Improving Wood Surface Wettability through Gas-phase Ozone Treatment of Air-dry wood

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An increase in wood free surface energy enhances the wettability of wood surfaces, leading to better interaction with water-based coatings. This study investigated the effect of gas-phase ozonation on the wettability of spruce, thermo-modified pine, and birch woods. The effects of the treatment were evaluated by measuring the water contact angle and the Cobb value on the wood sample surfaces, and by determining the surface free energy of the wood surfaces using the Owens, Wendt, Rabel, and Kaelble (OWRK) calculation method. Furthermore, water absorption and evaporation rates were assessed through water immersion and subsequent drying of the wood samples. The results indicated that ozone treatment increased the surface energy, and especially its polar component, thus accelerating water spreading and absorption on the wood surfaces. The most probable cause of the observed effects is the formation of new carbonyl and carboxyl groups resulting from reactions of the ozone with the wood surface. The findings suggest that the ozone treatment technique can enhance spreading, absorption, and adhesion of water-based adhesives and coatings to wood surfaces. This research may facilitate the development and use of new environmentally friendly water-based adhesives and coatings.

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INTRODUCTION

Ozone (O_3) is a highly reactive molecule due to its strong oxidative properties (E° = 2.07 V, 25 °C), and it can have various reactions with different materials. Ozone readily reacts with organic compounds, particularly those containing carbon-carbon double bonds. This reaction is called ozonolysis, where ozone breaks down the double bonds and forms compounds including aldehydes, ketones, and carboxylic acids. Ozone can also react with aromatic compounds, breaking the aromatic ring and forming aldehydes and acids. Ozone molecules are unstable, the lifespan of gaseous ozone in atmosphere varying from a few minutes to several months depending on environmental conditions (Greene *et al*. 2012; Travaini *et al*. 2015; Epelle *et al*. 2023). Industrially produced ozone is commonly generated by dissociation of molecular oxygen using electric discharge. The short-lived oxygen atoms (O) react rapidly with oxygen (O_2), forming ozone (O_3) molecules. The electrical energy consumption of ozone produced in this way is 10 to 20 kWh/kg, depending on the efficiency of the equipment (Magara *et al* 1995; Hostachy and Wyk 2014). Ozone's strong oxidizing capability is utilized across a range of applications,

including fresh and wastewater treatment, air purification, odor removal, and equipment sanitization in the food and medical industries. In the pulp and paper industry, ozone is used as a bleaching agent, reducing reliance on chlorine-based chemicals. It is also employed in the textile industry to bleach textile fibers (Magara *et al*. 1995; Van Lierop *et al*. 1996; Epelle *et al*. 2023).

In lignocellulosic materials, such as wood or wood-based papermaking fibers, ozone primarily targets the electron-rich lignin, and it has significantly less reactivity toward carbohydrates. Oxidation of lignin by ozone results in lignin depolymerization, the opening of an aromatic rings, and the formation of water-soluble organic acids. With polysaccharides, oxidation of primary and secondary hydroxyl groups generates carbonyl and carboxyl groups (Travaini *et al*. 2015; Coca *et al*. 2016; ET Zhang *et al*. 2024) (Fig. 1a and 1b). Due to its selectivity, ozone is well suited to the removal and bleaching of residual lignin in papermaking and textile fibers, as it leaves the cellulose largely intact. In bleaching applications, which aim to react with and remove residual lignin, fiber water content is an important process parameter as the water functions as the transport medium of both the ozone and lignin degradation products. If the amount of water is insufficient, the reaction is limited by mass transfer and the ozone is unable to reach the substrate and react there. The reactions are then limited to the outer surface of the lignocellulosic particles. On the other hand, an excess of water reduces the ozone concentration in the liquid phase, and thus the reaction rate. Other important process parameters of the bleaching processes are the ozone concentration of the medium and the ozone treatment time in particular (Travaini *et al*. 2015, 2016; Zhang *et al*. 2024). In ozone treatment to improve enzymatic hydrolysis of lignocellulosic biomasses for biofuel and biochemical production, the criteria for determining the optimum moisture content can be the amount of removed lignin, the degradation of hemicellulose and cellulose, or the yield of sugar release in the enzymatic hydrolysis step following the ozone treatment (Travaini *et al*. 2015; Coca *et al*. 2016). Reported optimal consistency for these processes varies widely, being typically in the range of 10 to 45% for papermaking pulp bleaching applications (Van Lierop 1996; Pikka *et al*. 2000).

So far, little information has been published on the effects of gaseous ozone treatment of dry wood, such as the composition and surface properties of the wood. Safiullina *et al*. (2020) and Mukhametzyanov *et al*. (2021) have studied how gaseous ozone treatment of room-dry natural and thermally modified birch wood affects the wettability of the wood surface. According to these studies, ozonation treatment results in improved wood surface wettability by polyvinyl acetate (PVA) water solution (10 wt.%), simulating water-based wood glue, and further, the treatment improved strength of load-bearing glued wood joints. Mukhametzyanov *et al*. (2021) concluded that ozonation results in wood surface oxidation, leading to the appending of carbonyl and carboxyl groups and contributing to an increase in wettability and in the free surface energy of the wood. According to Mamleeva *et al*. (2020), at moisture contents of above 30 to 40%, where the wood is in a swollen state, ozonation reactions take place in both the aqueous phase with dissolved ozone and in the gas phase, resulting in lignin degradation and the formation of low molecular water-soluble acids, whereas at moisture contents below 30% the reactions occur predominantly on the outer wood surfaces, at the interface with the gas phase. To the best of our knowledge, no comprehensive studies detailing the reaction products of gas-phase ozonation on dry wood surfaces have been published.

For this study, air-dry (RH 50%, 23 °C) planed Nordic spruce sawn timber (spruce), planed thermally modified Scots pine timber (TM pine), and sanded 12-ply

Nordic birch plywood (birch) were treated with gaseous ozone, and the effects of the treatment on the wettability and water absorption of the samples were examined using water contact angle and Cobb-value measurements, water soaking tests, and determination of wood surface free energy and its dispersive and polar components using the Owens, Wendt, Rabel, and Kaelble (OWKR) calculation method. Additionally, the effect of ozone treatment on the chemical composition of the wood sample surfaces was examined using ATR-FT-IR-spectroscopy. The motivation for this study lies in the potential to enhance wood surface wettability, which could facilitate the development and use of new, more environmentally friendly water-based wood adhesives and coatings (Chandler *et al*. 2005; Kamke and Lee 2007). The wettability, water absorption, and surface energy measurements were conducted without washing the wood samples after the ozonation treatment, as this approach would be most feasible in practice. Also, the improved adhesion of paints and other coatings to wood surfaces after ozone treatment could potentially eliminate the need to use primers to improve paint adhesion to wood. Overall, an improvement in the adhesion of coating materials to wood surfaces could extend the maintenance intervals of coated wooden surfaces and thus improve wood products' technical competitiveness compared to other materials.

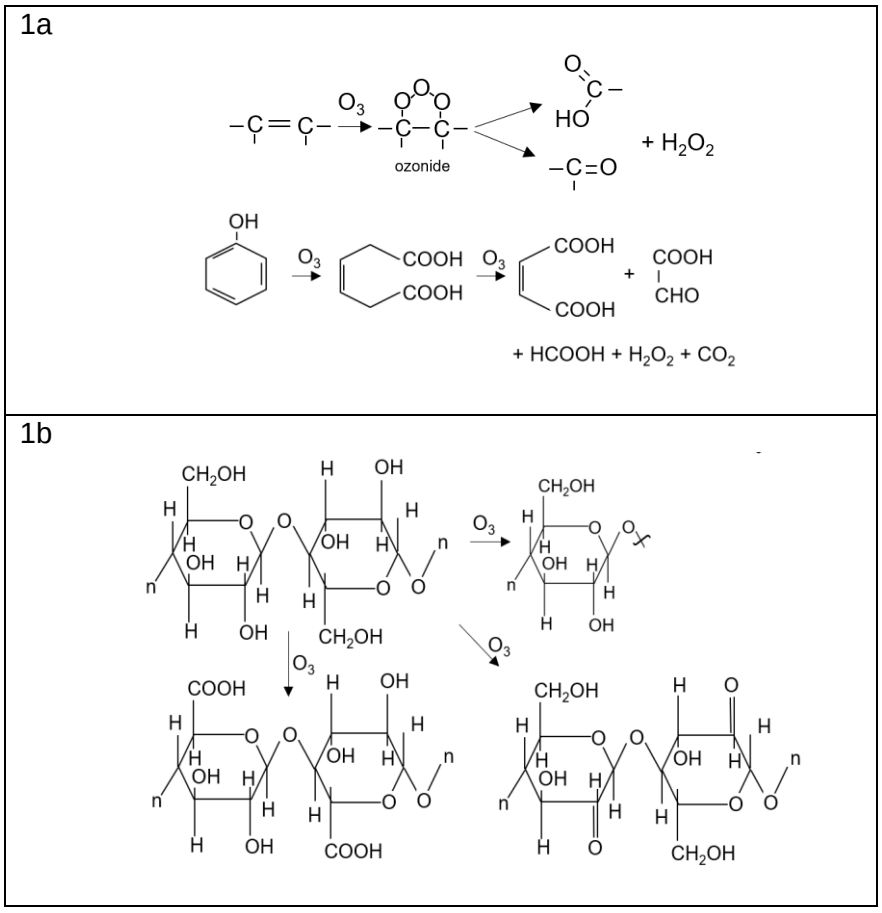

Fig. 1. Suggested mechanism of carbonyl and carboxyl group formation in reactions of ozone with lignin (1a) and cellulose (1b) (Coca *et al*. 2016; Zhang *et al*. 2024). The carbonyl and carboxyl groups formed, which are polar in nature, are assumed to be largely responsible for the increased surface energy of the lignocellulosic material.

EXPERIMENTAL

Materials

Planed Nordic spruce sawn timber (spruce) (9 cm × 5 cm × 2 cm), planed thermally modified Scots pine sawn timber (TM Pine) (10 cm × 5 cm × 2 cm), and sanded 12-ply Nordic birch plywood samples (birch) (10 cm × 5 cm × 1.7 cm) were sourced from Finnish wood product manufacturers. In the spruce samples, the long axis was oriented perpendicular to the grain direction, while in the thermally modified pine and in the top-plies of the birch plywood it was aligned parallel to the grain. All wood samples were stored in dry indoor conditions, protected from light, for at least one year. Before the ozone treatment and subsequent measurements, the samples were conditioned for at least two weeks at a relative humidity (RH) of 50% and a temperature of 23 °C. Water absorption tests were carried out using Milli-Q water. Surface energy was determined using Milli-Q water, analytical-grade formamide (Merck KGaA, Germany), 99.8% ethylene glycol (Sigma-Aldrich Co., USA), and 99% diiodomethane (Sigma-Aldrich Co., USA).

Ozonation

The ozonation treatment of the air-dry (RH 50%, 23 °C) wood samples was carried out using VTT's (Espoo, Bioruukki) laboratory ozonation equipment, originally designed for bleaching papermaking pulp fibers. Basically, the equipment consisted of an ozone generator (Emery-Trailigaz Model Sorbios, USA) and a flow-through ozonation reactor (Ø 235 mm, height 395 mm). The treatment entailed placing the wood samples in the bottom of the reactor and directing an ozone-air mixture into the chamber. For the treatment, first the ozone generation in the carrier oxygen gas was stabilized, then the ozone flow was led to the potassium iodide solution to determine the ozone content of the gas, and then it was led into the reactor. After oxygen flushing of the reactor, the ozone-air mixture was led though the reactor. Both the oxygen flushing and the ozonation were performed at a constant overpressure of 0.5 bar to control the ozone flow. The gas consisted of 10% ozone and 90% oxygen, and the flow rate through the reactor was two liters per minute. After 90 minutes of reaction time, the reactor was rinsed with oxygen again. The ozone content of the residual gas from the reactor was analyzed, and the ozone generation was checked after the reaction time. Ozone formation and consumption were determined from the potassium iodide solution by titration with sodium thiosulfate. In the treatment, the realized ozone consumptions expressed as wt.% of the air-dry (RH 50%) samples were 1.2 wt.% and 3.9 wt.% for the spruce, 1.0 wt.% and 4.0 wt.% for the pine, and 1.0 wt.% and 4.1 wt.% for the birch. After treatment the samples were stored at RH 50% and 23 °C for two weeks before measurements were taken.

Contact Angle Measurement and Determination of Surface Free Energy

Water contact angle measurements were conducted with an Attention Theta Optical Tensiometer (Biolin Scientific, Sweden). The measurements were performed at 23 °C and 50% relative humidity using distilled water as the probe liquid with a droplet volume of 4.0 µL. The measurements were conducted on tangential wood surfaces using three parallel wood samples, with three measurements taken on the surface of each sample (a total of nine measurements). The measurement points on the wood surfaces were selected to evenly represent both earlywood and latewood. The results reported are the averages of the measurements taken after a 1-second application time. Due to measurement constraints, the contact angles were measured perpendicular to the grain direction for the spruce

samples, and parallel to the grain direction for the TM pine and birch samples. The surface energy measurements were conducted with four test liquids: water, ethylene glycol, formamide, and diiodomethane (DIM). Surface free energy values and dispersive (non-polar) and polar components were calculated using the Owens, Wendt, Rabel, and Kaelble (OWRK) method. Measurements with different solvents were conducted using the same wood sample. The calculations and the reported results are based on averages of three to five parallel measurements with each test liquid.

Cobb and Water Soaking Tests

Water absorption on the tangential wood surfaces of three parallel wood samples was measured using a modified Cobb test. The Cobb test was originally used for determining the amount of water (g/m^2) absorbed by a paper in a given time. In the performed tests, a rubber sealing ring and a metal cylinder were placed on the surfaces of pre-weighed wood samples (RH 50%, 23 °C), with the base area of the cylinder and the sealing ring being 10 cm^2. Then a 1 kg weight was placed on top of the cylinder to improve sealing between the wood and the cylinder. Finally, 10 mL of Milli-Q water was added to the cylinder. After 120 seconds, the wood sample and the cylinder were turned upside down, and the extra water was wiped off the wood surface with blotting paper. The sample was then weighed, and the amount of absorbed water in g/m^2 was calculated. The reported results are the averages of three parallel measurements.

For the water soaking test three parallel pre-weighed air-dry (RH 50%, 23 °C) wood samples were soaked in Milli-Q water and the total weight gain of the samples measured after 5 min, 30 min, 90 min, 320 min, and 24 h. After this, the parallel samples were allowed to dry at RH 50% and 23 °C, and the weight change of the samples measured after 45 min, 135 min, 405 min, and 24 h.

ATR-FT-IR Analysis

ATR-FT-IR spectral data on the wood samples was obtained with a Nicolet iS50 FTIR spectrometer (Thermo Fisher Scientific, USA). The spectrometer was equipped with a single reflection diamond ATR crystal, and for each sample 32 scans of tangential springwood were taken at multiple different surface locations. All spectra were obtained with a resolution of 4 cm^{-1} in absorption mode using a wavelength range of 400 to 4000 cm^{-1}. The collected data were processed with OMNIC™ software (Thermo Fisher Scientific, USA) with averages computed using a built-in option in the software. The data was then sorted and compared to data reported in the existing literature.

Microscopic Examinations and Shore Hardness Measurement

Microscopic examinations of the fibers were done using a Merlin FE-SEM scanning electron microscope (Carl Zeiss Microscopy GmbH, Oberkochen, Germany). The FE-SEM samples were sputter coated with an EM ACE200 (Leica, Germany) with 5 nm layer of Au/Pd prior to SEM imaging. The imaging was carried out with the gun voltage of 2 kV and current 60 pA using SE2 and InLens detectors. Original imaging was done with several magnifications between 200X and 4000X. The pixel resolution of the FE-SEM was 1024×768. Wood samples surface hardness was measured at 20 randomly selected points using a Shore D durometer (Sauter GmbH, Germany).

RESULTS AND DISCUSSION

The water contact angle measurements and Cobb tests conducted on the tangential wood surfaces showed that ozone treatment increased surface wetting and enhanced the water absorption of the wood surfaces (Fig. 2). The effects on spruce, TM pine, and birch were generally similar, although the changes in the case of the spruce were slightly less pronounced compared to the other wood types, possibly due to the perpendicular-to-the-grain measurement of the spruce. To date, only a limited number of studies have been published on the treatment of dry wood with gaseous ozone. The observed decrease in water contact angle on the wood surfaces is in line with the observations of Safiullina *et al*. (2020) and Mukhametzyanov *et al*. (2021), who reported a decreasing effect of ozone treatment on the contact angle of a 10% aqueous solution of polyvinyl acetate (PVA) on natural and thermo-modified birch wood.

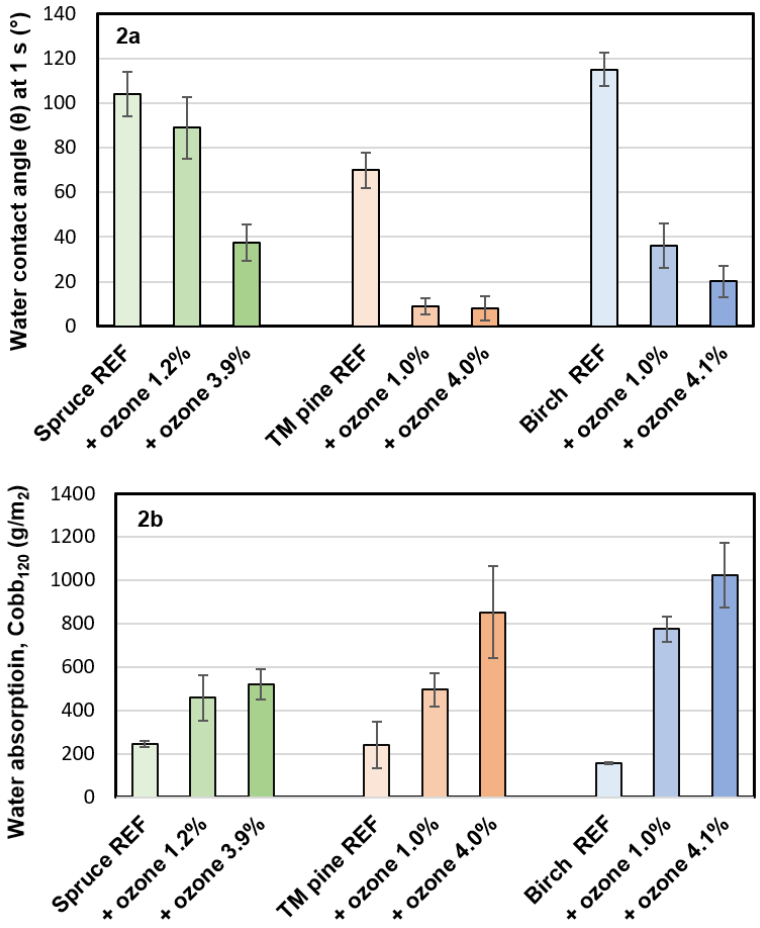

Fig. 2. Effect of gas-phase ozone treatment on the water contact angle (Fig. 2a) and water absorption (Cobb test, Fig. 2b) of spruce, TM pine, and birch wood. The ozone% indicates the ozone consumption (wt.% of air-dry (RH 50%, 23 °C) wood weight) of the treatment.

Figure 3 presents the effects of ozone treatment on the total surface free energy (γ_t) of the tested wood samples, and its dispersive (γ_d) and polar components (γ_p). The results were determined using the Owens, Wendt, Rabel, and Kaelble (OWRK) calculation method which decomposes the total surface free energy into dispersive and polar

components ($\gamma_t = \gamma_d + \gamma_p$). A high dispersive component suggests that the material surface interacts primarily through non-polar interactions like Van der Waals forces, whereas a high polar component indicates the presence of polar groups on the surface, such as hydroxyl (-OH), carbonyl (C=O), or carboxyl (-COOH) groups. In general, polar groups interact with polar liquids, such as water, thereby enhancing surface wetting by polar coatings such as water-based paints. Similarly, surfaces that are rich in dispersive (non-polar) components interact with non-polar coatings such as solvent-based paints. In practice, an increase in the polar component of a material surface can enhance the wetting of water-based coatings and improve the adhesion to the surface (Chandler *et al*. 2005; Kamke and Lee 2007). The results in Fig. 3 show that ozone treatment increased the surface free energy of the spruce, TM pine, and birch, particularly by enhancing the polar component while concurrently reducing the dispersive component. The untreated spruce exhibited a high dispersive component and a low polar component, which may be due to the migration of extractives to the surface during storage of the samples. The observed alterations in surface free energy and its components are likely due to the formation of carboxyl, carbonyl, and hydroxyl groups in the lignin. Additionally, the generation of carbonyl and carboxyl groups in cellulose and hemicellulose, along with the cleavage of lignocellulosic polymers, may contribute to these changes in surface energy (Travaini *et al*. 2015; Coca *et al*. 2016; Zhang *et al*. 2024).

The calculated surface free energy (γ_t), dispersive components (γ_d), and polar components (γ_p) of the reference samples are consistent with previously reported values for wood (Nguyen and Johns 1978; Liptáková and Kúdela 1994). However, it is important to note that the values reported in the literature may not be comparable, as they are significantly affected by the surface free energy calculation method used, as well as sample surface roughness, moisture content, and aging of the sample before analysis.

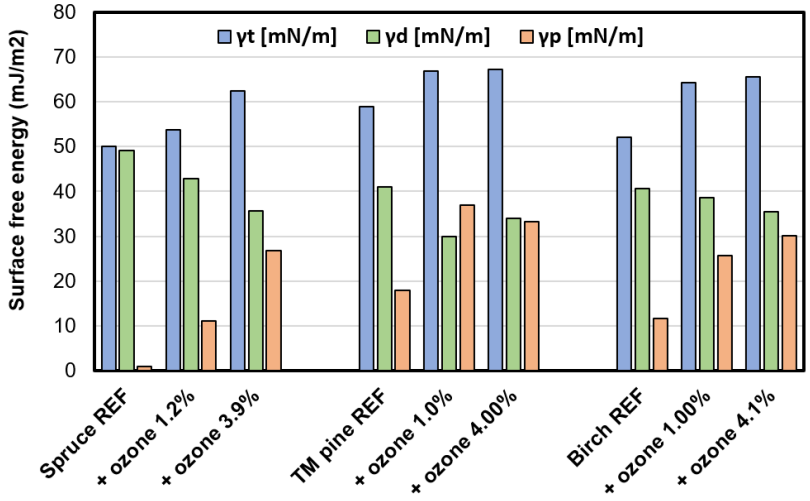

Fig. 3. The effect of gas-phase ozone treatment on surface free energy (γ_t) and its dispersive (γ_d) and polar (γ_p) components, determined using the OWRK calculation method. The ozone% indicates the ozone consumption (wt.% of wood weight) of the treatment.

Based on the results presented in Figs. 4a-c, ozone treatment did not have as clear or consistent an effect on the water absorption of the wood samples when measured by the water soaking test compared to that observed in the Cobb test. This result may be due to the ozone reacting primarily with the outermost surface layer of the wood, or to the rapid

absorption of water by the cross-cut surfaces of the wood samples, which may obscure the effects of the ozone treatment. Unfortunately, in the present study, the depth of ozone reactions in the wood surface could not be measured. Nevertheless, the samples treated with the highest ozone dose showed slightly higher water absorption. This could be due to depolymerization reactions in the wood's surface layer, which is most susceptible to ozone exposure, resulting in increased wood swelling capacity or an increase in wood surface porosity. Further research is needed to confirm this.

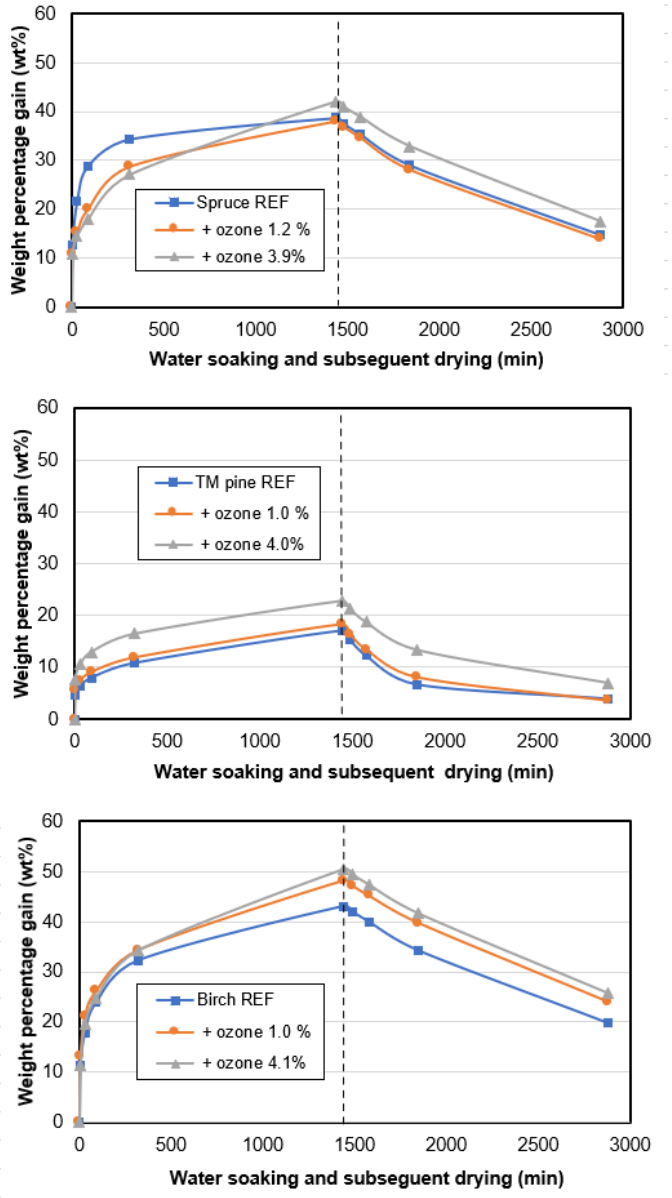

Fig. 4. The effect of ozone treatment on weight percentage gain of wood samples after water soaking and subsequent drying at RH 50% and 23 °C. The dotted line indicates the starting time of sample drying.

In the present study, the ATR-FT-IR method was used to examine chemical changes caused by ozone treatment on wood sample surfaces. For ATR-FT-IR measurement, a thin wood chip was taken from the sample surface and the tangential wood surface was placed in contact with the ATR-crystal, enabling qualitative and semi-

quantitative analysis of the chemical composition of the surface. In the case of the birch and TM pine the ozone treatment resulted in a slight increase in the absorbance at wave number 1732 cm^{-1}, which is the characteristic absorption wavelength of carboxyl and carbonyl groups in lignocellulosic materials (Fig. 5). These observations were consistent with the results reported by Hoang *et al*. (2014) on the effects of gaseous ozone on the surface chemistry of oak and bamboo wood. Also, the anomaly at the 1648 cm^{-1} band might be the result of O–H bending of adsorbed water or H-bonded C=O stretching in coniferyl/sinapyl aldehyde (two main precursors of lignin in wood) (Piqueras *et al*. 2020). In the case of the spruce wood, however, the absorption spectra located at wave number 1732 cm^{-1} were not consistent. Also, insignificant shifts in the bands were observed in the spruce and pine. The increase in absorption observed at 1732 cm^{-1} in the ATR-FT-IR spectrum supports the hypothesis that ozone treatment of wood leads to the formation of new carbonyl and carboxyl groups (Bodirlau and Teaca 2009; Kostryukov *et al*. 2023). However, the spectral differences observed in the present study were quite small, rendering interpretation of the spectra somewhat uncertain.

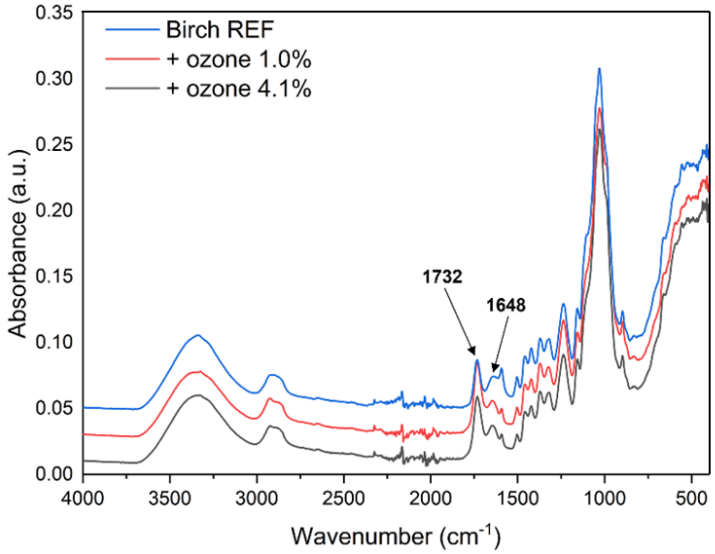

Fig. 5. ATR-FT-IR spectra of untreated reference birch wood and ozone-treated birch wood at two different ozone uptakes. The slightly higher absorbance at wave number 1732 cm^{-1} is likely due to the formation of carbonyl and carboxyl groups in the lignin, and possibly also in the cellulose and hemicelluloses.

The ozone treatment had no visible effect on the color or smoothness of the wood samples. The measured weight gains determined at RH 50% and 23 °C, caused by the ozone treatment, were not consistent, being in the range of 0.2 to 0.6 wt.%. In principle, the small measured weight gain may be due either to the formation of carboxyl and carbonyl groups in the wood and/or to increased absorption of water by the ozone-treated wood at RH 50% and 23 °C. No consistent differences were observed either between the reference samples and the ozone-treated samples in the examinations of wood samples surface structure conducted with the SEM microscope. The measured Shore D hardness values for spruce REF, birch REF and TM pine REF samples were 62 (st.dev 7), 67 (st.dev. 3) and 51 (st.dev. 8), respectively. The ozone treatments also had no consistent or statistically significant effect on wood Shore D hardness. Thus, the results suggest that the most significant effect of the gaseous ozone treatment in terms of practical applications is

the improvement in wood surface wettability. It is possible that this effect could be utilized to improve the adhesion and durability of paints, wood oils and waxes, and other wood surface coatings. In particular, the increase in wood surface free energy and its polar component may facilitate the use of novel environmentally friendly water-based coatings and potentially reduce the need for primers under water-base topcoats.

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CONCLUSIONS

1. Gas-phase ozone treatment of air-dry wood resulted in an increase in wood surface free energy and its polar component, and it improved the wettability of the wood surface.
2. The increase in wood surface free energy and its polar component is primarily a consequence of the ozone reactions with the lignin present on the wood surface, leading to the degradation of lignin and the formation of lignolytic carboxyl, carbonyl, and hydroxyl groups, which are polar in nature. Also, the formation of carbonyl and carboxyl groups is possible in cellulose and hemicellulose, as well as cleaving of the polymer chains.
3. The gaseous ozone treatment had no visible effect on the color and overall appearance of the wood samples nor on their hardness. The weight gain was also small. The results suggest that the most significant effect of the treatment in terms of practical applications is the improvement in wood surface wettability.
4. The findings indicate that ozone treatment may be an effective method to improve the spreading, absorption, and adhesion of water-based adhesives and coatings on wood surfaces especially. This could support the adoption of environmentally friendly water-based adhesives and coating materials in wood product manufacturing and finishing.

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