

**Agata STACHOWIAK-WENCEK, Włodzimierz PRĄDZYŃSKI,
Magdalena MATEŃKO-NOŻEWNIK**

EMISSION OF VOLATILE ORGANIC COMPOUNDS (VOC) FROM UV-CURED WATER-BASED LACQUER PRODUCTS

The paper presents the results of chamber tests conducted on UV-cured water-based lacquer coatings applied on different wood species. Volatile organic compounds were transferred onto Tenax TA and then they were assayed by gas chromatography coupled with mass spectrometry and thermal desorption. It was found that UV-cured water-based coatings may be potential sources of the emission of volatile substances and thus pollute indoor air. Concentrations of these compounds in the chamber depended on the type of the lacquer system used and the type of finished surface. The highest emission of volatile substances was recorded from coatings applied onto the surface of pine wood. The coatings tested released aldehydes, ketones and alcohols, while in the case of pine wood, terpenes were also emitted. Analyses conducted 24 h and 72 h after the samples had been placed in the chamber, showed that the amounts of compounds released varied over time.

Keywords: VOC, water-based lacquers, UV-cured, surface coatings, chamber tests, TD/GC/MS

Introduction

Wood is lacquered in order to increase its durability and resistance to the action of external factors and to enhance its colour and pattern. The contribution of lacquered wood products to indoor air pollution varies depending on the chemical composition of the applied lacquer product, its solvent content, coating thickness as well as the application, drying and curing conditions. Solvents contained in lacquer products are considered to be the primary source of VOC (Volatile Organic Compounds) emissions.

Agata STACHOWIAK-WENCEK, Poznań University of Life Sciences, Poznan, Poland
e-mail: astachowiak@up.poznan.pl

Włodzimierz PRĄDZYŃSKI, Wood Technology Institute, Poznan, Poland
e-mail: wpradzynski@up.poznan.pl

Magdalena MATEŃKO-NOŻEWNIK, Poznań University of Life Sciences, Poznan, Poland
e-mail: magdamatenko@wp.pl

UV-cured lacquers can be products free from solvents. They contain 100% film-forming substances and no components evaporating into the environment [Proszyk 1999; Bongiovanni et al. 2002]. An interesting solution is to combine formulations of water-based products with UV-cured products [Anonymous 2007].

The application of UV radiation to cure lacquer coatings is currently being developed. UV curing technology facilitates the almost immediate further woodworking of finished elements and the application of further layers, thus shortening the duration of the manufacturing process. Moreover, this technology provides excellent performance properties of the finished surface [Salthammer 1996; Bongiovanni et al. 2002].

UV radiation may be applied to cure only certain types of lacquer products, containing first of all unsaturated polyester and acrylic resins and their copolymers. Special photoinitiators are indispensable components of UV-cured systems.

As a result of photoinitiator fragmentation, several volatile products may be formed, which may constitute a source of indoor air pollution. Studies on VOC emissions from the surface of wood and wood-based materials finished with UV-cured products have been presented by several researchers, e.g. Salthammer [1997], Salthammer et al. [1999], Uhde and Salthammer [2007] and Kagi et al. [2009]. According to literature, UV-cured coatings are also sources of emissions of volatile substances.

The aim of this study was to determine the type and amounts of volatile compounds emitted from the surface of selected wood species finished using selected UV-cured water-based systems.

Material and methods

Sample preparation

Analyses were conducted on three wood species: pine, oak and beech wood. Samples of $280 \times 200 \times 16$ mm were prepared under commercial scale production conditions. Wood was dried in a drying kiln [Hamech SK 55, Hajnówka] equipped with software by Automatex. Samples for analyses were collected from strips of 100 mm in width, glued with polyacetate vinyl adhesive. The wood surface was sanded with sandpaper of 180 and 220 grits. The moisture content of the samples determined by the gravimetric method fell within the range of 7.5–8.5%. In turn, the density determined by stoichiometry was 569 kg/m^3 for the pine wood (an average level), 672 kg/m^3 for the beech wood and 680 kg/m^3 for the oak wood.

The samples of the selected wood species were covered on both sides with three types of UV-cured water-based products (WB/UV1, WB/UV2, WB/UV3B), whose characteristics are presented in table 1. The selected products are used in the surface finishing of most wood species used indoors. They are multicoat products, which may be used as undercoat and topcoat lacquers. They form transpa-

rent coatings of high mechanical and chemical resistance. The technical parameters of the water-based UV lacquers are shown in table 1.

Table 1. Technical parameters of water-based UV

Parameters	Lacquers		
	WB/UV1	WB/UV2	WB/UV3
Film-forming substance	unsaturated acrylic oligomers and prepolymers		
Photoinitiator	HCPK*		
Dilutant	water		
Solid content [%]	35 ± 1	38 ± 1	45 ± 1
Gloss [°G]	4–7	26–29	8–12
Specific gravity [kg/cm ³]	1050 ± 30	1050 ± 30	1050 ± 30
Commercial viscosity [s]	40–50	40–50	40–50
Application viscosity	delivered		

* 1-hydroxycyclohexyl phenyl ketone

Lacquering products were applied on the wood surfaces by means of a pneumatic spray gun (nozzle diameter – 1.4 mm). The application amount of the lacquering product applied was 110 ± 5 g/m².

The drying and curing process consisted of three parts. The first part was a flash-off-period – 10 min at a temperature of 23–25°C. The second part was the proper drying period – 15 min at a temperature of 60°C. The third part was the ultraviolet curing with a UV unit equipped with a Mercury lamp and an elliptical (focused) reflector. The power of the UV lamp was characterised as 120 W/cm. The speed of the conveyor during the drying and curing process was 10 m/min.

Chamber tests

All the experiments were carried out in a 0.225 m³ glass chamber under the following conditions: temperature: $23 \pm 2^\circ\text{C}$, relative humidity: $45 \pm 5\%$, air exchange rate: 1 h⁻¹ and loading factor: 1.0 m²/m³.

Sampling and TD/GC/MS analyses

Air samples were collected on a Tenax TA (35/60 mesh, Alltech Company) at a 1 l total volume and at a flow rate of 100 ml/min. Air samples for the analyses were collected 24 h and 72 h after the elements had been placed in the chamber. In each case, three air samples were collected. Analytes adsorbed on the Tenax TA were analysed using a GC/MS apparatus, equipped with a thermal desorber, according to the procedure presented in table 2.

Table 2. Operating conditions of TD/GC/MS

Elements of measuring system	System's working conditions
Injector	Thermal desorber connected to sorption microtrap; Purging gas: argon at 20 m ³ min ⁻¹ ; Purge time: 5 min.
Microtrap	Desorption temperature: 250°C Sorbent: 80 mg Tenax TA/30 mg Carbosieve III; Desorption temperature: 250°C for 90 s
Gas chromatograph	TRACE GC, Thermo Finnigan
Column	RTX – 624 Restek Corporation, 60m x 0.32mm ID; D ₁ – 1.8 mm: 6% cyanopropylphenyl, 94% dimethylpolyoxosilane
Detector	Mass spectrometer (SCAN: 10 – 350)
Carrier gas	Helium: 100 kPa, ~2 cm ³ min ⁻¹ .
Temperature settings	40°C for 2min, 7°C min ⁻¹ to 200°C, 10°C min ⁻¹ to 230°C, 230°C for 20 min.

Individual compounds were identified by comparing the obtained mass spectra with the spectra stored at the NIST MS Search library – program version 1.7, and were then confirmed by juxtaposing the mass spectra and retention times of the identified compounds with the spectra and retention times of appropriate standards.

Quantitative analyses of VOCs emitted from the examined wood surfaces were carried out by adding the 4-bromofluorobenzene standard (Supelco).

Results

In order to provide a more detailed characteristics of the wood used in this study and to determine the source of the emissions for the identified VOCs, their emissions were recorded from the wood prior to the finishing procedures. The results of these analyses are listed in table 3.

Among the tested wood species, the greatest amounts of volatile substances were emitted to the atmosphere by the pine wood. The total amount of all the assayed compounds at 24 h after the tested samples had been placed in the chamber was 377 µg/m³. At 72 h, it had decreased to 215 µg/m³. The beech and oak wood were characterised by lower emissions of volatile substances, which after 24 h amounted to 143 and 169 µg/m³, respectively, while after 72 h amounted to 86 and 96 µg/m³.

The relatively low emission of VOCs from the pine wood was probably related to the drying process.

Studies by Lavery and Milota [2000], Milota [2000], Manninen et al. [2002] and Thompson and Ingram [2006] concerning the timber drying process and its

effect on VOC emissions indicate that timber drying conditions have a significant effect on later VOC emissions from wood products.

Table 3. Chamber concentrations of VOC from uncoated pine, beech and oak wood after 24 and 72 h exposure

Compound	Pine		Beech		Oak	
	VOC concentration [$\mu\text{g}/\text{m}^3$]					
	24 [h]	72 [h]	24 [h]	72 [h]	24 [h]	72 [h]
Butanal	-	-	19.2	11.3	-	-
Pentanal	12.8	8.7	14.8	-	-	-
Hexanal	36.2	27.6	36.4	40.1	49.8	30.2
Furfural	31.2	22.8	44.5	27.6	52.1	28.2
Acetone	31.6	21.2	47.5	18.2	21.6	16.1
α -pinene	169.8	98.2	-	-	-	-
Camphene	12.9	7.2	-	-	-	-
3-carene	36.3	12.8	-	-	-	-
Limonene	46.2	16.2	-	-	-	-
Acetic acid	-	-	-	-	45.8	21.3
TVOC*:	377	215	143	86	169	96

*TVOC – sum of emitted VOC

The spectrum of compounds released by the tested wood species varied. The broadest spectrum of volatile compounds was emitted by the pine wood. The analysed air collected from the chamber contained both monocyclic monoterpenes (α -pinene, delta-3-carene, camphene) and bicyclic monoterpenes (limonene), as well as aldehydes and ketones. The oak and beech wood mainly released compounds from the groups of aldehydes and ketones. In the case of the oak wood, an emission of acetic acid was also detected. As found in literature reports [Salthammer et al. 1999; Uhde, Salthammer 2007], the presence of aldehyde compounds is caused by degradation reactions of fatty acids found in the wood. Typical degradation products of oleic acid are saturated aldehydes from heptanal to decanal, while linoleic acid releases mainly hexanal. In turn, the presence of acetic acid and furfural may be explained by the degradation reactions of hemicelluloses. The spectrum of identified volatile compounds released by the tested wood species may be confirmed based on the analyses of VOC emissions from different wood species conducted in previous years e.g. by Risholm-Sundman et al. [1998], Jansen et al. [2001] and Manninen et al. [2002].

The process of finishing the wood samples with selected lacquer coatings altered both the type and amounts of the released compounds. The test results of UV-cured water-based lacquer coatings applied on the surfaces of the pine, beech and oak wood are presented in table 4.

Table 4. Chamber concentrations of VOCs from UV-cured water-based systems

Lacquer	Compound	Pine		Beech		Oak	
		VOC concentration [$\mu\text{g}/\text{m}^3$]					
		24 [h]	72 [h]	24 [h]	72 [h]	24 [h]	72 [h]
WB/UV1	Acetone	48.9	22.1	49.4	15.2	28.1	8.9
	1-butanol	61.2	21.4	41.5	20.1	53.8	22.8
	1,4-dioxane	28.41	14.2	22.7	24.0	31.0	12.8
	Hexanal	33.5	36.2	16.1	12.9	25.4	30.1
	Furfural	10.1	-	6.1	-	11.2	2.2
	Cyclohexanone	89.2	36.4	78.2	34.2	81.2	39.2
	α -pinene	78.9	58.2	-	-	-	-
	camphene	6.9	-	-	-	-	-
	3-carene	12.1	4.2	-	-	-	-
	Limonene	12.5	3.9	-	-	-	-
	Benzaldehyde	152.4	81.2	142.2	79.8	132.1	75.9
	Unidentified	22.4	5.2	17.1	4.8	19.2	9.8
TVOC		557	283	373	191	382	202
WB/UV2	Acetone	29.1	19.2	27.2	15.2	26	16.8
	1-butanol	37.8	24.6	45.2	18.2	58.3	29.2
	1,4-dioxane	10.7	-	8.2	3.2	28.2	16.5
	Hexanal	13.1	16.2	14.2	16.2	19.2	20.1
	Furfural	4.7	-	8.2	0	6.2	2.2
	Cyclohexanone	50.8	31.2	42.2	21.1	61.2	25.9
	α -pinene	59.2	42.2	-	-	-	-
	camphene	13.8	5.2	-	-	-	-
	3-carene	12.5	4.3	-	-	-	-
	Limonene	29.8	15.2	-	-	-	-
	Benzaldehyde	113.2	88.4	115.2	75.2	102.5	62.3
	Unidentified	22.1	7.2	25.2	17.2	12.9	7.7
TVOC		397	254	286	166	315	181
WB/UV3	Acetone	43.5	25.2	42.0	28.7	33.5	18.1
	1-butanol	48.5	31.2	30.9	13.3	31.9	17.2
	Toluene	12.8	4.3	11.4	6.2	9.6	6.4
	Butyl acetate	11.5	7.2	11.5	5.4	12.5	6.6
	Hexanal	28.9	36.4	25.9	27.2	37.2	33.2
	Furfural	8.2	2.2	6.9	4.2	9.5	7.5
	Cyclohexanone	12.1	5.2	10.0	4.8	16.4	5.7
	α -pinene	86.1	58.0	-	-	-	-
	Camphene	12.2	9.2	-	-	-	-
	3-carene	19.2	8.2	-	-	-	-
	Limonene	26.4	16.3	-	-	-	-
	Benzaldehyde	98.5	77.2	112.2	79.8	103.8	81.2
TVOC		408	281	251	170	254	176

*TVOC – sum of emitted VOC

These results indicate that coatings produced from UV-cured water-based lacquer systems are also sources of emissions of volatile substances. The concentrations of volatile compounds in the first stage of the study, i.e. 24 h after the placement of samples in the chamber, ranged from 251 to 557 $\mu\text{g}/\text{m}^3$.

When comparing the test results with those obtained for the coatings containing water-based products not cured by UV radiation, it may be stated that the results were relatively low. The concentrations of volatile substances emitted from the water-based lacquer coatings not cured by UV radiation fell within a markedly greater range of 388 to 1794 $\mu\text{g}/\text{m}^3$ [Stachowiak-Wencek, Prączyński 2011].

Among the tested UV-cured systems, the greatest amounts of volatile substances were released by the coatings based on WB/UV1 lacquer. The emission of volatile substances from the coatings on the pine wood after 24 h was 557 $\mu\text{g}/\text{m}^3$. The amount of substances released from the coatings on the beech and oak wood was lower, amounting to 373 and 382 $\mu\text{g}/\text{m}^3$, respectively. Lower amounts of volatile substances were released by the coatings produced from the WB/UV2 and WB/UV3 lacquers. After 24 h, the coatings on the pine wood released volatile substances at 397 and 408 $\mu\text{g}/\text{m}^3$, those on the beech wood emitted 286 and 251 $\mu\text{g}/\text{m}^3$, while on the oak wood released 315 and 254 $\mu\text{g}/\text{m}^3$, respectively.

The analyses showed that the air samples collected from the testing chamber contained compounds mainly from the groups of aldehydes, ketones and alcohols, as well as terpenes, in the case of the pine wood.

Benzaldehyde was the predominant component released by all the tested coating types. Moreover, the coatings from the WB/UV1 and WB/UV2 lacquers released considerable amounts of cyclohexanone. According to literature reports [Salthammer et al. 1999; Salthammer 2004; Uhde, Salthammer 2007, Kagi et al. 2009], cyclohexanone and benzaldehyde are degradation products of the photoinitiator such as HCPK (1-hydroxy-cyclohexyl-phenone).

The greatest concentrations of benzaldehyde were recorded in the case of the coating based on WB/UV1. After 24 h, the concentration of this compound in the chamber, depending on the wood species, ranged from 132.1 to 152.4 $\mu\text{g}/\text{m}^3$. The coatings of the WB/UV2 and WB/UV3 lacquers released lower amounts ranging from 102.5 to 113.2 $\mu\text{g}/\text{m}^3$ and 98.5 to 112.2 $\mu\text{g}/\text{m}^3$. The concentration of cyclohexanone in the air from the chamber was lower and more varied. It ranged from 78.2 to 89.2 $\mu\text{g}/\text{m}^3$ for WB/UV1, from 42.2 to 61.2 $\mu\text{g}/\text{m}^3$ for the WB/UV2 and from 10.0 to 16.4 $\mu\text{g}/\text{m}^3$ for WB/UV3.

Moreover, acetone was also detected in the tested air. According to literature data [Salthammer 2004], acetone may also be a degradation product of UV systems. However, the analyses of the emissions of the volatile substances from the wood not covered with lacquer coatings (table 3) showed that the wood itself may be a source of emission of this compound. The concentration of acetone in the unfinished wood ranged from 21.6 to 47.5 $\mu\text{g}/\text{m}^3$. When analysing the recorded results, it may be found that the coatings from WB/UV1 and WB/UV3 constituted

an additional source of acetone emissions, while the coatings from the WB/UV2 lacquer reduced its emission into the air.

In the air samples collected from the chamber, the presence of 1-butanol was also detected. Salthammer et al. [2002] stated that certain acrylates may hydrolyse of producing volatile alcohols. After 24h, the concentration of butyl alcohol ranged from 30.9 to 61.2 $\mu\text{g}/\text{m}^3$.

Moreover, the emission of 1,4-dioxane was detected in the case of the coatings from the WB/UV1 and WB/UV2 lacquers, while slight amounts of toluene and butyl acetate were released from the WB/UV3 coatings.

Additionally, terpenes were released from the pine wood. The application of finishing coatings resulted in a reduction in their emissions. When comparing the results of the tests conducted on the pine wood uncoated with lacquer coatings with the results recorded for the finished wood, it may be stated that the coatings caused a reduction in terpene emissions by 41–58%, depending on the type of lacquer product. The greatest reduction in the amount of released terpene compounds was caused by the WB/UV1 coatings, while the decrease was slightest in the case of the WB/UV3 coatings. After 72 h, the terpene concentrations were even lower. Depending on the type of applied lacquer product, it decreased by a further 17–22%.

When comparing the test results recorded after 24 h and 72 h, it may be stated that except for hexanal, the concentrations of the other determined compounds in that period decreased.

After 72 h, the total concentration of all the determined compounds ranged from 166 to 283 $\mu\text{g}/\text{m}^3$, i.e. between the first and second stage of the analyses, the amounts of released compounds decreased in the case of the WB/UV1 coatings by 47–49%, the WB/UV2 coatings by 36–43%, while for the WB/UV3 product, with the highest solids content, its reduction was the smallest, amounting to 31–32%.

Based on these results, it may be stated that the level of these changes was dependent to a greater extent on the type of lacquer system applied rather than on the type of finished surface. In the case of the WB/UV2 and WB/UV3 lacquers, the lowest reduction in the concentrations of the assayed compounds was recorded for the coatings on the pine wood. In turn, in the case of the WB/UV1 coating, the lowest decrease in the amounts of the released substances was recorded for the coatings on the beech wood, while it was greatest for the coatings on the pine wood.

Conclusions

Studies on the emission of volatile substances from UV-cured systems were also presented by Salthammer [1997], Salthammer et al. [1999], Uhde and Salthammer [2007] and Kagi et al. [2009].

Salthammer et al. [1999] identified 12 compounds, including solvents, monomers and photoinitiators, e.g. also cyclohexanone and benzaldehyde. They also detected the presence of propylene glycol-monomethyl ether (isomers), acetophenone, 2-phenyl-2-propanol, 1-methoxy-ethanol-benzoate, Texanol-1, Texanol-2, T4MDD, acrylic monomer, benzophenone and hydroxy-cyclohexanoid acid.

In addition, emissions of cyclohexanone and benzaldehyde were shown in studies analysing a wooden floor finished with a UV-cured system conducted by Kagi et al. [2009]. They also found that in the case of reference samples, there was a release of formaldehyde, acetaldehyde, crotoaldehyde, butyl aldehyde, acetic acid, toluene, methylenecyclohexane and high emissions of benzene.

Tests on cork parquet coated with UV-cured coatings, published by Uhde and Salthammer [2007], also showed emissions of cyclohexanone and benzaldehyde from UV-cured systems. Moreover, Uhde and Salthammer identified CS₂, acetic acid, furfural, styrene, phenol, methyl benzoate, 4-phenyl cyclohexane and benzophenone inside the testing chamber.

When comparing results presented in this study with those reported by other authors, it may be stated that the tested systems differed in the type of volatile substances released. However, all the elements tested released cyclohexanone and benzaldehyde into the atmosphere.

1. Based on the results of the analyses, it was found that coatings produced from UV-cured water-based lacquers may be sources of emissions of volatile substances. Total concentrations of all the identified compounds were lower than those recorded for conventional water-based products not cured by UV radiation [Stachowiak-Wencek, Prądyński 2011].
2. Compounds emitted by the lacquer coatings and those originating from the wood were detected in the tested air. The determined compounds mainly included aldehydes, ketones and alcohols, while in the case of the pine wood there were also terpenes. Benzaldehyde was the compound released in the greatest amounts, probably being the degradation product of the photoinitiator contained in the lacquer systems.
3. The studies on the emission of volatile organic compounds from the lacquer coatings applied on the wood species differing in their anatomical structure and physico-chemical properties showed the effect of the wood species on the level of released volatile substances. The greatest amounts of volatile substances were released by the coatings on the pine wood.
4. The chamber test showed that the concentrations of volatile substances released by the tested samples decreased in time. The level of recorded changes was also dependent on the type of lacquer product used and on the wood species.

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EMISJA LOTNYCH ZWIĄZKÓW ORGANICZNYCH (VOC) Z WODOROZCIEŃCZALNYCH WYROBÓW LAKIEROWYCH UV

Streszczenie

W pracy przedstawiono wyniki testów komorowych wodorozcieńczalnych powłok lakierowych utwardzanych promieniowaniem UV, naniesionych na różne gatunki drewna. Lotne związki organiczne pobierano na Tenax TA, a następnie oznaczono metodą chromatografii gazowej sprzężonej z spektrometrią masową i termiczną desorpcją. Stwierdzono, że powłoki oparte na wodzie, utwardzane UV mogą stanowić potencjalne źródło emisji substancji lotnych i zanieczyszczać powietrze wewnętrzne. Poziome stężenia tych związków w komorze był uzależniony od rodzaju użytego systemu lakierowego oraz od rodzaju wykończonego podłoża. Najwyższą emisję substancji lotnych stwierdzono z powłok naniesionych na powierzchnię drewna sosny. Badane powłoki wydzielaly związki należące do aldehydów, ketonów i alkoholi a w przypadku drewna sosny również do terpenów. Badania wykonane po 24 h i 72 h po umieszczeniu prób w komorze wykazały, że ilość uwalnianych związków jest zmienna w czasie.

Słowa kluczowe: VOC, lakiery wodorozcieńczalne, UV- utwardzane, powłoki powierzchniowe, badania komorowe, TD/GC/MS

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