA449F3, manufactured in Germany) at a heating rate of 10 °C/min up to a temperature of 1000 °C. The samples for thermal analysis were cut out objects weighing 10 g.

2.3. MWCNT functionalization and production of urea-formaldehyde resin/MWCNT composites

To form carboxylic acid groups, approximately 8.0 grams of MWCNT were added to two moles of nitric acid and heated for 5 hours at 60 °C with constant stirring using a magnetic stirrer. (Hasany et al., 2013). The mixture was heated to 25°C while removing excess nitric acid and washing the MWCNTs with distilled water to obtain a neutral pH. Next, the mixture was filtered and dried in an oven set at 65°C for 12 hours.

To obtain an adhesive composition of urea-formaldehyde resin / f-MWCNTs, mix the components on a magnetic stirrer with a rotation speed of 2500 rpm for 30 minutes with the addition of the following concentrations of f-MWCNTs: 0.5; 1.0; 1.5; 2.0; 4.0. To harden the adhesive mixture, 1% ammonium chloride was added from the total mass of the composition. Ultrasonic dispersion of the adhesive composition with constant cooling was carried out on an ultrasonic dispersant (homogenizer) Sonicator Q500 for 30 minutes with a power of 100 W and a frequency of 22 kHz in a resin-filler system (Figure 1).

To measure gelation time, 5 g of resin was placed in a glass tube in the presence of 1 mass., % solid NH_4Cl and reacted to a high viscosity gel at 100 °C. The onset of gelation was recorded by the loss of fluidity of the adhesive system. The viscosity of the resin containing different amounts of filler was determined in accordance with the state standard of the Russian Federation No. 14231 for samples of UFR and adhesive systems based on them, the conditional viscosity at 20 °C is determined using a VZ-4 viscometer, where 4 is the nozzle diameter, mm. (Tsvetkov et al., 2023).

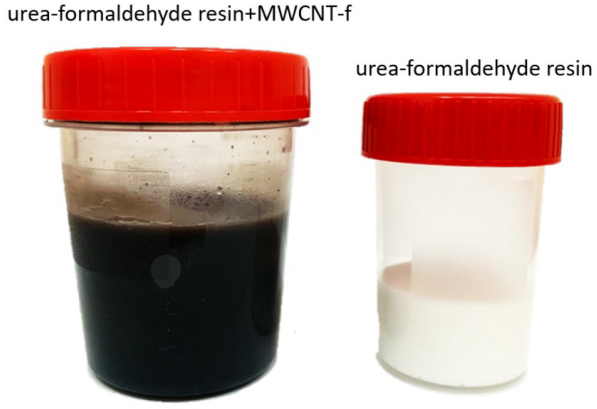

Figure 1. Urea-Formaldehyde Resin Before and After Adding MWCNT-f (2%).

2.4. Preparation and study of the properties of plywood using UFR/f-MWCNTs

Plywood is obtained from birch wood veneer, cut using a peeling method, in the following dimensions: 1.5-2.0 mm thickness; 400 mm length; 400 mm width. Veneer sheets for plywood production are dried to a moisture content of 4-5%. Plywood is produced from three sheets of birch veneer using hot pressing. The adhesive composition was applied to two sheets using a brush, with an adhesive consumption of 0.17 kg/m². For pressing, a D2430B press (GIDROPRESS LLC, Orenburg, Russia), with a capacity of 100 kN, supported by electric heating, was used. Pressing was carried out at a temperature of 135±5 °C, and a pressure of 1.25 MPa, pressing time was 12 minutes.

Samples for studying the physical and mechanical properties (modulus of rupture MOR and modulus of elasticity MOE) of plywood were obtained using a Metabo saw in accordance with IS 1734 (Karri et al., 2022). To test the shear strength of the adhesive joint, the samples were glued together in an overlap in accordance with the state standard of the Russian Federation № 3916.1. The strength of the adhesive joint was determined before and after soaking for 24 hours in distilled water. To determine water absorption, the samples were immersed in water under a desiccator insert at a constant temperature of 20 °C for 24 hours. At least 12 samples were used to determine the physical and mechanical tests. To determine water absorption (WA) we used set ratios (Zhuzhukin et al., 2023).

Formaldehyde emission from plywood was determined by the desiccator method in accordance with (Yu et al., 2018). The amount of formaldehyde released was obtained from the concentration of formaldehyde absorbed by distilled water, measured after a certain time interval at a controlled temperature. To do this, samples measuring 150x50 mm in an amount of 10 pieces were placed in a desiccator with a volume of 10 liters. Next, 20 desiccators with the test samples were placed in drying ovens at 40 °C for 1, 7, 14, 21 and 28 days. A Petri dish filled with 300 ml of distilled water was placed at the bottom of the desiccator, which was used to absorb the released formaldehyde. The amount of formaldehyde emission was determined on a PE-5400VI spectrophotometer (Russia, Moscow) at a wavelength of 412 nm. The principle of determining the concentration of formaldehyde absorbed in distilled water or ion exchange water is based on the Hantzsch reaction, in which formaldehyde reacts with ammonium ions and acetylacetone to form diacetyldihydrolutidine. In the process of determining formaldehyde emission, at each of the control time intervals (1, 7, 14, 21 and 28 days), 4 desiccators

with 10 samples were opened and formaldehyde emission was determined, the determination error did not exceed 10%.

2.5. Statistical analysis

The results of determining the physical and mechanical properties of plywood were statistically analyzed using R Studio. Comparison of the indicators was carried out using the Student's test with a significant level of 0.05.

3. RESULTS AND DISCUSSION

3.1. Characteristics of f-MWCNTs

To determine the dimensional characteristics of MWCNT, morphological studies were carried out using transmission and scanning electron microscopes, presented in Figure 2.

TEM analysis showed that the sample contains multi-walled carbon nanotubes with an average outer diameter of about 10-30 nm and an inner diameter of 5-15 nm. Nanotubes are cylindrical structures consisting of several layers of carbon atoms intertwined and forming concentric circles. The end caps of the nanotubes are closed, meaning that they are connected to each other and form a continuous network. The length of nanotubes is approximately 2 microns.

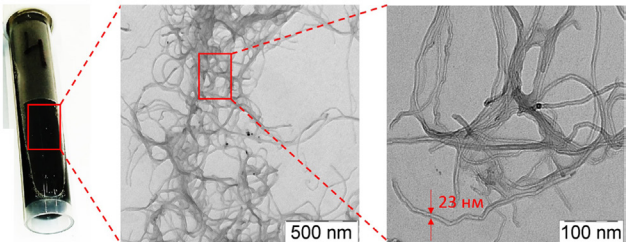

Figure 2. Microphotographs of Multi-Walled Carbon Nanotubes.

Table 1. Characteristics of the used MWCNTs.

An object	Outer diameter, nm	Inner diameter, nm	Length, μm	Specific surface area, m2/g	Distance between layers, nm
MWCNT	10–30	5-15	≥2	≥270	0,34

Energy dispersive analysis was conducted to determine the elemental composition of MWCNT. The results are presented in Figure 3.

As a result of energy dispersive analysis, it was established that the carbon content in the samples was the largest amount - 97%, all other elements were impurities formed during the production of MWCNTs, their percentage content did not exceed 1.5%. Raman spectroscopy made it possible to establish the presence of a double-humped peak (D and G) in the frequency range 1200-1600 cm^{-1}, as well as the presence of a band at 2730 cm^{-1} when considering the Raman spectrum (Figure 3b). The G band is located at ~1580 cm^{-1} and is characteristic of C–C bond stretching vibrations. In addition, the disorder bands (D and G') are installed. The results obtained are consistent with the data from other researchers (Golubewa et al., 2021).

The change in functional groups as a result of the functionalization of MWCNTs with nitric acid in the frequency range 400-4000 cm^{-1} was determined by IR spectroscopy (Figure 4a). The results are presented in Figure 4b). The thermal degradation process of MWCNTs before and after functionalization in nitric acid is presented in Figure 4c.

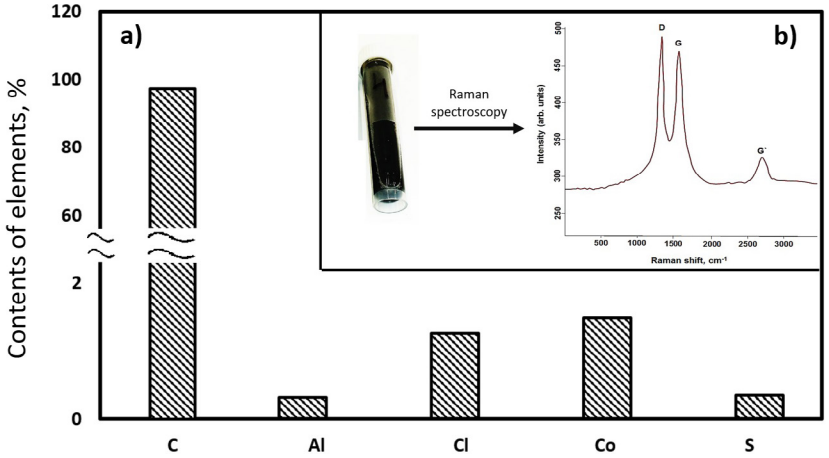

Figure 3. Results of elemental analysis (a) and Raman spectroscopy of MWCNTs.

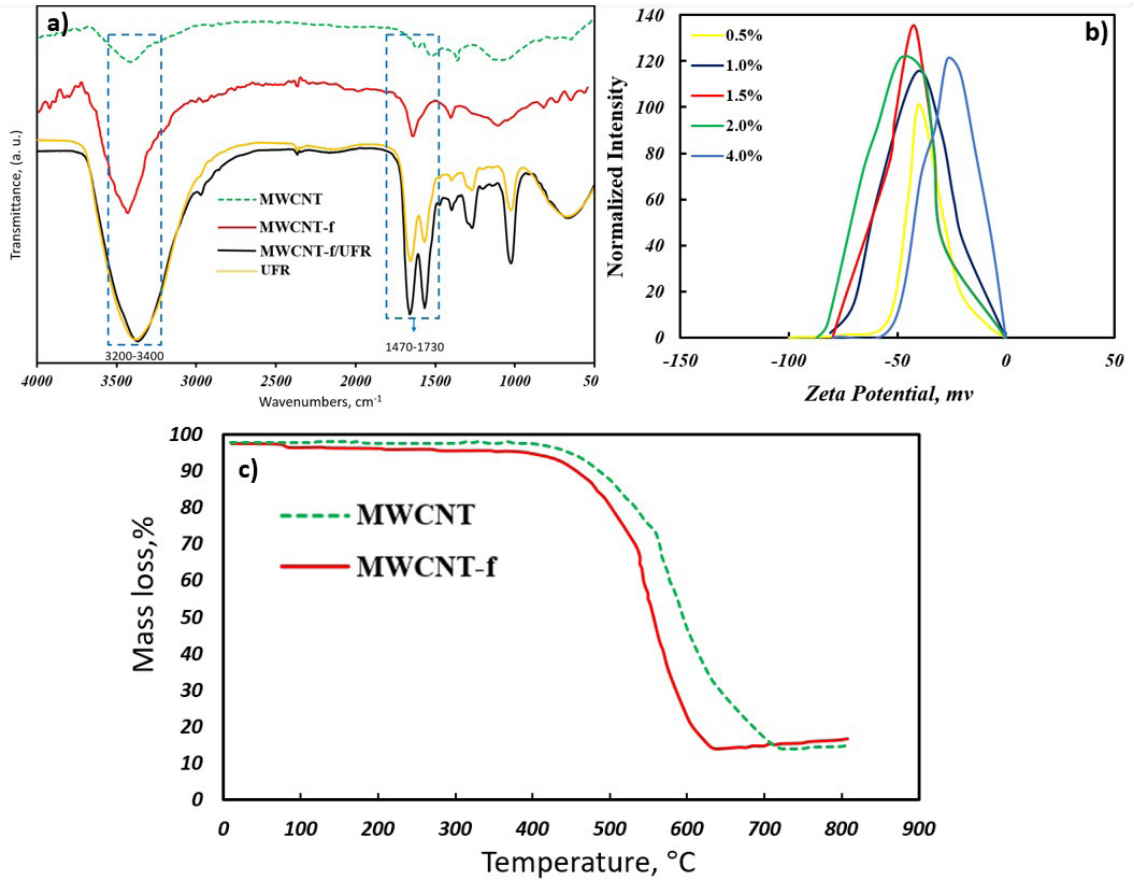

Figure 4. Results of IR spectroscopic analysis of MWCNTs and f-MWCNTs (a), determination of zeta potential (b), and thermogravimetric analysis (c).

The IR spectrum of the UFR curve revealed the presence of a broad absorption band in the region of 3366 cm^{-1}, corresponding to the stretching vibrations of hydroxyl groups (-OH) associated with the existing hydrogen bonds in the UFR. Intense signals of the bands at 1652 and 1558 cm^{-1} correspond to amides in the region of carbonyl absorption. The second amide band at 1558 cm^{-1} is associated with bending vibrations of N-H. The small band at 1259 cm^{-1} is associated with bending vibrations of free hydroxyl groups, which are included in the hydroxymethyl group (CH_2OH) UFR. The band at 1013 cm^{-1} is attributed to the stretching vibration of the ether bond (C-O), an integral part of the dimethylene ether bond in the resin.

When examining the curves of the IR spectra of MWCNTs and f-MWCNTs, the presence of a band at 3300-3400 cm^{-1} was established, caused by stretching vibrations of the OH group involved in intermolecular hydrogen bonding, the intensity of which is significantly enhanced in the IR spectrum of f-MWCNTs in comparison with non-activated MWCNTs. When f-MWCNTs are added to urea-formaldehyde resin, the intensity of the band at 3300 cm^{-1} remains virtually unchanged, but it shifts towards lower frequencies. This may indicate the formation of a hydrogen bond between the OH groups in f-MWCNT and the resin. In the IR spectrum of f-MWCNTs after treatment, a band at 1700 cm^{-1} appears, characteristic of vibrations of the carbonyl group C=O. The intensities of the bands at 1652 and 1558 cm^{-1} and especially the band at 1013 cm^{-1} decreased due to the possible intermolecular interaction of the functional groups of the resin (C=C, C-N, N-H) and nanotubes (C=O).

Using the zeta potential method for studied samples (0.5, 1.0, 1.5 and 2.0), a high resistance of the composition against aggregation ($\zeta > \pm 30$ mV was established). Figure 4c shows thermal decomposition process for MWCNT and f-MWCNT. Up to 400 °C, degradation of f-MWCNT is practically indistinguishable from MWCNT. As temperature increases, degradation becomes more significant reaching a maximum at 600 °C, then degradation of both f-MWCNT and MWCNT practically stops at temperatures up to 800 °C.

Table 2 presents the results of determining the gelation time and relative viscosity of UFR and UFR/f-MWCNTs.

Table 2. Results of determination of viscosity and gelation time of UFR/f-MWCNTs at 100 °C

Composition	Gel formation time, s	Conditional viscosity
UFR	53	44
UFR/f-MWCNTs (0.5%)	57	46
UFR/f-MWCNTs (1.0%)	60	48
UFR/f-MWCNTs (1.5%)	63	51
UFR/f-MWCNTs (2.0%)	67	54
UFR/f-MWCNTs (4.0%)	74	59

As a result of assessing the gelation time and conditional viscosity of the samples, a linear dependence of the increase in the studied parameters on the amount of f-MWCNTs in the solution was established. The higher viscosity of the resin containing f-MWCNTs was presumably a consequence of increased chemical reactivity and the formation of intermolecular bonds between the resin and f-MWCNTs Figure 4 c. This is confirmed by data from other researchers (Kawalerczyk et al., 2021; Kumar et al., 2015b).

3.2. Study of the properties of MWCNT-f/URF based plywood

3.2.1. Results of the study of strength and water resistance indicators

Figure 5 shows the results of determining the water resistance of plywood (water absorption for 2 and 24 hours), obtained with the addition of f-MWCNT in various percentage concentrations (0.5% - 4%) and without f-MWCNT, as well as the results of strength indicators: modulus of rupture (MOR), modulus of elasticity (MOE) and shear strength test.

The shear strength of the adhesive joint for the control sample was 0.84 and 0.66 MPa before and after soaking in water, respectively (Figure 5a). The maximum value for this indicator was observed with the addition of 1.5% MWCNT-f, both before and after soaking in water. The adhesive lines exhibited lower strength after soaking in water, presumably due to hydrolysis of the methylene bonds in the UV resin in the presence of water. During the study of water absorption, a stable dependence of the decrease in this indicator with increasing concentration of f-MWCNT was observed. For control samples, the water absorption value was 24.62% and 43.11% after 2 and 24 hours in a desiccator, respectively. Statistically significant differences were established between control samples for concentrations of 1.5%, 2.0% and 4.0%. With increasing concentration of f-MWCNT, water absorption decreased by 22% when kept for 2 hours (4% f-MWCNT). After 24 hours of aging, water absorption for samples with 1.5% filler decreased by 9%, 16% and 18% when adding 2.0% and 4.0% f-MWCNT, respectively. To establish the influence of the amount of filler, the physical-mechanical (MOE, MOR) properties of plywood were determined (Figure 5 c,d).

Multi-walled carbon nanotubes are a proven reinforcing filler in many polymer composite materials (Kandhola et al., 2023). The results obtained for the MOR and MOE indicators show a pronounced parabolic dependence with an extremum at 1.5% f-MWCNT in the composite. The modulus of rupture for a given amount of filler has increased by 62.7% and MOE has increased by 113%. It should be noted that, with a further increase in the amount of filler, a decrease was observed in physical and mechanical properties, which may be due to the presence of microvoids in the adhesive joint, due to the low distribution of f-MWCNTs in the adhesive.

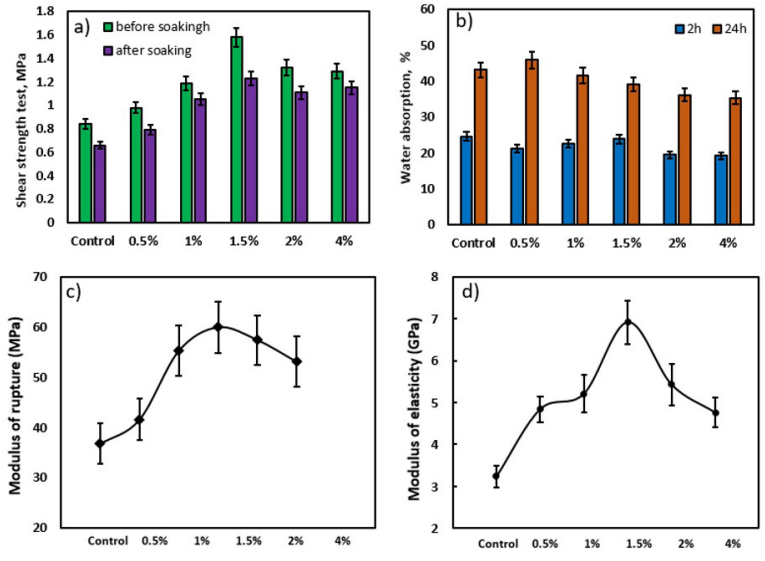

Figure 5. Results of the determination of shear strength test (a), water absorption (b), modulus of rupture (c), and modulus of elasticity (d).

3.2.2. Results of thermogravimetric analysis of plywood with MWCNT and f-MWCNT

Figure 6 shows the thermogravimetric curves and their derivatives (TG/DTG) for urea-formaldehyde resin, with and without the addition of f-MWCNTs (a, b) and for plywood obtained by gluing with UFR and UFR + f-MWCNT (c, d), at a heating rate of 10 °C min^{-1}. Thermal analysis was carried out using f-MWCNT, added in an amount of 1.5%, due to the best physical and mechanical properties of the plywood for the given amount of filler. (Figure 6).

Thermal degradation of urea-formaldehyde resin typically occurs in three major stages (Natarelli et al., 2019). The first stage of thermal decomposition is mass loss when drying samples in the temperature range from 33 to 104 ˚C, the percentage of mass loss for this period was 4.63% and 4.84% for UFR and MWCNT-f/UFR (Figure 5ab), significant differences in At the first stage, no difference was established between samples with nanoadditives. The second stage of thermal destruction occurs in the temperature range of 160-400 ˚C, in this range the most significant weight loss occurs in the samples. For UFR, there is an exothermic peak observed at a temperature of 171 °C as a result of the destruction of dimethylene ether bridges ($–CH_2–O–CH_2–$) and the formation of formaldehyde and more stable methylene ($–CH_2–$) bridges (Esmaeili et al., 2017). The main percentage of weight loss was observed in the temperature range of 160-400 ˚C, which is justified by the thermal destruction of aminomethyl bonds in the resin (decomposes at 230˚C) (Samaržija-Jovanović et al., 2011), leading to rapid weight loss at 247 ˚C (exothermic peak figure 6b). Further significant loss of resin mass at this stage is caused by the decomposition of resin polymers with the formation of a large number of volatile components (Ding et al., 2022). When examining the Plywood/UFR curve, a double-humped exothermic peak is observed, resulting from the process of thermal decomposition of the structural components of the wood cell wall, leading to a total mass loss of up to 24% at 370˚C. An exothermic peak appears on the Plywood/UFR-MWCNT-f curve with a maximum at 370˚C, which indirectly characterizes the greater thermal stability of this plywood sample and is confirmed by the TG data shown in Figure 5c: the process of intense thermal degradation begins at a higher temperature of 244 ˚C (without adding nanotubes at 214 ˚C).

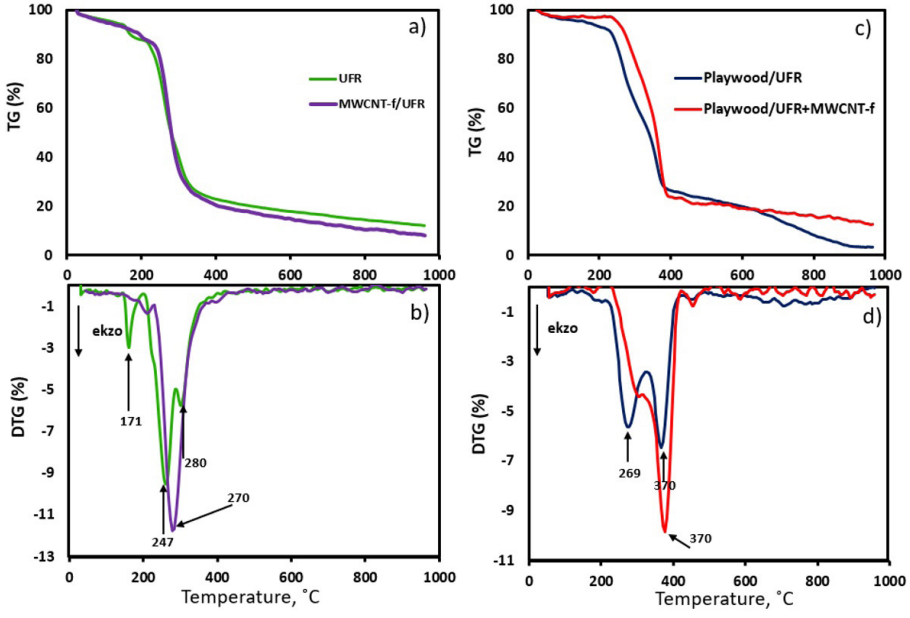

Figure 6. results of thermogravimetric and differential thermal analysis for resin (UFR) and composite (f-MWCNT/UFR)(a, b) and plywood (c, d) without and with the addition of f-MWCNT

3.2.3. Results of formaldehyde emission studies

The formaldehyde emission indicator was determined by the desiccator method for 28 days (Figure 7). A hyperbolic dependence of formaldehyde emission on experimental time was observed for all tested samples. The amount of formaldehyde released for the control sample reached its maximum value after 21 days of testing and amounted to 45.3 mg/L. When MWCNT-f was added to the adhesive composition, a decrease in the release of formaldehyde was observed. This decreased to a minimum value of 15.9 mg/L when 4% nanotubes were added.

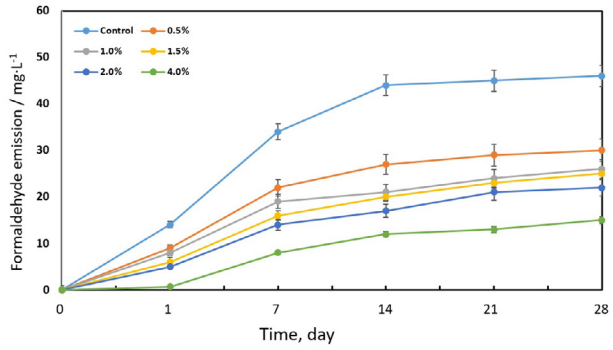

Figure 7. Emission of formaldehyde from plywood obtained using an adhesive composition containing different amounts of f-MWCNT

The decrease in formaldehyde released from plywood samples when filled with MWCNT-f resin has a nonlinear relationship, presumably due to the intermolecular interaction between formaldehyde and the functional groups of the MWCNT-f. This assumption is confirmed by IR Fourier spectroscopy data and the results of thermogravimetric analysis.

4. CONCLUSION

The results of studies of the physical-mechanical, water-resistant and environmental properties of plywood obtained on the basis of a complex binder using the adhesive composition UFR/MWCNT-f are presented. The morphological characteristics of MWCNTs were determined using TEM. Structural features were characterized using Raman spectroscopy and elemental analysis, and composition stability was determined using the Zeta potential method. Based on the results of IR spectroscopic analysis, the appearance in the IR spectrum of nanocarbon tubes activated in nitric acid, a carbonyl group and a more intense band corresponding to the stretching vibrations of hydroxyl groups connected by hydrogen bonds, was determined. The possibility of intermolecular interaction between the functional groups of MWCNT-f and urea-formaldehyde resin has been established. The thermal stability of plywood with the UFR/f-MWCNT adhesive composition was assessed, and a higher (30 ˚C) thermal stability was revealed for plywood when a nanofiller was added to the resin. During the research, a change in the strength parameters of plywood was established: the modulus of rupture increased by 62.7%, the modulus of elasticity by 113%. Water absorption of the plywood composite decreased by 43% (after 24 hours of testing). A steady decrease in the emission of free formaldehyde from plywood was determined depending on the increase in the number of multi-walled nanotubes in the adhesive composition: with the addition of 4% nanotubes, the decrease reaches a maximum of 15.9 mg/L (emission decreases by 35%). Thus, the work shows the role of carbon multi-walled nanotubes activated in nitric acid added to a urea-formaldehyde binder, which makes it possible to obtain a high-quality plywood composite with improved physical-mechanical, water-resistant and environmental performance.

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