Organic Compounds Released by Thermal Decomposition of Wood Composite

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Abstract

Combustion of biomass materials based on wood combined with synthetic polymers was studied. Material types included plywood and wood chipboard. Wood chips prepared from hardwood – beech and softwood – spruce were used for comparison. Combustion process was characterized by thermal analysis – thermogravimetry and differential scanning calorimetry. Pyrolysis gas chromatography with mass spectrometric detection was used for determination of organic compounds and their fragments formed during thermal decomposition. Attention was paid to organic compounds contained in wood-polymer composites.

Keywords: organic geochemistry, biomass, pyrolysis-GC/MS, thermal analysis TG/DSC, plywood, chipboard

Introduction

The amount of waste composite materials based on wood and synthetic polymers (composite wood materials) is currently increasing. They are extensively used for furniture, floating floors, structural elements (floors, walls, boarding, fencing, parts of transport vehicles, etc.). Laminated and high pressure laminated materials contain decorative paper with aminoplastic resins. Foil materials, e.g. waterproof plywood, are formed by film layers containing phenols. Almost all composite wood materials contain synthetic adhesives based on phenol-formaldehyde (PF), urea-formaldehyde (UF) and melamine-urea formaldehyde (MUF) resins. Phenol-formaldehyde resin is used to manufacture plywood for exterior applications because of its excellent water resistance. Phenol-formaldehyde tends to be more chemically stable and less susceptible to hydrolysis than UF or melamine-urea formaldehyde resin (MUF), and it is considered waterproof, unlike UF [1]. Additionally, polyvinylacetate (PVAc) has been added as a replacement for melamine-based resin systems to reduce the formaldehyde emissions from the adhesives used between plywood and fancy veneers [2]. Flame retardant composite materials for the interior of buildings and vehicles contain flame retardant from synthetic polymers.

Waste wood composite materials are recycled again or they can have power utilization. There is very little information on the content of organic substances in the emissions released by combustion and pyrolysis of wood composite materials (plywood and wood chipboard). The paper deals with the identification of organic compounds that are formed during the thermal decomposition of wood composite materials and determining the differences in the chemical composition of the composite materials and natural wood.

Materials and methods

Samples of waste resin bonded plywood and wood chipboard were obtained from the company TRESTLES, a.s. Dětmarovice, which produces industrial shelves. A sample of plywood was obtained from the store Hornbach. For comparison of the products formed during pyrolysis of wood chipboard and plywood, pure hardwood (beech) and softwood (spruce) were analysed. The samples were crushed and milled to a size of approximately 100 µm.

Analysis was performed by pyrolysis gas chromatography with mass spectrometry detection (py-GC/MS) using Pyroprobe 5200, CDS Analytical. The sample was pyrolyzed for 10 seconds at a temperature of 400°C, with the rate of temperature increase of 20°C/ms. Proximate analysis was performed according to the standard methods: ISO 602 Determination of ash content, EN 15403 Solid recovered fuels – Determination of ash content, EN 15402 Solid recovered fuels – Determination of volatile matter content, ISO 1928 Determination of combustion heat using the calorimetric method in pressure vessel and calculating the calorific value. Thermal analysis was performed using the TG/DSC 2 Thermoanalyzer Mettler Toledo. Samples of approximately 10 mg were heated in aluminium oxide crucibles of 70 µl volume. Measurements were performed with heating rate of 10°C/min in the air atmosphere. Thermogravimetric curve (TG) and differential scanning calorimeter curve (DSC) were recorded simultaneously in the temperature range from 25 to 1000°C.

Results

The results of proximate analysis (wt.%) of composite wood waste (CH – chipboard, P – plywood) are as follows: ash (815ºC) CH – 0.91, P – 0.30, volatile matter CH –

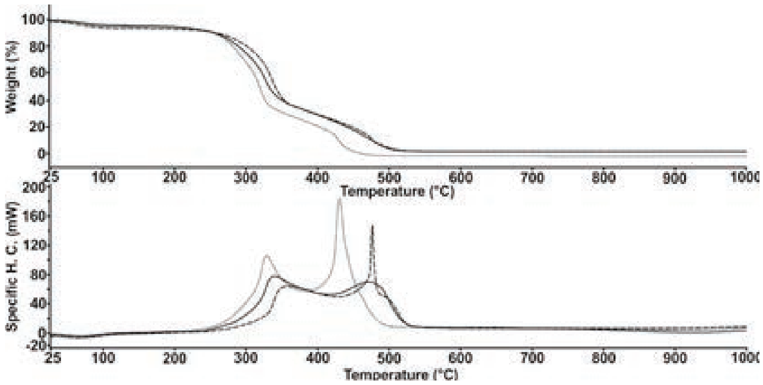

Fig. 1. Thermal analysis - TG/DSC (chipboard – solid line, plywood – dashed line, wood chips – dotted line, H.C. – heat capacity)

Rys. 1. Analiza termiczna - TG/DSC (płyta drewniana – linia ciągła, sklejka - linia przerywana, zrębki - linia z kropkami, H.C. - pojemność cieplna)

83.26, P – 82.15, fixed carbon CH – 15.83, P – 17.52. Combustion heat (MJ/kg) for chipboard and plywood was 18.91 and 17.54, respectively. The results of TG/DSC analysis are presented in Fig.1. The TG curves of three samples are in the upper part, DSC curves in the lower part of the figure: CH – chipboard (solid line), P – plywood (dashed line), W – wood chips (dotted line).

The decomposition of organic compounds utilized for production of chipboard and plywood begins at the temperature around 70°C. Slow weight loss between 70°C and 250°C is caused by volatilization of formaldehyde (CH – 4.4 wt.%, P – 15.2 wt.%). Most of the weight loss of the wood chips occurs between 250 and 500°C. The upper limit of this interval is higher for samples of chipboard and plywood, and the decomposition process ends around 550 °C. It is possible to distinguish the two stages of thermal decomposition. The first phase with peak temperatures between 330 and 360°C is connected with weight loss representing approximately two thirds of the original sample weight. The beginning of this phase at 250°C indicates the decomposition of hemicellulose. The decomposition of cellulose begins with an increasing temperature. Peak temperatures of this reaction are increased for chipboard and plywood compared with wood chips (W – 328°C, CH – 340°C, P – 355°C).

The loss of weight during this phase is the highest for wood chips and the lowest for plywood (W – 73%, CH – 70%, P – 63%). The second phase begins at the temperature from 380 to 430°C. This reaction indicates the intensive decomposition of lignin. The relative positions of temperature peaks are similar to the first phase: W – 430°C, CH – 472°C, P – 478°C. In addition, the DSC curve of the plywood sample exhibits the sharp peak superimposed over the peak at 478°C. The values of the weight loss during this step corresponds approximately to one quarter of the original sample weight: W – 24 wt.%, CH – 24 wt.%, P – 22 wt.%. The values of heat released during thermal decomposition were obtained by integration of DSC curve over the interval of both phases unresolved: W – 9134 J/g, CH – 8444 J/g, P – 6434 J/g. The shapes of TG and DSC curves are influenced by flame retardants utilized in production of chipboard and plywood. The presence of melamine was confirmed by the method of py-GC/MS at a temperature of 750°C. Flame retardants are components of adhesives (phenol-formaldehyde resin and urea-formaldehyde resin with impregnated paper containing melamine). The presence of melamine (2,4,6 triamino-1,3,5 triazine) and urea substantially decreases flammability of these materials and shifts the complete thermal degradation of plywood and chipboard over 480 °C. Similarly to our results, it was determined that most of the weight loss of the plywood and wood chipboard occurs between 250 and 470 – 480°C, the thermal degradation of wood particles in oxygen atmosphere occurs over a wide temperature range approximately from 250 to 430°C [3].

In pyrolysates of plywood and wood chipboard, a total of 425 organic compounds were identified belonging to the groups: alkanes, cycloalkanes, alkenes, cykloalkenes, alkines, alcadiens, cycloalkadiens, hydrazines, compounds containing benzene, aldehydes, ketones, alcohols, pyrans, furans, benzofurans, compounds containing heterocyclic a aliphatic nitrogen, compounds containing phenols, PAHs (polycyclic aromatic hydrocarbon) a derivatives of PAHs, carboxylic acids, acetates, alkanoates, oxazines, saccharides, anhydrosaccharides, phytosterols, terpens and their alteration products. The concentrations of the individual groups of organic compounds (μg/g) found in pyrolysates of wood chipboard, plywood and wood are shown in Table 1.

The highest concentrations of organic compounds in samples of plywood and wood chipboard are reached by PAHs and their derivatives followed by sterols, anhydrosaccharides and their alteration products, compounds containing benzene and compounds containing phenols. In the concentration range of 100 to 300 μg/g, pyrolysates of plywood and wood chipboard contain alkanes, cycloalkanes, alcadiens, aldehydes, ketones, alcohols, furans, pyrans, benzofurans, compounds containing heterocyclic nitrogen.

Tab. 1. Concentrations of organic compounds (µg/g) in pyrolysates of wood chipboard, plywood, hardwood (HW) and softwood (SW), components typical for wood composites are in bold

Tab. 1. Zawartość składników organicznych (µg/g) w płytach drewnianych po pirolizie, sklejce, drewno twarde(HW) i drewno miękkie (SW), typowe składniki kompozytów drewnianych oznaczono boldem

Organic matter	Chipboard	Plywood	HW	SW	Difference from SW	
			Beach	Spruce	Chipboard	Plywood
Hydrazines	2.0	1.1	19.3	25.2		
Compounds containing benzene	**389.1**	**225.9**	36.0	33.6	355.4	192.2
Alkanes, cycloalkanes	**289.0**	**167.8**	109.2	145.2		
Alkenes, cykloakenes	276.7	160.6	597.8	433.1		
Alcadiens, cycloalcadiens	**108.0**	**62.7**	22.6	15.8	92.2	46.9
Alkines	**29.9**	**17.3**	3.2	2.3	27.5	14.9
Aldehyde and ketones	99.2	57.6	62.6	88.6		
Alcohols	102.4	59.9	110.5	62.6		
Furans, pyrans, benzofurans	158.2	107.5	210.8	169.8		
Compounds containing heterocyclic nitrogen	**247.2**	**143.5**	71.5	61.9	185.2	81.5
Compounds containing aliphatic nitrogen	43.8	25.4	45.2	23.3		
Compounds containing phenols	**339.7**	**197.2**	111.3	121.4	218.2	75.8
Oxazines	**3.9**	**2.3**	ND	ND	3.9	2.3
PAHs and their derivatives	**788.6**	**457.8**	384.1	410.2	378.3	47.5
Carboxylic acids	292.2	169.6	370.3	412.9		
Sterols, anhydosaccharides and alteration products	418.6	296.0	803.8	885.0		
Acetates, alcanoates, oxiranes	29.8	17.3	28.7	35.4		
Unresolved organic matter	362.8	349.5	722.0	868.0		
Total	3982	2519	3710	3795	1257	459

Hydrazines, alkines, compounds containing aliphatic nitrogen, oxazines, acetates, alkanoates, oxiranes can be found in pyrolysates in the concentration of up to 50 µg/g.

Pure natural wood, hardwood (beech) and softwood (spruce) shows the highest concentration in the compounds from the group of sterols, anhydrosaccharides, terpens and their alteration products, followed by alkenes and cykloalkenes, PAHs and their derivatives, and carboxylic acids. The concentration of sterols, anhydrosaccharides, terpens and their alteration products together with alkenes are 2-3 times higher in natural wood samples than in samples of plywood and wood chipboard. Carboxylic acids in samples of natural wood are 1.2 to 2.4 times higher compared to samples of plywood and wood chipboard.

There are significant differences between pyrolysates of natural wood, plywood and wood chipboard in concentrations of hydrazines, compounds containing benzene, alcadiens and cycloalcadiens, alkines and compounds containing phenols, compounds containing heterocyclic nitrogen, oxazines, PAHs and their derivatives. These differences are significant for the determination of natural and synthetic origin of organic compounds in the individual pyrolysates.

PAHs in plywoods and chipboard are predominantly formed by pyrolysis of the original components of wood during of the aromatizing and condensation reactions [4]. Lignin, carboxylic acids, and saccharides participate in formation of PAHs and their derivatives [5]. PAHs in pyrolysates of plywood and wood chipboard (at the pyrolysis temperature of 400°C) occur during demethoxylation and demethylation of lignin [6]. PAHs are formed of the carboxylic acids within cyclization reactions [7]. Furthermore, the reaction between the hydrocarbon free radicals also participates in the formation of PAHs and their derivatives. Part of PAHs can also results from reactions during thermal decomposition of synthetic polymers and adhesives. The quantity of PAHs and their derivatives formed from synthetic polymers and adhesives in samples of plywood and wood chipboard was determined from the difference in concentrations of PAHs in wood composites and PAHs determined by pyrolysis of softwood (Table 2). Concentrations of PAHs and their derivatives are 2 times lower for samples of natural wood in comparison with the samples of plywood and wood chipboard.

Simultaneously, compounds containing phenols are released with PAHs, e.g. dimethoxyphenols, dimethylphenols, acetophenone, vanillin, vanillic acid and accompanying compound indicating lignin contained in the wood and its thermal changes during pyrolysis. In addition to compounds containing phenols arising from lignin, phenolic compounds indicating the presence of phenol-formaldehyde adhesives (resins) were determined in both pyrolysates. These compounds include 6-benzyl-4-methyl-2-vinylphenol, 2-ethyl-5-methylphenol, trimethylphenols, 4-ethyl-3-methylphenol, isopropoxylphenols allylphenols. The concentration of compounds containing phenols from synthetic adhesives is 43.4 μg/g for plywood and 83.3 μg/g for wood chipboard.

Formaldehyde from resins was not identified. Formaldehyde reacted with phenols and primary amines to form benzooxazines, which are part of the organic group of oxazines. Occurrence of oxazines and benzooxazines is typical of synthetic materials (Table 2).

Compounds containing benzene (aromatic hydrocarbons) include compounds from the group of BTEX (benzene, toluene, xylenes, ethylbenzene), methylated and ethylated forms of benzene. In total, 22 organic compounds containing benzene were identified in plywood and wood chipboard. Aromatic hydrocarbons result from the decomposition of wood, their exact origin is not fully understood yet. The formation of aromatic hydrocarbons is partly caused by pyrolysis of proteins, lignin, tannin, and carboxylic acids [8]. Pyrolysis of unsaturated fatty acids results in the formation of toluene and ethylbenzene [9]. In the case of thermal decomposition of composite wood materials, synthetic polymers and adhesives significantly contribute to the formation of aromatic hydrocarbons. Part of the compounds containing benzene is formed by the decomposition of adhesives, it is the case of, e.g. trimethylbenzenes, 1,1'-methylenebis (2-methyl) benzene, trimethoxybenzenes. Concentrations of compounds containing benzene in pyrolysates of natural wood are about 10 times lower compared to pyrolysates of composite wood materials. The concentration of compounds containing benzene formed during the thermal degradation of adhesives is 192.3 μg/g for plywood and 355.5 μg/g for wood chipboard.

Carboxylic acids contained in plywood and wood chipboard range from 169.7 to 292.2 μg/g. The presence of the “synthetic component” indicates occurrence of benzenedicarboxylic acids (ester bis(2-ethylhexyl)-1,4-benzenedicarboxylic acid). Other identified acids are generated during the decomposition of wood. Significant carboxylic acids of natural origin include hexadecanoic, octadecanoic, tetradecanoic, oleic, palmitoleic, octadecenoic, tricosenoic, and their esters. The presence of 7-oxodehydroabietic acid fragments together with accompanying fragments retene and methyldehydroabietate, pinene indicates that the original wood for the production of plywood and chipboard came from conifers.

Furans, and their substituted forms are generated during the thermal decomposition of cellulose [10]. In total, 20 organic compounds from the groups of furans, pyrans and benzofurans were identified. The ratio between dibenzofuran and benzofuran (1.43) indicates the intense decomposition of cellulose and release of saccharides [11]. In pyrolysates, 2,3-dihydrobenzofuran, benzofuran, furfural and levoglucosan dominate. The presence of levoglucosan in the thermal pyrolysis indicates a thermal change of cellulose. The dominance of benzofuran in pyrolysates indicates materials rich in cellulose.

Alkanes and alcohols are formed by pyrolysis of waxes contained in wood. To a lesser extent, alkanes can be produced by thermal decomposition of synthetic polymers and additives. In total, 14 representatives from the group of alcohols were identified. The dominant representatives for both pyrolysates are indenol and phenantrenol. Concentrations of alcohols in plywood and wood chipboard are identical to concentrations of alcohols in samples of natural wood. Alcohols contained in pyrolysates of plywood and wood chips are of natural origin. The dominant alkanes and cycloalkanes with concentrations above 15 μg/g in wood composites include tetrapentacontane, cyclotriacontane and 2-methyleicosane. Alkanes and cycloalkanes derived from thermal degradation of synthetic polymers and adhesives in plywood and wood chipboard represent 22.5 and 143.8 μg/g. Alkenes are formed during thermal degradation of alkanes and alkanoates. In comparison with wood composites, concentrations of alkenes in natural wood are about two times higher (433.2 – 597.8 μg/g). Nearly all alkenes in pyrolysates of plywood and wood chipboard can be considered natural. From alkenes, tricyclo (8,2,2,2 (4.7) hexadecylcitric-2,4,6,8,10,12,13,15-oktaene, eicosenes and octadecenes dominate in pyrolysates of chipboard and wood plywood.

Compounds containing nitrogen include two forms – nitrogen bonded aliphatically and heterocyclically. Aliphatic nitrogen compounds are represented by the presence of nitrils. The concentration of nitrils for wood chipboard is almost two times higher (43.85 μg/g) than for plywood

(25.46 μg/g). Benzonitrile, 2-methylbenzonitrile, benzylnitrile and hexadecannitrile occur at the highest concentration. Nitrils comes from the reaction of long-chain fatty acid with derivatives of nitrogen [12] or they are formed by dehydration of aromatic amides or amines [13], [14]. Benzonitrile is probably derived from the thermal decomposition of aromatic amines [15]. Compounds containing heterocyclic nitrogen (pyridine, pyrrole, diketopyrrols, imidazoles, methylated forms of pyrrole, indole, pyridin) identified in pyrolysates of plywood and wood chipboard come from thermal degradation of proteins, amino acids and aminosaccharides. Pyrrole results from the decomposition of amino acids: proline, hydroxy proline and glutamine. Substituted pyrrole (2,5-dimethyl-1H-pyrrole and 2-methyl-1H-pyrrole) are formed during pyrolysis of porfyrines, which are components of wood chlorophyll. Indole is the pyrolysis product of tryptophan. Imidazoles come from pyrolysis of histidine. Pyrazines originate from degradation of proline [16]. In addition to compounds with aliphatic and heterocyclic nitrogen, which was contained in the original matrix of wood, a lot of nitrogen compounds originating from adhesives – urea-formaldehyde and melamine-formaldehyde resins were identified in plywood and wood chipboard. Compounds containing nitrogen released from adhesives include urea, 3,5-dihydroxybenzamide, 4-chlorpyridinesulfoamide, n,n-dimethylurea, n-methoxy-n-acetyl-2-carboxymethoxyethylamine, n-ethyl-n,n-dimethyl-1,2-ethanediamine and compounds containing carboimide. These compounds are released during the thermochemical conversion – combustion, pyrolysis, gasification. Verification of the origin of the compounds was performed by comparing the concentration of compounds containing heterocyclic nitrogen in pyrolysates of natural wood. The concentration of compounds containing heterocyclic nitrogen in natural wood is 2.3 to 3.4 times lower compared to plywood and wood chipboard; they range from 61.9 to 71.6 μg/g. The total concentration of nitrogen compounds derived from the degradation of adhesives is 81.6 and 185.3 μg/g for the sample of plywood and wood chipboard, respectively.

Adamantane, saponine, carvone, coniferylaldehyde, camfolenal, copaene, methyldehydroabietate, pinene, tocopherol, terpineol, 3-acetylpyrrole, calacorene, stigmasterol, stigmasta-3,5-diene, limonene, sitosterol, squalene, quercetin, cymene, syn-3-methylbutylaldoxime, bicyclosequiphelandrene were identified from groups of sterols, terpenes, volatile oils, and alteration products. The concentration of the above-mentioned products for plywood and wood chipboard were 224.8 and 326.6 μg/g, respectively. From the synthetic compounds, bis(2-ethylhexylphthalate) in the concentrations of 10.8 and 13.9 μg/g were identified in the two repeated analyses for the samples of wood chipboard and plywood, respectively. The concentration of sterols, terpenes, volatile oils, and alteration products is higher in pyrolysates of natural wood; it ranges from 803.8 (hardwood) to 885.0 μg/g. Pyrolysates of natural wood contain multiple products derived from volatile oils, terpenes, sterols and alteration products, as a result of its structure chemically unchanged.

Conclusions

Pyrolysis of wood chipboard and plywood resulted in the formation of more than 420 organic compounds that can be identified. During pyrolysis of natural wood, 440 organic compounds were formed. The most abundant organic compounds in plywood and wood chipboard include compounds containing benzene, PAHs, phytosterols, terpenes and alteration products, compounds containing phenol. Pyrolysis products (organic compounds) resulting from pyrolysis of wood chipboard and plywood have a double origin – natural and synthetic. Organic compounds originating from synthetic polymers and adhesives are of synthetic origin. Organic compounds which originate from the pyrolysis of wood components (cellulose, lignin, proteins, waxes etc.) are of natural origin.

The concentration of organic matter produced during the degradation of the natural compound ranges from 1292.7 (wood chipboard) to 3545.6 μg/g (plywood). The quantity of organic matter derived from thermal degradation of synthetic polymers and adhesives ranges from 436.4 (plywood) to 1226.6 μg/g (wood chipboard). The percentage of the organic matter from the degradation of synthetic polymers and adhesives constitute 11.0 % for plywood and 48.7 % for wood chipboard. Higher concentrations of synthetic polymers with wood chipboard are the result of a greater content of synthetic adhesives and polymers.

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Związki organiczne uwalniane w czasie termicznego rozkładu kompozytów drzewnych

Zbadano spalanie materiałów biomasowych na bazie drewna w połączeniu z syntetycznymi polimerami. Materiałz zawierały sklejkę i drewniane płyty wiórowe. Do porównania użyto zrębków z drewna liściastego - drewna bukowego i miękkiego - świerka. Proces spalania scharakteryzowano prowadyc analizą termiczną - termograwimetrią i różnicową kalorymetri€ skaningową. Do oznaczenia zawartosci związków organicznych i ich pochodnych powstałych w trakcie procesu użyto pirolitycznej chromatografii gazowej z detekcją spektrometryczną. Zwrócono uwagę na związki organiczne zawarte w kompozytach drewno-polimer.

Słowa kluczowe: geochemia organiczna, biomasa, piroliza - GC / MS, analiza termiczna TG / DSC, sklejka, płyty drewniana