Floresta Ambient., Rio de Janeiro, 2023; 30(4): e20230030
https://doi.org/10.1590/2179-8087-FLORAM-2023-0030
ISSN 2179-8087 (online)

ORIGINAL ARTICLE – Forest Products Science and Technology

Improving the Performance Properties of Impregnated Wood with its Subsequent Utilization into a Biochar Sorbent

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Abstract

Wood is a common natural polymeric material with hydrophilicity and low biostability with a limited service life. The article considers the issue of using a multicomponent composition (used motor oil, rosin, paraffin) for impregnating birch wood and giving it increased water resistance, bio- and dimensional stability. The possibility of recycling the used impregnated wood to obtain a biochar adsorbent was shown. The impregnating composition was characterized by the FTIR method to assess the possibility of interaction between the functional groups of the impregnating composition and wood. Moisture and water absorption decreased by more than 4 and 6 times. Volumetric swelling decreased by 57% relative to untreated wood, the effectiveness against swelling after 30 days was 46%. The high efficiency of the impregnating composition against the wood biodegradation has been established. The method of micro-X-ray tomography determined a high degree of filling of the anatomical structures of wood.

Keywords: Water resistance, biostability, micro-X-ray tomography, biochar adsorbent.

1. INTRODUCTION AND OBJECTIVES

Wood is a natural polymeric material with complex macro- and microstructure, consisting of wood fibers responsible for mechanical properties, as well as vessels and tracheids performing a conductive function (Zhong and Ma, 2022; Shi et al., 2022a). Downy birch (*Betula pubescens* Ehrh.) and silver birch (*Betula pendula* Roth.) are among the most common hardwood species in the Russian Federation and Central Europe, which can meet the demand for raw materials (Vergarechea et al., 2023) in these regions.

While having significant benefits over other materials, wood has a significant disadvantage associated with high susceptibility to atmospheric moisture (Broda and Plaza, 2023; Lee et al., 2020; Taskhiri et al., 2019). The hydrophilicity of wood is due to the presence of hydroxyl-containing components in its composition. This leads to a change in the physical and mechanical properties of wood, a decrease in water resistance, dimensional stability, and contributes to its biological destruction under the influence of wood-destroying microorganisms (Meyer and Brischke, 2015; Ning et al., 2022; Zhenyu Wang et al., 2022).

In order to preserve and improve the properties and structure, the wood is subjected to hydrofibization. Numerous studies have been carried out aimed at improving the hydrophobic properties of wood during physical or chemical modification. Among them are methods for creating a superhydrophobic coating on a wood surface with more than 150° water contact angle and less than 10° slip angles (Janesch et al., 2020; Pfeffer et al., 2012; Zhangheng Wang et al., 2022; Yang et al., 2022). Applying impregnation with urea-formaldehyde, phenol-formaldehyde, melamine-formaldehyde thermosetting resins (Altgen et al., 2023;

Boneka et al., 2021; Dorieh et al., 2022; Li et al., 2022). The use of paraffin and wax impregnation methods (Brischke and Melcher, 2015; Liu et al., 2018, 2019; Niu and Song 2021; Scholz et al. 2010; Wang et al. 2020). A relatively new method of wood surface modification is plasma treatment (Czarniak et al., 2022; Talviste et al., 2019; Han et al., 2011; Hünnekens et al., 2018). Heat treatment of wood is effective in improving dimensional stability and resistance to biodegradation (He et al., 2022). However, when using these methods, the strength characteristics of wood can be reduced and chemical modification is characterized by the complexity of the process and high energy costs. In accordance with current standards in Russian Federation, wood used in high humidity conditions and constant contact with water must be subjected to an impregnation process. In a previous study (Belchinskaya et al., 2021) we showed the possibility of utilization of used motor oil to waterproof the wood used in non-residential construction and protect it from sizing and biodegradation. Although it has been proven that used motor oils may contain heavy metals (Mishra, Asmita, et al., 2021) and other organic toxic components (García-Hernández et al., 2022; Widodo, Setyo, et al., 2018).

In this study, it is proposed to consider the use of a multicomponent impregnating composition based on used motor oil with the addition of technical paraffin and tall oil rosin as fillers. The process of using this material implies a certain period of its service. One of the areas for impregnated wood utilization is the production of a bio-char technical sorbent for the purification of industrial wastewater, gases, aerosols, and for the collection of spilled oil and oil products. In this regard, it is proposed to reuse this material as a biochar adsorbent to extract colorants such as methylene blue (MB), which is widely used in the textile industry and is a highly toxic cationic dye (Ghosh and Bhattacharyya, 2002).

Thus, the purpose of this work was to study the influence of birch wood impregnation on its water and moisture resistance, dimensional stability, and susceptibility to biological degradation, as well as to demonstrate the potential possibility of recycling the used material to obtain a technical biochar adsorbent.

2. MATERIALS AND METHODS

2.1. Materials used in the study

The wood of silver birch (*Betula pendula* Roth.) up to 30 years old obtained from the educational and experimental forestry of the Voronezh State University of Forestry and Technologies (Voronezh region, Russia) was used in the study. Samples for research were cut along the entire section of the trunk at a height of 1.3 meters. The samples had dimensions of 20 mm × 20 mm × 20 mm (tangential, radial, longitudinal) with an initial moisture content of 80±5%, according to GB/T 1931-2009.

Used engine oil GULF Formula GX Powermax SAE 5W-40 was purchased from Avrora Avto (Voronezh, Russian Federation), LLK International manufacturer. The oil was drained from the gasoline engine of the Lada Granta car (Tolyati, Russia) with engine capacity 1.6 liters. Physicochemical characteristics and elemental analysis were presented in (Belchinskaya et al., 2021).

Tall rosin was purchased from Yugreaktiv, Rostov-on-Don and used without further purification. The percentage of resin acids according to the manufacturer is indicated in Table S1.

Technical petroleum paraffin purchased from LLC "Promkhim" (Voronezh) met the requirements of GOST 23683-89, 2022. The main properties of the paraffin according to the manufacturer are presented in Table S2.

2.2. Method for obtaining impregnating composition

At the first stage, a melt of tall oil rosin was obtained during heating for 25 minutes to a temperature of 90 °C. At the same time, technical paraffin was heated and mixed with used engine oil of 80 °C. Then the components of the impregnating composition were mixed at a constant temperature (85 °C) in the ratio of mass percent: used motor oil (70%), technical paraffin (15%), tall oil rosin (15%) for 40 minutes using a magnetic stirrer (AMTAST MS -400, USA) with a frequency of 1250 rpm. The ratio of impregnating composition components was chosen in accordance with previous studies (Belchinskaya et al., 2021). The resulting impregnating composition was cooled to ambient temperature and used to treat wood samples.

2.3. Wood impregnation process

Impregnation of wood samples was carried out by hot (120°C, 60 min) and cold baths at atmospheric pressure. At least 30 samples were used in each experiment. The control group of samples was not impregnated. In the next step the wood samples were dried at room temperature at ambient conditions for 3 days, and then oven-dried at a constant temperature of 60 °C for 72 hours.

2.4. FTIR spectroscopy technique

IR spectroscopic studies were carried out with Bruker VERTEX 70 IR-Fourier spectrometer, which provides registration of IR spectra in the range of 400-4000 cm^{-1}.

2.5. Method for studying the properties of impregnated wood

The percentage weight gain (WPG) was determined based on the weight changes before and after impregnation. Water absorption (WA), volumetric swelling (S), moisture absorption (MA), anti-swelling efficiency (ASE) and swelling (a) in the radial and tangential directions were determined in accordance with Belchinskaya et al., (2021).

2.6. Micro X-ray tomography technique for impregnation quality control

For the research, 2 cylindrical wood samples with a diameter of 3 mm were prepared. Cylindrical cores were taken from centers of impregnated and unimpregnated wood samples with dimensions of 150 mm × 40 mm × 40 mm (tangential, radial, longitudinal). Then, digital models of these samples were obtained using a SkyScan 1172 X-ray microtomograph (Bruker, Germany). For a detailed picture of the void space in the studied samples additional porosity and size estimation were carried out followed by 3D-visualization.

2.7. Field annual tests for biostability in soil, water and sand media

To determine the biostability of impregnated and untreated wood, field tests were carried out for one year in soil, sand and water conditions according to (Stepina and Klyachenkova, 2014) on the basis of the test site of Voronezh State University of Forestry and Technologies. During the test, 50x50x10 mm samples were placed under 50 cm layer of the test media. Weight changes were determined after incubation in the test media for 365 days and further drying at 102±3 °C for 12 hours. The number of samples for each of the test media was at least 15 pieces.

2.8. Obtaining biochar from impregnated wood

Samples of used impregnated birch wood were utilized as feedstock. Carbonization was carried out in a PE-4610M laboratory oven (EKROS, Russia) with a digital PID controller (Fuzzy Logic) for implementing a programmable heating temperature. The carbonization process was carried out at 500°C for 3 hours in a closed, sealed, quartz crucible with a heating rate of 10°C min^{-1}. The biochar yield at these carbonization parameters was 31%.

Quantitative elemental analysis and determination of the morphological features of biochar samples were performed by scanning electron microscopy (SEM, JSM-6380LV JEOL with INCA 250 microanalysis system).

2.9. Sorption experiment

The sorption capacity of the original and modified biochar samples was determined in relation to methylene blue, that was chosen as a sorbate due to its wide use for assessing the adsorption properties of porous materials (lzaydien Atef S., 2009).

Adsorption studies were carried out under dynamic conditions at room temperature with a constant rotation speed of 250 rpm using 0.015-0.030 g sorbent and 20 ml of 100 mg/L sorbate, pH 9. The adsorption equilibrium at 25 °C was reached after 4 hours of the rotation.

The time spent for the establishment of biochar sorption saturation with sorbate was determined using kinetic studies. Weighed 0.025 g biochar was placed in a conical flask containing 20 ml of 100 mg/L MB solution. Then, the adsorption value was studied for 8 hours at 25 °C. The experiments were carried out in triplicate, the data obtained had good reproducibility, so errors were not indicated. The equilibrium sorption capacity of MB was calculated using equation of the mass balance (1) (Islam et al., 2018), and purification from MB degree was evaluated using (2).

$$X = \frac{(C_x - C_y) \cdot V}{m}, \quad (1)$$

$$\omega\,(\%) = \frac{(C_x - C_y) \cdot 100}{C_x}, \quad (2)$$

Where C_x is the initial concentration of the solution, mg/L; C_y – the equilibrium concentration of the solution after treatment, mg/L; V – the volume of the dye solution, L; m – the mass of biochar, g; – is the equilibrium sorption capacity, mg/g; – is the degree of purification, %.

2.10. Statistical analysis

The statistical significance of the study results was determined using VASSARSTAT (http://vassarstats.net/anova1u.html) to perform one-way analysis of variance.

3. RESULTS AND DISCUSSION

3.1. FTIR spectroscopy

In the process of impregnating composition and wood interaction, apparently, the formation of intermolecular bonds occurs between the functional

groups of wood and the impregnating composition. Hydroxyl functional groups play an essential role in this process. Water resistance, dimensional stability, bioresistance are directly determined by the presence of a large number of hydroxyl groups (OH) in wood, the reduction of access to which improves its performance (Ding et al., 2022; Olsén et al., 2020; Wang et al., 2023). To study the possible formation of intermolecular bonds between the components of the impregnating composition, an IR spectroscopic analysis of the impregnated and untreated wood, the impregnating composition and its main components was carried out (Figure 1).

The main characteristic functional groups of rosin had absorption bands in the range of 3300–3600 cm^{-1}, which corresponded to the stretching of the OH bond.

A band at 2922 cm^{-1} on the IR spectra of rosin and resin corresponded to the stretching asymmetric vibrations of the CH_2 group, an intense absorption band of stretching vibrations of the C=O groups 1696 cm^{-1}, characteristic of abietic acid. The characteristic absorption bands for the IR spectrum of rosin were observed in the frequency range of 2920 and 2900 cm^{-1} and represented the C–H stretching vibrations of methyl and methylene.

As a result of birch wood impregnation clear absorption bands were found in the spectra regions of 2925 cm^{-1} and 2850 cm^{-1}, characterized by symmetric and asymmetric vibrations of the methylene (CH_2) and methyl groups (CH_3) in aliphatic chains, which were present in large quantities in used motor oil and maintained in impregnated wood.

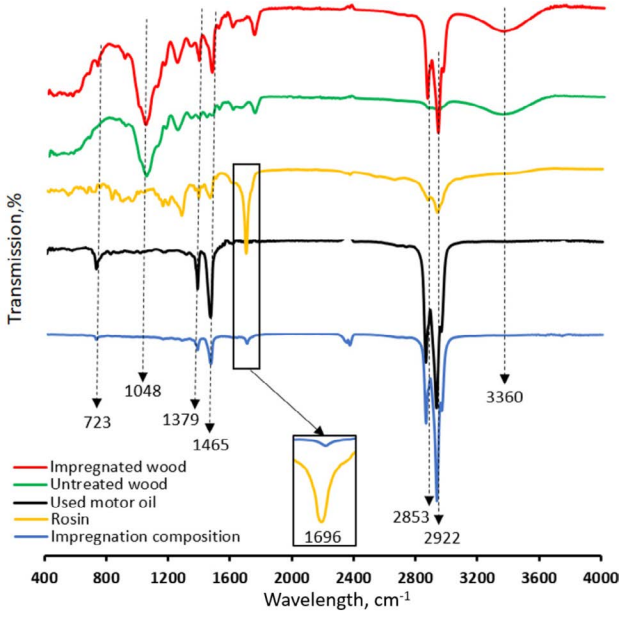

Figure 1. FTIR analysis of impregnated and untreated wood, impregnating composition, as well as the main components of the impregnating composition.

More distinct transmission minimum at 3360 cm^{-1} corresponded to OH groups bound to each other by hydrogen bonds into associates. This may be the result of the appearance of additional hydrogen bonds of the OH groups of the wood components with the impregnating composition, which made these hydroxyl groups inaccessible for interaction with water molecules, and accordingly reduced the hydrophilicity of wood.

On the IR spectra of the impregnated wood, the minima present in the IR spectrum of the untreated wood and the impregnating composition in the region of 1735 cm^{-1} were retained. It is typical for the stretching vibrations of the carbon skeleton in wood (Esteves et al., 2013), and the vibrations of the carbonyl group in used motor oil (Mishra et al., 2020) with an increase in the intensity of this band after wood impregnation. The absorption bands at 1380 cm^{-1} and 1460 cm^{-1} presented in used engine oil and appearing in wood after impregnation represented an asymmetric deformation of the CH, CH_2 and CH_3 groups. In the spectrum of treated and untreated wood, the absorption band at 1048 cm^{-1} showed the symmetrical stretching of the C-O-C ether bond, as well as the deformation of CH bonds and β-O-4 bonds in lignin (Esteves et al., 2013b). After the treatment of wood with an impregnating composition, the intensity of this band rose significantly, which indicated an increase in the number of C-O-C bonds in the sample. This may be an indirect confirmation of the chemical interaction of OH groups with the components of the impregnating composition. On the IR spectrum of treated wood, an absorption band of 723 cm^{-1} was clearly visible characterizing paraffin hydrocarbons, which were present in large quantities in used motor oil.

Thus, FTIR analysis made it possible to establish a decrease in hydroxyl groups content in the composition and the possibility of the interaction between functional groups of wood and used motor oil and rosin, which made it possible to change the surface properties of wood and increase its hydrophobicity.

3.2. Measurements of weight percent gain

The weight percent gain (WPG) showed the absorption of the impregnating composition. The average weight of dried samples before impregnation with the impregnating composition was 2.64 g, and after impregnation it was already 4.76 g. WPG after impregnation of the samples was 80.3%, which made it possible to assess the degree of impregnation and penetration of the impregnating composition into the wood structure as quite high.

3.3. Dimensional stability and water resistance of wood

To study the hydrophobic properties of wood treated with an impregnating composition, the kinetic dependences

of water absorption (WA) and moisture absorption (MA) of impregnated and unimpregnated birch wood were constructed (Figure 2 (a,b)). Volumetric, tangential and radial directions swelling, effectiveness against swelling are presented in Figure 2 (b,c). Figure 1a shows the MA after conditioning the samples in a climate chamber at 20 °C and 65% humidity. After the samples were kept in the climatic chamber for 1 day, a sharp increase in MA was observed for untreated samples up to 14.6%, while for the treated ones this indicator was less than 1% (Figure 2a). After conditioning the samples for 40 days, the MA for the untreated samples was 24.97%. For the treated samples this indicator was 4 times less reaching 6.22%.

The change in water absorption shown in Figure 2b followed a similar trend. A significant increase in WA was shown during the first 12 days for untreated wood (more than 120%). For treated wood this indicator was 6 times lower and amounted to 25% after the same period of time. After keeping the samples for 30 days in distilled water, the WA of the treated samples was 6 times less than for the untreated samples and amounted to 22.2% and 133.0%, respectively. In addition, wood treatment significantly influenced on swelling and effectiveness against swelling (Figure 2 c,d). Volumetric swelling after 30 days was 11.32% and 19.83% for treated and untreated wood, respectively. Swelling decreased after treatment from 9.31% to 6.36% in the tangential direction and from 7.78% to 5.29% in the radial direction. The effectiveness against swelling after 1 day was more than 85% and after 30 days of the study reached 46%, which indicates strong influence of the treatment on swelling.

The hydrophilicity of wood is due to the presence of free and bound hydroxyl groups in the macromolecules of the structural components of wood. Consequently, the hydrophilicity of impregnated wood was significantly reduced as a result of intermolecular interaction, which promotes improving dimensional stability and increasing MA and WU values.

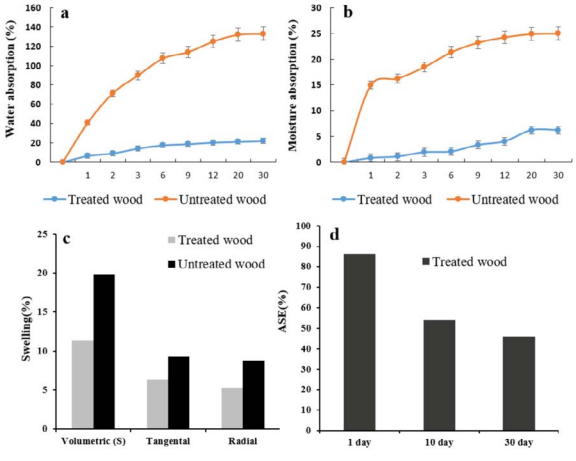

Figure 2. Moisture absorption (a), water absorption (b), swelling (c) and anti-swelling efficiency (d) of treated and untreated birch wood.

3.4. Biostability test

Figure 3 shows the results of determining the biological stability of impregnated and untreated birch wood after a year of testing. As a result of field experiment, a significant degree of protection against biodegradation in three types of conditions was established. The weight loss of the samples after a year in soil was 25% and 4% for untreated and impregnated wood, respectively. This indicator for a sand was minimal for both untreated and impregnated wood namely 20% and 3.2%, respectively. This indicator showed maximum when testing in an aqueous medium. The weight loss of untreated samples reached more than 40%, and for the treated samples it decreased more than 8 times and was less than 5%. Thus, the impregnation of wood with the proposed composition to obtain a new composite material was effective and allowed to give high rates of biostability to the material.

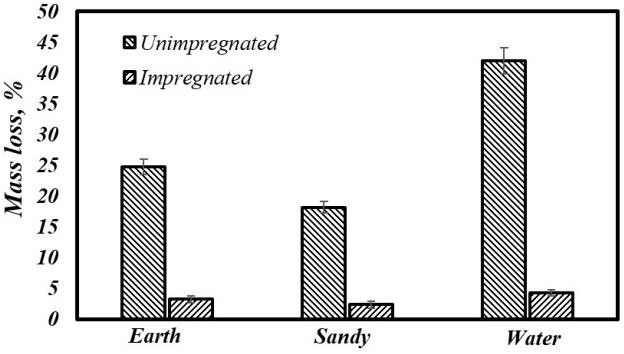

Figure 3. Biostability of impregnated and non-impregnated wood in terms of weight loss after one-year polygonal tests in soil, sand and water.

3.5. Study of the porosity and distribution of the impregnating composition in wood by micro-X-ray tomography

The method of X-ray microcomputed tomography is reliable and non-destructive, allowing an assessment of the internal microstructure of wood. This method was originally used to detect and localize internal defects in logs (Shi et al., 2022b, 2022c). With the continuous improvement of technology, it has become more widely used, for example, to check the density and moisture (Martin et al., 2021), to study the development of xylem (Lehnebach et al., 2021), the distribution of fiber texture at the junction of branches and stems (Hu et al., 2022). In addition, X-ray microscopic tomography has been used to study the microstructure and properties of acetylated wood (Sedighi Moghaddam et al., 2017) and wood impregnated with furfuryl alcohol resin (Liu et al., 2021). We have performed 3D

modeling of untreated and impregnated wood with a separate segmentation of the impregnating composition in the pores. Figures 4 show the cross-sectional planes of the analyzed sample, as well as 3D models of the pore space structure with pore segmentation into vessels and small cell is fiber, the intercellular space and the distribution of the impregnating composition among the intracellular space of wood (Figure 4).

The anatomical orientation of wood structures significantly influences the wood impregnation process (Frias et al., 2021; Hass et al., 2012; Tondi et al., 2013; Ponomarev et al., 2021b, 2022). The obtained images of the distribution of the impregnating composition (Figure 4b and d) show that its movement occurs along the path of least resistance in the end direction along large anatomical structures, such as vessels and fibers.

Used motor oil may contain some toxic components, as well as heavy metals formed as a result of thermal destruction during engine operation (García-Hernández et al., 2022; Widodo, Setyo, et al., 2018). However, it can be used in remote areas with limited populations where continuous supply of goods cannot be ensured, and as an alternative to the more toxic creosote.

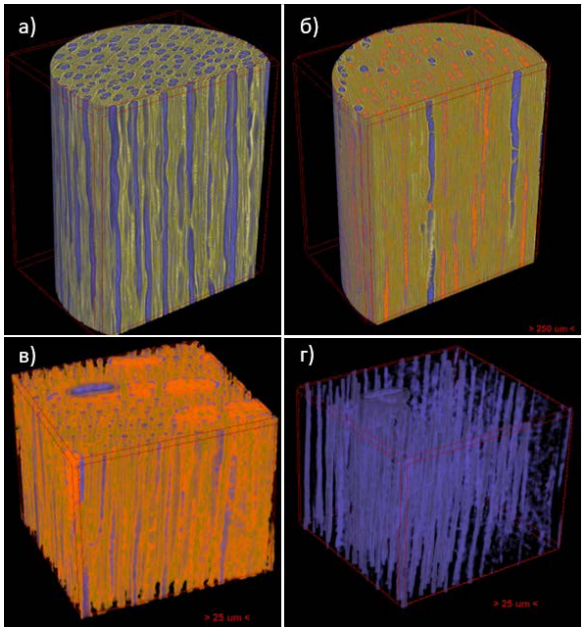

Figure 4. 3D models of wood samples before (a) and after (b) impregnation, as well as visualization of the void space (c) and the distribution of the impregnating composition (d).

3.6. Study of surface morphology and particle size of biochar

As a result of elemental analysis in biochar adsorbent samples, it was found that the content of the total atomic weight of carbon (C), oxygen (O), potassium (K) and calcium (Ca) in biochar adsorbent samples was 76.46, 23.24, 0.02% and 0.28, respectively. The C/O ratio for the resulting sample was 0.30, hence the resulting material can be considered biochar.

A study with a scanning electron microscope makes it possible to establish the dispersion degree and the particle size distribution of obtained biochar (Figure 5a, c).

The images obtained clearly show particles of different fractional sizes. Most of the particles (over 50 percent) were in the 25-30 micron size range. Particles of biochar adsorbent with a size of 50-75 microns were less than 25%.

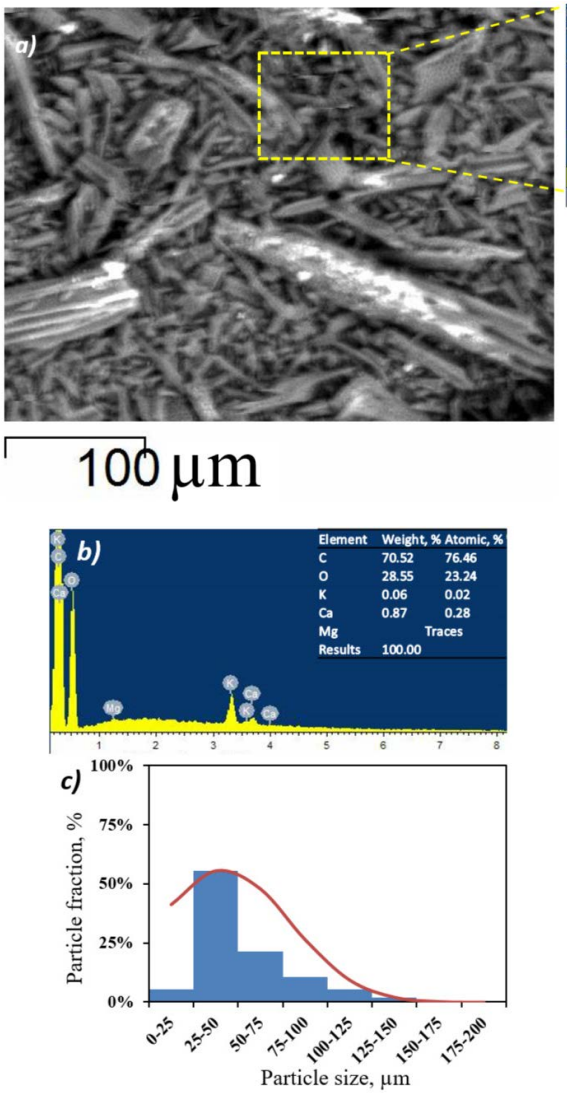

Figure 5. SEM analysis of biochar adsorbent (a), EDX spectrum (b) and particle size distribution (c).

3.7. Sorption properties of biochar

The mass of biochar sorbent is one of the main factors influencing the effect of MB adsorption. On the one hand, ultimate dye amount should be removed from the solution,

and on the other hand, wastes caused by excessive use of the adsorbent must be avoided. Figure 6 shows change in the degree of purification of the aquatic environment from MB (Figure 6a) and sorption kinetics of the studied sorbate (Figure 6b). The increase in sorption of MB on biochar has an almost linear dependence. With an increase in sorbent mass to 0.0255 g, the purification of the solution from MB increased to 91%, reaching a maximum value.

According to the data obtained (Figure 6b), the highest adsorption rate of the MB dye on biochar was found to be in the range of 0-2 hours, then the rate slowed down (from 2 to 8 hours, Figure 6b) due to the saturation of the adsorbent surface with adsorbate molecules (Wu et al., 2022a). Similar results have been obtained by other researchers. For example, in (Wu et al., 2022b), the authors used an eggshell membrane to obtain biochar and adsorb MB. The degree of decolorization was about 95%. In the study (Rubio-Clemente et al., 2021), the authors used Scotch pine wood to obtain biochar to purify water from malachite green, the degree of purification was somewhat lower and reached 87%.

Thus, the possibility of obtaining a biochar sorbent from used impregnated wood with high sorption properties relative to MB had been proved. The processing of impregnated wood into biochars can be considered as the utilization of impregnated wood to obtain a sufficiently valuable material.

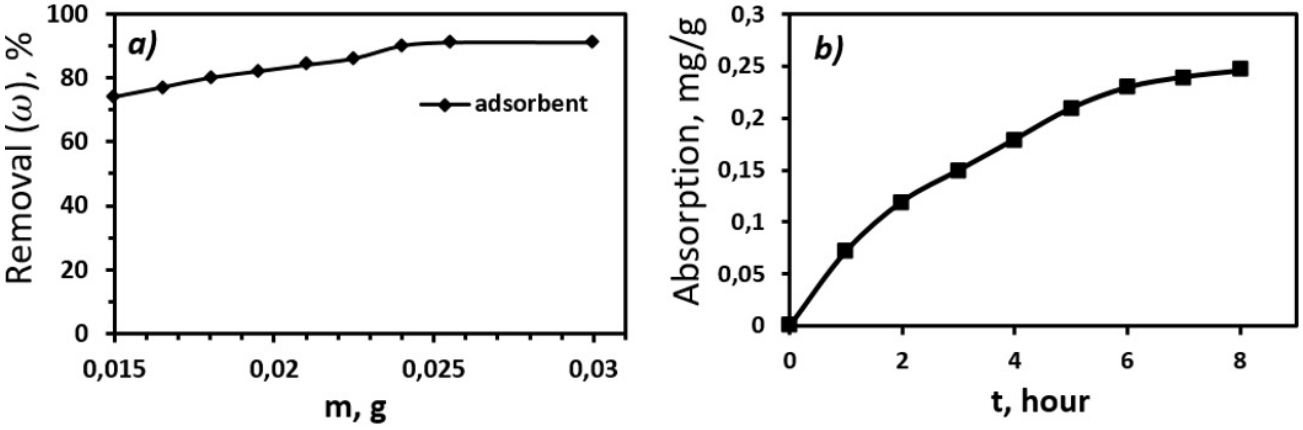

Figure 6. The percentage of purification from methylene blue with a change in the mass of the adsorbent (a) and the sorption kinetics in 0 to 8 hours period (b)

4. CONCLUSIONS

The paper presents a complex composition developed by the authors for wood impregnation consisting of used motor oil (70%), paraffin (15%), tall oil rosin (15%), the use of which makes it possible to increase the water and biostable performance of birch wood. Chemical bonds formation between the functional groups of wood and the components of the impregnating material during the preparation of the impregnating composition and the impregnated wood composition was characterized by FTIR analysis. Moisture absorption of impregnated wood decreased by more than 4 times and amounted to 6.22%, and water absorption by more than 6 times (22.2%). Volumetric swelling was reduced by 57% relative to untreated wood. The effectiveness against swelling was 46% after 30 days of the experiment. The high efficiency of the impregnating composition against the biodegradation of wood was shown during annual field tests in soil, sand and water. The method of micro-X-ray tomography was used to study the quality of the impregnation and the degree of its distribution over the wood structure. A high degree of filling of the wood anatomical structures was established. The study showed the possibility of obtaining a biochar sorbent from used impregnated wood for utilization in wastewater treatment using MB as an example. The purification of the solution from MB was more than 90%. Thus, the proposed impregnating composition was able to protect wood from water and moisture, give it dimensional stability and high biostable performance. Proving the potential of recycling used impregnated wood as a biochar adsorbent makes it possible to create conditions for a waste-free wood impregnation technology.

ACKNOWLEDGEMENTS

The study was supported by the Russian Science Foundation grant No. 23-23-00122, https://rscf.ru/project/23-23-00122/

SUBMISSION STATUS

Received: 29 Aug. 2023
Accepted: 14 Nov. 2023
Associate editor: Fernando Gomes

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SUPPLEMENTARY MATERIAL

The following online material is available for this article:

Table S1 - The percentage of resin acids in the rosin according to the supplier.

Table S2 - Technical parameters of the paraffin according to the manufacturer.

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