Nikolina Barlović[1], Anka Ozana Čavlović[1], Stjepan Pervan[1],
Miljenko Klarić[1], Silvana Prekrat[1], Nikola Španić[1]

Chemical Changes and Environmental Issues of Heat Treatment of Wood

Kemijske promjene i ekološka problematika toplinske obrade drva

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ABSTRACT • *The paper gives an overview of chemical changes during heat treatment of wood and their influence on environment. Wood is one of the most used building materials because of its physical and technological properties. Various procedures improve its properties, and due to its environmental acceptability, heat treatment of wood is one of the most commonly used. Heat treatment causes changes in the cell walls and degradation of the main components of the wood structure (cellulose, hemicelluloses, lignin) and extractives. Hemicelluloses, as the most unstable components, are broken down first, followed by cellulose and finally lignin. Degradation results in a change in chemical composition and chemical reactions of colour change causing a technological problem because wood processing tends to make the colour as uniform as possible. The uniformity of colour and the desired colour tone are obtained by the process of steaming and thermal modification. The processing temperature is the most important factor that causes all the changes. Due to its wide use, need has arisen to investigate the ecological consequences of such a heat treatment process, as well as the impact of harmful substances and types of compounds released during the process and their toxicity.*

KEYWORDS: *heat treatment; heat-treated wood; wood components; colour change; ecology*

SAŽETAK • *U radu je naveden pregled kemijskih promjena tijekom procesa toplinske obrade drva te njihova utjecaja na okoliš. Zbog svojih fizikalnih i tehnoloških svojstava drvo je jedan od najčešće upotrebljavanih građevnih materijala. Njegova se svojstva poboljšavaju raznovrsnim postupcima, a toplinska je obrada drva zbog njezine ekološke prihvatljivosti jedna od najzastupljenijih metoda obrade. Pri toplinskoj obradi drva dolazi do promjena u staničnim stijenkama drva i do razgradnje njegovih glavnih gradivnih komponenata (celuloza, hemiceluloze i lignin) i ekstraktivnih tvari. Hemiceluloze se kao najnestabilnije komponenete razgrađuju prve, zatim slijedi razgradnja celuloze te, na kraju, lignina. Razgradnja rezultira promjenom kemijskog sastava drva i kemijskim reakcijama promjene boje, što je tehnološki problem jer se pri preradi drva teži tome da boja bude što ujednačenija. Stoga se ujednačavanje boje i željeni ton nastoje postići procesom parenja i pregrijavanja drva. Najvažniji činitelj koji uzrokuje sve promjene jest temperatura obrade drva. Zbog vrlo česte primjene toplinske obrade drva nametnula se potreba za istraživanjem ekoloških posljedica tog postupka, pri čemu se istražuje i utjecaj nusprodukata i vrsta spojeva koji se oslobađaju za vrijeme tog procesa, kao i toksičnost oslobođenih spojeva.*

KLJUČNE RIJEČI: *obrada drva toplinom; toplinski obrađeno drvo; drvne komponente; promjena boje drva; ekologija*

[1] Authors are researchers at University of Zagreb, Faculty of Forestry and Wood Technology, Zagreb, Croatia.

1 INTRODUCTION
1. UVOD

Wood is one of the most common construction materials with very good aesthetic, acoustic, thermal and mechanical properties; but with some disadvantages, such as dimensional anisotropic instability due to hygroscopicity and low durability. In order to improve these properties, heat treatment of wood is performed, which due to its environmental acceptability has become a better option compared to chemical modification (which is also very commonly used). Heat treatment of wood is a process in which the chemical structure of cell walls changes only in the presence of heat, pressure and moisture, in an oxygen-poor environment, without the addition of chemicals. When the structure is modified, there are changes such as reduced affinity for water, improved dimensional stability, greater resistance to biodegradation, uniform changes in colour to darker tones. At the same time there are negative changes such as reduced mechanical properties, the appearance of cracks and light induced discoloration. The process consists of drying, steaming and thermal modification. Prolonged thermal modification process increases dimensional stability and durability of wood, but at the same time reduces density and mechanical properties (except hardness). That results in brittleness and increased cracking. By prolonging the duration of process and due to chemical changes, the colour of the wood also changes. The observed changes in the structure of cell walls can mainly be attributed to the degradation of hemicelluloses because they are thermally less stable than cellulose, while lignin decomposes over a broad range of temperatures. The occurrence of colour changes and its chemistry is a very complex phenomenon and has been researched for a long time. In heat treatment, drying is used to prevent discoloration, while in the case of steaming and thermal modification, changes are deliberately induced, especially in species with false heartwood. Prolonged exposure to heat darkens the wood (which is often the aim) or equalizes the colour of the false heartwood and the surrounding wood material. With some types of wood, steaming can cause side effects such as reduced strength or appearance of cracks, and in that case cooking treatment is applied (Pervan *et al*., 2008).

In general, heat treatment of wood can be defined as a process that improves its current properties or alters the wood in such a way that it gains new properties. At the end of its life, such wood should not represent a danger to the environment greater than unmodified wood (Hill, 2006). Treated wood should be non-toxic and should not emit any hazardous substances during processing or in later stages of use. Due to the increasing use, there is a need to research the impact of harmful substances from such processes on the environment and ecology in general.

2 CHANGES AND DEGRADATION OF MAIN CHEMICAL COMPONENTS
2. PROMJENE I RAZGRADNJA GLAVNIH KEMIJSKIH KOMPONENATA

Heat treatment causes changes in the cell wall polymers structure, influencing their properties and it also directly affects the chemical degradation of wood, *i.e.* the degradation of its main components (hemicelluloses, cellulose, lignin) and extractives (Figure 1). Chemical changes caused by heat depend on the duration and temperature of the treatment, where temperature is the main factor (Bourgois *et al*., 1989). At temperatures between 20 and 100 ºC, the wood is dried, starting with the loss of free water and ending with the loss of bound water until desired moisture content (MC)

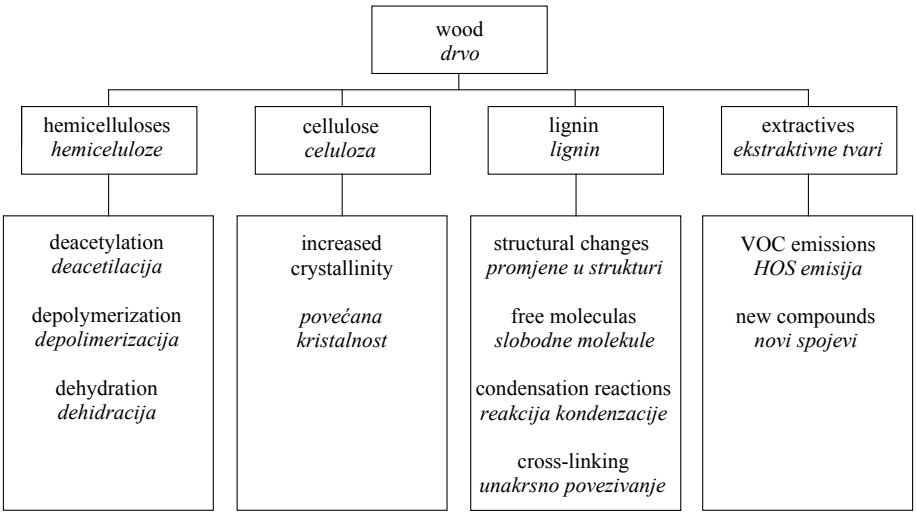

Figure 1 Chemical changes occurring in the main components of wood due to heat treatment (Esteves and Pereira, 2009)
Slika 1. Kemijske promjene glavnih komponenata drva prouzročene toplinskom obradom (Esteves and Pereira, 2009.)

is achieved. At 180 to 250 ºC, which is commonly used for heat treatment, wood undergoes major chemical transformations, and at temperatures above 250 ºC the charring process begins with the formation of CO_2 and other pyrolysis products (Esteves and Pereira, 2009).

Kubovský *et al*. (2020) performed thermal modification of wood at temperatures from 160 to 210 °C and concluded that the higher the processing temperature, the better the durability, stability and biological properties of wood became (except for the mechanical properties that are lowered in such conditions). FT-IR analysis and HPLC revealed that heat treatment leads to an increase in the number of carboxyl groups in lignin with splitting of aliphatic side chains and demethoxylation. Degradation of hemicelluloses results in deacetylation, and the acetic acid released catalyses the hydrolysis of polysaccharide chains. This process is more visible at a temperature of 180 °C, which leads to a decrease in molecular weight and an increase in polydispersity. In addition to the splitting of polysaccharide chains, crosslinking reactions take place at 210 °C, with an increase in molecular weight and polydispersity. Fengel and Wegener (1984) state that the hemicelluloses are the most sensitive of the structural components to thermal degradation, as they are the first to degrade at temperatures of 160 to 260 °C. The reason for this is their low molecular weight and branched structure, which helps faster degradation compared to other components present in wood. However, Funaoka *et al*. (1990) observed that the degradation of hemicelluloses begins as early as 100 °C, with the cellulose and lignin contents remaining constant up to 150 °C. Degradation of cellulose results in reduction of hydroxyl groups and the formation of O-acetyl groups. By subsequently creating cross-links between the wood fibres, the wood becomes more hydrophobic. At the same time, cellulose is degraded to form acetic and formic acid, and the lignin ratio increases as extracted substances from hemicelluloses and cellulose bind to it (Alén *et al*., 1996; Gailliot, 1998). The crystallinity of cellulose is increased by thermal modification up to 200 °C due to the degradation of its amorphous part, which is not only related to temperature but also to the duration of heat treatment, and results in reduced availability of hydroxyl groups to water molecules (Wikberg and Maunu, 2004; Yildiz and Gümüskaya, 2007). Mitsui *et al*. (2008) suggested that hydroxyl groups in cellulose degrade in the following order: amorphous, semi-crystalline, and crystalline regions. Acetic and formic acid, formed during the heat treatment, increase the hydrolysis of hemicelluloses and cellulose, while the degree of polymerization of polysaccharides decreases (Kačíková *et al*., 2013; Nuopponen *et al*., 2004). Degradation of lignin is visible at lower temperatures but takes place at a lower rate than other polysaccharides, while at higher temperatures there is a condensation reaction and an increase in molecular weight. In a study conducted by Bourgois and Guyonnet (1988) on maritime pine (*Pinus pinaster*), the results showed that lignin content at the same temperature increased when longer process duration was applied. The same results were reported for other species by Zaman *et al*. (2000) and Esteves *et al*. (2008b). Kotilainen (2000) and Manninen *et al*. (2002) conducted a research on heat-treated hornbeam (*Carpinus betulus* L.) and Turkish fir (*Abies bornmuelleriana* Mattf.), where the contents of hemicelluloses significantly decreased with increasing temperature and duration of treatment. The reduction in hemicelluloses content was found to be greater than in cellulose content. A significant reduction in hemicelluloses content began at temperatures above 180 °C. Due to the structural heterogeneity of hemicelluloses, it is difficult to detect their behaviour under the influence of heat. Xylan (pentosan) is the most reactive wood hemicellulose, and pentosans are generally very sensitive to degradation and dehydration reactions. For example, the content of pentosan in pine wood treated for 7 hours at 130 °C decreased from 11 % to 9.1 % (Potutkin and Shirayena, 1975). As stated, the cellulose content was reduced compared to the control sample, but to a lesser extent compared to hemicelluloses. The largest and the smallest changes in cellulose content in all heat treatments were found at 170 °C for 4 hours and at 210 °C for 12 hours (Tumen *et al*., 2010). By increasing the temperature above 200 °C, thermal degradation of cellulose and the formation of volatile products take place rapidly (Manninen, 2002; Fengel and Wegener, 1989). Kotilainen (2002) concluded that the most intensive thermal degradation takes place in the temperature range from 200 °C to 260 °C. The lower thermal stability of hemicelluloses compared to cellulose is usually explained by a lack of crystallinity.

Changes in cell walls and individual chemical components of wood also lead to changes such as discoloration. Pervan *et al*. (2006) and Koch (2008) state that it is one of the most common phenomena that occurs during steaming and drying of wood. It is assumed that the main factors involved in the chemical reactions are oxidation and condensation of phenolic compounds (Wengert, 1990), but colour changes can also be caused by hydrolysis, although in many cases the reasons are unknown. Investigating the influence of hemicelluloses, lignin and extractives on the colour, Sundqvist and Moren (2002) found that extractives are involved in the formation of colour during hydrothermal wood processing. This was also proven by Wiberg (1996) in his study of Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*). The results showed that during the drying process the extractives move towards the

surface, making it redder, while the substances that give a yellow colour remain inside. The above authors conclude that this colour change is due to the concentration of low molecular weight carbohydrates and nitrogen on the surface during wood drying. Similar results were obtained by Sandoval-Toress *et al*. (2012) for vacuum-dried samples of oak (*Quercus robur* L.). They concluded that saturation increases with increasing temperature and that the yellow hue is increased by the thermal effect, with red hue associated with extractives. Colour changes were measured on the surface because substances such as sugars, phenols, antioxidants and others move from the centre to the surface layer of wood by evaporating water and are retained there. According to White and Dietenberger (2001), heat darkening of wood is caused by thermal degradation of hemicelluloses and lignin and can start at temperatures below 65 °C (depending on wood pH, moisture content, heating medium and exposure period and wood type). Kosikova´ *et al*. (1999) used ^{13}C CP MAS NMR (carbon cross polarization magic angle spinning nuclear magnetic resonance) spectroscopy to characterise structural changes in cell wall polymers during drying and determined partial depolymerisation of lignin and changes in amorphous and crystalline parts of cellulose. This happened when wood was treated with superheated steam at a temperature of 135 °C, which also affected the colour change. Pervan *et al.* (2008) state that the heating is known to reduce sapwood brightness (in all species), with varying intensity, depending on the humidity, temperature and duration of the process. The influence of the cooking process on the colour change of walnut was studied by Charrier *et al*. (1998). The colour of sapwood changed in parallel with the duration of the process, with the decrease in the value of brightness following the increase of the value of the red component. Brauner and Conway (1964) also studied the colour change in walnut and stated that the black walnut (*Juglans nigra*) sapwood changes colour after 4 to 6 hours of steaming at 100 to 120 ° C. In this particular case the darkening was uniform in width and depth. Gray (1961); Hon (1981); Haluk *et al*. (1991); Klumpers *et al*. (1994); Abe *et al*. (1994); Hon and Shiraishi (2000); White and Dietenberger (2001); Sundqvist (2002); Alén *et al*. (2002); Charrier *et al*. (2002); Koch *et al*. (2003); Stenudd (2004); Thompson *et al*. (2005); Rowell (2006) suggest that the main colour change reactions due to wood steaming are oxidation, hydrolysis, polymerization of phenolic compounds, enzymatic reactions, and thermal degradation of lignin and hemicelluloses.

3 ENVIRONMENTAL ISSUES
3. EKOLOŠKA PROBLEMATIKA

The increasing use of heat-treated materials has made it necessary to investigate the impact of harmful substances of such treatments on the environment, especially on the possible formation of ozone in the lower atmosphere or tropospheric ozone that adversely affects humans, animals and plants. The toxicity of harmful substances was studied, and the types of compounds released during the process were listed. Worldwide standards set strict limits on emissions of volatile organic compounds that occur during wood drying (Zwick *et al*., 1997). In Croatia, these restrictions are currently prescribed by the Regulation on limit values for air pollutants from stationary sources [NN 42/2021] and the Regulation on limit values for exposure to hazardous substances at work and on biological limit values [NN 13/2009]. Exposure limit values are average concentrations of substances (gases, vapours, aerosols, dusts) in the air at the workplace in the breathing zone of workers at a temperature of 20 °C and air pressure of 1013 mbar, which according to current knowledge do not cause damage to health in everyday eight-hour work time. Table 1 shows the exposure limit values for substances that are most often produced by heat treatment of wood, the possible hazards they cause and their harmful effects.

Studies on volatile organic compounds (VOCs) in the atmosphere have shown that atmospheric chemistry

Table 1 Exposure limit values for harmful substances from heat treatment of wood [NN (42/2021)]
Tablica 1. Granične vrijednosti izloženosti štetnim tvarima zbog termičke obrade drva [NN 42/2021]

Substance *Tvar*	**Exposure limit values, mg/m³** *Granična vrijednost izloženosti,* mg/m³	**Hazards and harmful effects** *Opasnost i štetno djelovanje*
Acetaldehyde *acetaldehid*	90	Irritating / *nadražujuće* Extremely flammable / *vrlo zapaljivo* Carcinogenicity / *kancerogeno*
Formaldehyde *formaldehid*	2.5	Very toxic / *vrlo otrovno* Carcinogenicity / *kancerogeno*
Methanol/ *metanol*	260	Very toxic / *vrlo otrovno* Harmful to skin / *štetno djelovanje kroz kožu* Highly flammable / *lako zapaljivo*
Formic acid / *mravlja kiselina*	9	Corrosive / *nagrizajuće*
Acetic acid / *octena kiselina*	25	Corrosive / *nagrizajuće*

is complex and that their degradation and transformation reactions can occur in the gas and water phases (Grosjean *et al*., 1992; Faust, 1994). Organic extracts emitted during the wood drying process include terpenes, methanol, acetic acid, formaldehyde, resin acids and fatty acids (McDonald *et al*., 1999b). In conventional wood drying kiln, such compounds are released into the atmosphere, and the resulting blue haze and unpleasant odours can cause concern for the environment and in some cases health problems (Cronn *et al*., 1983). Harley and Cass (1994) indicate that some toxic compounds found in ambient air are formed in the atmosphere as oxidation products of other volatile organic compounds, and those oxidation products are, for example, ***α***-pinene, formaldehyde, acetone, pinonaldehyde, and glyoxal (Grosjean *et al*., 1992). Terpenes have also been found to react rapidly with ozone, hydroxyl or nitrate radicals to give various organic compounds. McDonald *et al*. (2002) conducted a research on pine and found that volatile components condense with water vapour during the vacuum drying process. Those VOCs were mainly monoterpenes, methanol, formaldehyde, furfural and diterpenes. Additionally, it was observed that the amount of oxygen and carbon suggest that it is necessary to reduce the concentration of organic compounds. Bicho *et al*. (1966) also found harmful substances such as formic, acetic and levulinic acid, furfural, hydroxymethyl-furfural, formaldehyde and acetaldehyde in the analysis of condensates at vacuum drying of ash, beech and oak. The analysis of condensate, obtained during the drying of California pine (*Pinus radiata*) in an experimental vacuum dryer, showed that the condensate contained 10 % monoterpenes, and alcoholic monoterpenes (endoborneol, ***α***-terpineol and 1,4-terpineol), methanol, acetic acid, formaldehyde, furfural and diterpenes were found as organic compounds. The values of BOD (a measure of the amount of oxygen required by bacteria to decompose organic components present in water/wastewater) and COD (chemical oxygen demand; i.e. the total measurement of all chemicals (organic and inorganic) in water/wastewater), for experimental condensate from vacuum kiln dryer indicate the fact that before the condensate is discharged, its treatment will be required in order to reduce the concentration of organic compounds (McDonald *et al*., 1999). According to Mayes and Oksanen (2002), VOC emissions from heat-treated wood are lower than from air-dried wood, as terpene emissions such as pinene, camphene and limonene during drying of untreated wood are higher than for treated wood. Similar results were obtained by Manninen *et al*. (2002), whose results show that VOC emissions from air-dried white pine (*Pinus sylvestris*) were about eight times higher than those of heat-treated wood and consisted of ***α***-pinene, 3-carnene, hexanal, 2-furancarboxaldehyde, acetic acid and 2-propanone. Peters *et al*. (2008) state that furfural and 5-methylfurfural are the main emission products from treated wood and Marutzky and Roffael (1977) conducted a study in which, by applying heat treatment independently of temperature, formaldehyde was formed due to the degradation of lignin and other hydrocarbons.

4 CONCLUSIONS
4. ZAKLJUČAK

Heat treatment is one of the most common wood treatments that results in changes in the structure of the cell walls, degradation of major components (hemicelluloses, cellulose and lignin) and extractives, and changes in chemical composition. The changes that occur due to heat treatment depend on the temperature and duration of treatment, and to a large extent on a number of external conditions due to which the results differ for the same wood species. Treated wood results in lower hygroscopicity with a positive effect on dimensional stability and durability, but very often with a negative effect on mechanical properties, which are reduced (except for hardness which causes an increase in brittleness). In the research on the degradation of the main chemical components of wood, most results showed that hemicelluloses are the most unstable components and they are the first to decompose due to their low molecular weight and branched structure. This is followed by cellulose, which is thermally more stable due to its crystallite structure. Lignin is more unstable at lower temperatures, while at higher temperatures there is an increase in molecular weight. The main components also affect the colour change, which is a common occurrence during thermal modification. The most common causes of discoloration are reactions of phenolic compounds and reactions and thermal decomposition of hemicelluloses and lignin. Although heat treatment is the most environmentally acceptable process, because it is performed under controlled conditions in the presence of heat and moisture without chemical additives, many volatile organic compounds (VOCs) are formed and released, such as alcohols, resins, terpenes, formic and acetic acid, resins and fatty acids, aldehydes, furfurals. Due to the increased use of heat-treated wood and compounds that are formed in the atmosphere during the process, it is necessary to investigate its impact on ecology. It should be emphasized that after heat treatment wood is environmentally acceptable, which means that it is not toxic and that at the end of its life cycle it does not pose a danger to the environment.

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Corresponding address:

NIKOLA ŠPANIĆ
University of Zagreb, Faculty of Forestry and Wood Technology, Svetošimunska cesta 23, 10000 Zagreb, CROATIA, e-mail: nspanic@sumfak.unizg.hr

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