Formaldehyde Emission from Wood-Based Panels Bonded with Different Formaldehyde-Based Resins

Emisija formaldehida iz drvnih ploča s različitim ljepilima na bazi formaldehida

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ABSTRACT • In this study, the formaldehyde emission (FE) from different types of particleboard, medium density fiberboard (MDF), and plywood products supplied from a commercial plant in the Czech Republic were evaluated by gas analysis (EN 717-2) and European small chamber (EN 717-1) methods. The significant effects of manufacturing variables (board type and thickness) as well as different types of formaldehyde-based resins on FE measured by gas analysis were obtained. When the E1 type adhesives were employed, a wide variation in the quantity of free formaldehyde was observed among the three product types. The FE values of plywood samples measured by gas analysis were lower than those of the particleboard and MDF samples. The correlation between the two methods for the particleboard and MDF were good (R² = 0.82 and 0.76, respectively) and however for plywood (R² = 0.52) it was not convincing. FE specified in EN 717-2 was comparable with the EN 717-1 values for the same board type and thickness as well as the resin type and below the E1-emission class.

Keywords: formaldehyde emission, formaldehyde-based resins, wood-based panels, chamber method, gas analysis method.

SAŽETAK • U radu se analizira emisija formaldehida (FE) iz različitih tipova ploča iverica, ploča vlaknatica srednje gustoće (MDF) i furnirskih ploča nabavljenih od komercijalnih proizvođača u Republici Češkoj. Emisija formaldehida određena je primjenom dviju metoda – analize plinova (EN 717-2) i male europske komore (EN 717-1). Istraživanja su pokazala signifi kantan utjecaj proizvodnih parametara ploča (vrste i debljine ploča) i vrste upotrijebljenog formaldehidnog ljepila na emisiju formaldehida mjerenu metodom analize plinova. Analizom uzoraka za koje je upotrijebljen E1 tip ljepila dobiven je širok raspon vrijednosti količine formaldehida za tri vrste proizvoda. Vrijednosti FE furnirskih ploča niže su od vrijednosti uzoraka ploča iverica i MDF ploča. Korelacija rezultata dobivenih dvaju različitim metodama dobra je za ploče iverica i MDF ploče (R² = 0.82 i

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1 INTRODUCTION

1. UVOD

In 1992, the California Air Resources Board (CARB, 1992) identified formaldehyde as a toxic air contaminant based primarily on the determination that it was a human carcinogen with no known safe level of exposure. The International Agency for Research on Cancer (IARC, 2004) conducted an evaluation of formaldehyde and concluded that there is sufficient evidence that formaldehyde causes nasopharyngeal cancer in humans (i.e., in the region of the throat behind the nose). Formaldehyde is a well-known allergen that causes contact dermatitis. Formaldehyde can be free on the material or bonded in different ways to the chemical structure. Free formaldehyde, including bonded formaldehyde, can be released under different analytical conditions.

Wood-based panels such as plywood, medium density fiberboard (MDF) and particleboard and the resins used like urea-formaldehyde (UF), melamine-modified urea formaldehyde (MUF), and phenol-formaldehyde (PF) are the main sources for FE. The present importance of such problematic results arises from the fact that formaldehyde was the first among the six chemical substances considered as industrial hazards (Tanabe, 2008). In 2001, the CARB initiated the development of a regulation to reduce public exposure to formaldehyde. The CARB regulation, effective January 1, 2009, placed limits on formaldehyde emission (FE) from wood-based panels.

Aminoplastic resins, especially UF-resins, are the main binders used in the industry of wood-based panels. UF-resins are fast curing resins and of uncontestable good performance. However, boards bonded with UF-resins are, in general, of limited moisture resistance and emit detectable amounts of formaldehyde (Roffael et al., 2010). Furthermore, concern about the emission of formaldehyde from particleboards and weakening glue bond caused by hydrolytic degradation of UF polymers have stimulated efforts to develop improved and/or new adhesives based on UF resins.

The MUF resins with reduced melamine content levels have been developed to improve durability and moisture resistance properties. These low-melamine content UF resins have been relatively popular in Europe and in the Asia-Pacific region (Parker and Crews, 1999) for many years.

Phenolic-based compounds also tend to be more chemically stable and less susceptible to hydrolysis than UF. Both of these characteristics are beneficial to PF resin. This is one of the reasons why PF resins are considered waterproof, while UF is not (Dunky, 2005). PF resin has generally been the resin chosen by the manufacture of exterior grade structural panels.

There are many factors affecting the FE of wood panel products. For example, many variables such as temperature, relative humidity, air exchange rate, loading ratio, etc. could affect the FE measurements of wood-based panel products (Myers and Nagaoka, 1981; Myers, 1985).

Actually, many different attempts have been made to compare the FE or to establish correlations between methods. Sundin et al. (1987) compared four different methods of testing the FE of particleboards, and found good relationships between the methods with correlation coefficients greater than 0.9. Risholm-Sundman et al. (2007) reported that the variations between the measured results were due to specific differences in test conditions. Bulian et al. (2003) also reported that the lack of certified reference material made it difficult to establish an inter-calibration between test methods.

The amount of free formaldehyde observed in the chamber under conditions that simulated mobile loadings of wood product, air change rate, temperature, and humidity relate to real wood formaldehyde levels (Que and Furuno, 2007). Recently, continuous methods have also been proposed for assessing the formaldehyde release during production in the factories (Engström, 2007, 2008). In Europe, mainly three laboratory methods for the determination of formaldehyde release have been standardized and namely: 1) Extraction method called the perforator method (EN 120, 1993), 2) FE by gas analysis method (EN 717-2, 1994) and 3) FE by the flask method (EN 717-3, 1996). Apart from these methods, the FE of the boards can be measured using the European chamber technique (EN 717-1, 2004), which is considered to be the reference method.

Among the above-mentioned laboratory methods, gas analysis technique gained wide acceptance for assessing the emission of formaldehyde from wood-based panels. The European gas analysis method is CARB-approved quality control test method (Ruffing et al., 2010).

This study aimed to determine the effects of some manufacturing factors on the emission of formaldehyde from different types of particleboard, MDF and plywood panels. The effects of board type and thickness were investigated, as well as the effect of resin adhesive type. The relationship between the European small-chamber and gas analysis values were also reported.


2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Sample collection
2.1. Skupljanje uzoraka

Wood-based panels used in this study were particleboard, MDF and plywood, with thickness of 12 mm (T12), 16 mm (T16) and 18 mm (T18), supplied from commercial plants in the Czech Republic. Samples of particleboards (500 mm × 500 mm) were cut at the mill from three full-sized boards (2840 × 1830, 2750 × 1830 and 2810 × 1810 mm) from each thickness type of uncoated (P2), laminated (PL) and veneered (PV) particleboards, respectively. In addition, the uncoated MDF (MDF) samples were cut from 2750 × 1840 mm boards for each thickness. The laminated MDF (MDFL) samples were taken from the boards with dimension 2750 × 1840 mm for T16 and T18 and from 2440 × 1220 mm for T12, respectively; and these boards were laminated with high-pressure laminate.

The uncoated plywood samples used in interior application (PLY) were cut from each of the three panels with dimensions 250 × 125 cm of T12, T16, and T18. These panels were produced from beech veneers. Samples of plywood with T12 and T18 used in construction applications (PLYs) were cut from panels with dimensions 125 × 250 cm and produced from birch veneer and with T16 panels they were produced from poplar veneer. The sampling was done in accordance with standards EN 312 (2003), EN 622–1 (2003), and EN 13986 (2002). These standards were designed for testing the requirements of wood-based panels.

All the samples were delivered to the laboratory of Timber Research and Development Institute in Prague, Czech Republic. The delivered samples were wrapped with polyethylene film prior to being cut into test specimens in order to measure the FE with EN 717-1 and EN 717-2.

The plywood samples are conditioned for 4 weeks at 20 °C and 65 % RH before measuring the FE by EN 717-2 according to German Federal Health Office (BGA, 2007). Particleboards and MDF samples were analyzed directly after opening the polyethylene film and sealing the edges (Risholm-Sundman et al., 2007).

The different types of particleboard and MDF were produced for many different purposes, especially for the manufacture of furniture and interior equipment. MDF is produced from spruce wood fiber, bonded together with MUF resin. The three types of particleboard panels used in this study were bonded with a high quality wholesome UF resin and produced from spruce particles. The veneered particleboards are made from the European oak decorative veneer, which is pressed onto the board on both sides. PLY panels were bonded with MUF resin adhesive and PLYs panels were bonded with PF resin. The numbers of different types of particleboard, MDF and plywood samples for gas analysis and European small-chamber tests were determined according to board thickness (Table 1).

2.2 Free formaldehyde test methods
2.2. Metode određivanja emisije formaldehida

To determine the FE, the European small-chamber and gas analysis methods were employed as specified in the standards EN 717-1 (2004) and EN 717-2 (1994), respectively.

2.2.1 Gas analysis method (EN 717-2)
2.2.1. Metoda analize plinova (EN 717-2)

In the gas analysis method, a test piece of 400 mm × 50 mm × board thickness was placed in a 4-litre cylindrical chamber with controlled temperature (60 ± 0.5 °C), relative humidity (RH ≤ 3%), air flow (60 ± 3 l/h) and pressure. Air was continuously passed through the chamber at 1L/min over the test piece, whose edge was sealed with self-adhesive aluminum tape before testing. The determinations were made in duplicate using two different pieces and formaldehyde value was the average of two pieces after 4 hours expressed in mg HCHO/m²·h. These determinations were repeated, as samples were available, for better homogeneity of the results. The E1-emission class for all types of wood-based panels is ≤ 3.5 mg/m²·h.

2.2.2 Small-chamber method (EN 717-1)
2.2.2. Metoda male komore (EN 717-1)

In the European small-chamber method, two test pieces (0.2 m × 0.28 m × board thickness) were cut from 500 mm × 500 mm samples for each wood product with a total area of 0.225 m² for free formaldehyde measurement by chamber method (0.225 m² in volume). The samples were not conditioned before the test. The loading factor was 1 m²/m³, so that the weights were partly sealed (1.5 m open edge/m²), where the edges of two pieces were sealed with aluminum foil to obtain a constant ratio of the length (U) of the open (unsealed) edges to the surface area (A), so that $U/A = 1.5$ m/m². The temperature was held at 23 ± 0.5 °C and the RH at 45 ± 3 %.

Formaldehyde released from the test pieces mixes with the air in the chamber, and a specified volume of air is drawn from the chamber twice a day. Sampling is periodically continued until the formaldehyde concentration in the chamber has reached a steady-state. The result of the test is given after 2-4 weeks as the

### Table 1 Number of specimens used for gas analysis and small chamber tests

| Wood-based panels | Particleboards | Fiberboards | Plywoods |
|-------------------|----------------|-------------|---------|
| Drvne ploče       | Ploče iverice  | Ploče vlaknatice | PLYs    |
| board type / tip ploče | P2  | PL  | PV | MDF | MDFL | PLY | PLYs    |
| Thickness, mm / debelina, mm | 12-18 | 12-18 | 12-18 | 12-18 | 12-18 | 12-18 | 12-18 |
| Number of samples | EN 717-2 | 36  | 22  | 24  | 28  | 26  | 20  | 22  |
| broj uzoraka       | EN 717-1 | 6   | 6   | 6   | 6   | 6   | 6   |

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steady-state emission value (mg/m³) or ppm. The E1-emission class for all types of wood-based panels is ≤ 0.1 ppm (0.12 mg/m³ of air).

The formaldehyde amount in the water from both methods was determined photometrically by acetyacetone spectrophotometric analysis. This technique, as described by Nash (1953), is widely applied and is a standard procedure for the specific analysis of free formaldehyde. The determination is based on the Hantzsch reaction, in which aqueous formaldehyde reacts with ammonium ions and acetylacetone to yield diacetylhydrolutidine (DDL).

2.3 Statistical Analyses

In order to achieve the study aims; the formaldehyde values measured by gas analysis method were statistically analyzed. Analysis of variance (ANOVA) with different repetitions was used to test for significant difference of factors and levels. When the ANOVA indicated a significant difference among factors and levels, a comparison of the means was done employing a Duncan’s multiple-range test (1954) at 0.05 level of probability. Formaldehyde values are reported as least square means (LS Means) with 95 % confidence intervals (95 % CI). Linear correlations were applied to the gas analysis versus European small chamber values using the CORR option. Analyses were performed by SAS version 8.2 (2001) Statistical Analysis System software.

3 RESULTS AND DISCUSSION

3.1 Gas analysis values

The values of FE measured by gas analysis were very significantly affected by board type (P < 0.001), board thickness (P < 0.001) and the interactions between them (P < 0.001) for almost all wood-based panels types used in this study (Table 2). For MDF boards, the interaction between them had a significant effect (P < 0.05).

The overall comparisons between the means of FE from different types of wood-based panels bonded with different formaldehyde-based resin are presented in Table 3. The FE of particleboard was the highest in PV T18 and PV T16 (2.52 and 1.68 mg/m²·h, respectively) followed by PV T12 (0.96 mg/m²·h), whereas the PL T12 had the lowest amount of FE (0.23 mg/m²·h). The FE from fiberboards showed a high amount presented in MDF T18 (0.77 mg/m²·h) followed by MDFL T18 (0.61 mg/m²·h). The PLY T18 had a high amount of FE (0.35 mg/m²·h) and the PLYs T12 had the lowest amount of FE (0.11 mg/m²·h) from plywood panels studied. The concentration of FE in PLY (it ranged from 0.16 to 0.35 mg/m²·h) had a higher value than the concentration of FE from PLYs (it ranged from 0.11 to 0.25 mg/m²·h) and this could be explained in the use of different glue types.

All values measured for the different types of wood-based panels used in this study were below the standard limit E1 specified in EN 717-2. Moreover, it has been shown that the applications of laminating over the boards were responsible for decreasing the emission of formaldehyde from the particleboard and MDF, and also for increasing FE due to the increase of the board thickness.

3.2 Small-chamber values

For the sake of comparison, the European small-chamber values that were obtained for almost all the boards examined - particleboard, MDF and plywood, are shown in Table 4. For particleboard samples, the formaldehyde values ranged from 0.048 (PL T12) to 0.123 mg/m³ (PV T18) and from 0.042 (MDF T12) to 0.087 mg/m³ (MDF T18) for the fiberboards. Plywood formaldehyde values also ranged from 0.051-0.066 mg/m³ for PLY and 0.005 to 0.007 for PLYs. In this regard, the emission of formaldehyde from the boards is close to the emission of solid untreated wood (i.e., between 0.008-0.01ppm for spruce wood flakes) (Marutzky and Dix, 2004).

The last study also showed that the veneered particleboards emitted a higher amount of free formaldehyde.

Table 2 Significant levels for the effects of board type within the thickness on overall values of gas analysis of wood-based panels

Table 2. Razina signifikantnosti utjecaja vrste ploče jednake debljine na vrijednosti dobivene analizom plinova za sve vrste istraživanih ploče.

| Wood-based panels | Source of Variation / Izvor varijacije | Significant level / Razina signifikantnosti | R² | CV |
|-------------------|----------------------------------------|--------------------------------------------|----|----|
| Particleboards    |                                        | A                                          | ***|    |
| ploče iverice     |                                        | B                                          | ***|    |
|                   |                                        | A × B                                      | ***|    |
| Fiberboards       |                                        | A                                          | ***|    |
| ploče vlaknatice  |                                        | B                                          | ***|    |
| Plywoods          |                                        | A                                          | ***|    |
| furnirske ploče   |                                        | B                                          | ***|    |
|                   |                                        | A × B                                      | ** |    |

(*) P < 0.05 (significant/signifikant), (**) P < 0.01 (highly significant / visoko signifikant), (***P < 0.001 (very highly significant / vrlo visoko signifikant). (A) Board type / vrsta ploče, (B) Board thickness / debljina ploče, (A × B) Interaction between board type and thickness / interakcija između vrste ploče i debljine ploče.

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The methods show similar results. The FE values of plywood samples measured by EN 717-1 and EN 717-2 were below the E1-class emission. The FE values of plywood samples measured by EN 717-1 and EN 717-2 were below the E1-class emission.

The FE values of plywood samples measured by the gas analysis method were lower than those of the particleboard and MDF samples. Our results were in agreement with Park et al., 2010, who found that the emission of formaldehyde from plywood samples measured by the 1-m³ chamber method was lower than those of particleboard and MDF samples. Furthermore, the boards-E1, had approximately the same value for the gas analysis method were lower than those of the plywood panels used in construction had the lowest FE concentration. All the values of the tested wood-based panels measured by EN 717-1 and EN 717-2 were below the E1-class emission.

The FE values of plywood samples measured by the gas analysis method were lower than those of the particleboard and MDF samples. Our results were in agreement with Park et al., 2010, who found that the emission of formaldehyde from plywood samples measured by the 1-m³ chamber method was lower than that of particleboard and MDF samples. Furthermore, the boards-E1, had approximately the same value for each method. This shows that for the same kind of material, the methods show similar results.

### 3.3 Effect of different types of formaldehyde-based resins

By introducing the above experimental data of the FE measured by gas analysis and corresponding effects of some manufacturing variables following a similar procedure as described above, it was found that the types of resins had a high effect on the FE.

As can be seen in Figure 1, a high amount of FE measured by gas analysis method was observed from PL/UF, while the MDFL/MUF had a lower amount of FE than particleboards, and the plywood values from PLYs/PF had the lowest amount of FE. In addition, these results were comparable with the chamber values for the same board type and thickness as well as the resin type. It is important to point out here that such low free formaldehyde values may be emitted from the wood itself. At such low levels of free FE, the boards are considered formaldehyde free.

The differences between the formaldehyde values emitted from different types of wood-based panels due to different formaldehyde-based resins can be explained as follows: the reaction of urea with formaldehyde first produces hydroxymethylated urea that then condenses to yield methylene and dimethylene ether bridged urea bonds with different letters are significantly different (P < 0.05).

Means with different letters are significantly different (P < 0.05).

Means with different letters are significantly different (P < 0.001).

Means with different letters are very highly significant effect (P < 0.001) / vrolo visoko statistično razlikite (P < 0.001)

Vertical bars denote 0.95 confidence intervals / Okomite dužine označavaju 0.95 intervala pouzdanosti.

Figure 1 Comparison of formaldehyde emission measured by EN 717-2 of different types of wood-based panels bonded with different formaldehyde-based resins

Slika 1. Usporedba emisije formaldehidih iz različitih tipova drvinskih ploča s različitim formaldehidnim ljepilima mjerene metodom prema normi EN 717-2.
polymers (Pizzi 2003; Meyer 1979). Although these reactions are not likely to produce the other formaldehyde-containing wood adhesives, the UF polymers were distinct as they are susceptible to hydrolysis under some normal conditions used (Myers, 1986).

In accordance with Dunky (2005), the stability against hydrolysis that increased in MUF may be due to stabilization of the C-N-bonding that resulted from the quasi-aromatic ring structure of the melamine and slower decrease of the pH in the bond line and due to the buffer capacity of melamine. In addition, the C-C bonding in the PF resins was very stable against hydrolytic attack.

3.4 Relationship between gas analysis and small-chamber values

Linear correlation analyses made between the gas analysis ($Y$) and the corresponding average small-chamber values ($X$) were affected by board type, board thickness, and formaldehyde-based resins for particleboard (Figure 2), MDF (Figure 3) and plywood panels (Figure 4). The regression equations ($Y = 24.74 \cdot X - 1.213$, $R^2 = 0.82$, $P < 0.001$ for particleboard panels measured at P2, PV and PL), ($Y = 9.477 \cdot X - 0.115$, $R^2 = 0.76$, $P < 0.05$ for the influences of MDF and MDFL), and ($Y = 2.635 \cdot X - 0.144$, $R^2 = 0.52$, $P < 0.05$ for the effect of PLY and PLYs) suggested that the emissions of formaldehyde resulted from the free formaldehyde or from hydrolysis of the cured resin that might be attributed to board type and thickness levels and different types of resin. The correlation between the gas analysis and the European small-chamber methods was not convincing for plywood panels, the obtained $R^2$ value was 0.52 (see Figure 4) and it was probably related to the difference in the resins used.

4 CONCLUSIONS

Samples of particleboard, MDF and plywood products were obtained from commercial plants that produce more than 70% of the capacity in the Czech Republic. These samples were used in well-controlled chamber tests (small chamber and gas analysis) to estimate the formaldehyde emissions. The European testing of the emitted formaldehyde and evaluation system is based in principle on EN 717-1 but in practice, it is carried out by derived test methods like EN 717-2.

A wide variation in the quantity of free formaldehyde was observed among the three product types. It was clear that the variations between the different values of formaldehyde emissions observed in both methods were resulted from board type and thickness as well as the resin type. In addition, the other factors like edge sealing and test temperature, which have a large effect on the final emission result, should be taken in consideration.

Similar values of free formaldehyde observed in this work are reported for EN 717-1 and EN 717-2. Moreover, all values measured were below the standard limit E1 for EN 717-1 and EN 717-2.

The correlation between the gas analysis and the chamber for the particleboard and MDF were good ($R^2 = 0.82$ and 0.76, respectively) and for plywood ($R^2 = 0.52$), however, it was not convincing.

Finally, emissions of formaldehyde are expected to decrease with the decrease of the coated wood-based panels and board thickness. Results reported in this study apply to freshly manufactured materials.

**Figure 2** Correlation between EN 717-1 and EN 717-2 values for particleboards, thickness 12-18 mm

**Figure 3** Correlation between EN 717-1 and EN 717-2 values for fiberboards, thickness 12-18 mm

**Figure 4** Correlation between EN 717-1 and EN 717-2 values for plywood, thickness 12-18 mm
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