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Research Article

Evaluation of Dynamic Microchamber as a Quick Factory Formaldehyde Emission Control Method for Industrial Particleboards

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The most common formaldehyde control method for wood panels in Europe, the perforator method, measures formaldehyde content, while most of the legal requirements in the world are based on emissions. Chamber methods typically used for emission measurements require too much time to reach steady state for factory quality control. The aim of this study was therefore to investigate whether emission values of particleboards measured one day after production would be usable for quality control purposes. The correlation between 1-day and 7-day emission values was determined using a dynamic microchamber (DMC). Three industrial board types that differed in density and emission levels were used for the evaluation. The online emission measuring equipment Aero-laser AL4021 connected to the 1 m^3 chamber was used to gain further information on the emission reduction behaviour of the different board types. Only the two particleboard types with higher densities showed good correlation between the 1-day and 7-day emissions. The overall results suggested that 1-day emission values can be used for factory quality control purposes; however, if the initial 1-day values are above the permitted level, extensive evaluation for each individual board type needs to be performed.

1. Introduction

The main substance of concern to indoor air quality from wood-based panels such as particleboards and fiberboards is formaldehyde. It has been classified as a nasopharyngeal cancer-causing carcinogen [1]. Formaldehyde is a naturally occurring chemical that can be found in lignocellulosic materials such as wood. Up until now, the main source of formaldehyde in wood-based panels has been urea-formaldehyde glue, and the main driver for developing sustainable and formaldehyde-free glue systems has been lowering emissions [2].

However, the use of formaldehyde-free glues will not lead to formaldehyde-free panels [3–7]. As the share of low-emitting glues increases in panel industry [8–10], the effect of wood particles and fibers on emissions will increase. Even small amounts of emission in indoor air will build up over time in modern energy-saving buildings with lower air exchange rates. As an example, in China, the building energy efficiency standard and heating policies lead to lower air exchange rates and higher indoor air temperatures, both of which increase indoor formaldehyde concentrations [11]. Natural wood can emit significant amounts of formaldehyde due to the thermohydrolytic cleavage of wood constituents [12–16]. In the panel production processes, wood is subjected to three main factors that increase the formaldehyde emissions: particle size reduction, thermal treatment, and drying [17]. The effect of particle size on the formaldehyde release of wood has been evaluated by Roffael et al. [16]. It was found that the formaldehyde release measured by the flask method (EN 717-3) significantly increased as the particle size was reduced, possibly due to the increase in surface area.

It is known that the formaldehyde emissions from thermally treated fibers are higher than those from the wood chips before thermal treatment. However, treatment temperature also has an effect on the final emissions of the fibers. Formaldehyde emissions increased significantly when temperature exceeded 40°C, and fibers treated at 160°C emitted higher formaldehyde quantities than fibers obtained from 140°C process [18].

Natural wood is not the only source of formaldehyde emissions. The current trend in the industry is to use more recycled wood in particleboard production. Recycled wood has higher emission levels, especially if the share of particleboards in the recycled mix is high [19]. It should be noted that some of the particleboards found in the recycled mix might have been produced before the emission limit in Europe was lowered to 0.1 ppm. Thus, when recycled wood is used in production, control of the incoming wood mix is extremely important for keeping the emissions low. Himmel et al. [19] managed to predict the formaldehyde emissions of pressed particleboards from the formaldehyde content of the raw materials or from the adhesive-free particle mat for different recycled material sources.

The technology to develop low-formaldehyde-emitting wood-based panels is becoming more common in the industry, but measurement methods suitable for fast panel quality control are lacking behind. Although chamber methods such as EN 717-1 and ASTM E1333 give good results, they require waiting until a steady state has been reached. In order to get results faster, modified techniques such as increased temperature (e.g., gas-analysis EN 717-2) or using extraction with solvents (e.g., perforator EN 120) have been used. This change in conditions affects their correlation to the chamber methods [20–23].

The perforator method (e.g., EN 120 and ISO 12460-5) is the most common formaldehyde measurement method used by European, and some Asian, panel producers. The perforator method has a short total running time (3 h), and the equipment is inexpensive. However, it has been discussed that the sensitivity of the perforator method decreases as the emissions decrease below 0.12 mg/m^{2}h [24]. It is a serious drawback as the modern particleboard and fiberboard glues have a molar ratio U:F above 1 : 1 and emissions below 0.09 ppm due to stricter formaldehyde limits [25]. The perforator method also measures formaldehyde content, while the legislations in the world are focusing on formaldehyde emissions (e.g., US Environmental Protection Agency, 2017). Although the accuracy of perforator at low formaldehyde levels is widely questioned, it is still used as there is no simple and fast alternative for factory formaldehyde quality control in Europe. Lately, there has been some interest to improve other methods, for example, modifications to the gas analysis method (EN 717-2) for faster and more accurate formaldehyde emission measurements.

In North America, chamber methods such as the dynamic microchamber (DMC) connected to electrochemical sensor or the small chamber ASTM D6007 are commonly used at wood panel factories. The DMC method is approved by the EPA as a quality control test method demonstrating correlation to ASTM E1333 [26]. The DMC method uses a gas analyser that contains a voltrammetric sensor to quantify the formaldehyde emissions from the test sample. When formaldehyde is present, the electrochemical sensing cell generates an electrical current. The magnitude of the current is proportional to the formaldehyde concentration [27].

In this work, the widely accepted (e.g., in both European and Japanese standards) acetylacetone (acac) method was used for DMC calibration. The Acac method can also be used internally in formaldehyde analysers such as in Aero-laser AL4021, that is, a continuous and automated formaldehyde analyser that can be connected to different chambers.

The acetylacetone (acac) method is based on the Hantzsch synthesis, where 2,4-pentanedione (acac), ammonium acetate, and formaldehyde are cyclized to form dihydropyridine 3,5-diacetyl-1,4-dihydrolutidine (DDL). The quantification can be done using UV/VIS spectroscopy at 412 nm, or with highly sensitive fluorescence spectroscopy at 510 nm. The acac method has shown good correlation with the 2,4-dinitrophenylhydrazine (DNPH) method used by ASTM and ISO standards [26, 28]. In the DNPH method, formaldehyde is collected from the chamber air by passing it through a cartridge. Formaldehyde is immobilized, thereby reacting with DNPH to form hydrazones. These are then eluted to acetonitrile and analysed by high-performance liquid chromatography (HPLC) with UV detection.

In North America, DMC is common formaldehyde quality control test equipment for wood panel factories. However, the measuring procedures vary between factories, the biggest difference being the time between production and measurement of the panels. In the present study, the correlation between 1-day and 7-day chamber emission values of three different industrially produced particleboard types was evaluated. The purpose was to investigate how well the 1-day chamber emission values would work as a quick factory formaldehyde control method. The 1-day emission values were chosen for this study to balance the time required to reach stable board emissions and the need to receive results as soon as possible after production. The emission reduction behaviour from day 1 to day 7 after the production, and the effect of particleboard-type, was analysed. Differences among the particleboard types as regards the emission reduction behaviour were further investigated using a 1 m^3 chamber with the online monitoring system Aero-laser AL4021 connected to it.

2. Materials and Methods

2.1. Particleboard Samples. Three types of industrially produced three-layered particleboards with formaldehyde emission values below the limit of US Environmental Protection Agency Title VI to the Toxic Substances Control Act (0.09 ppm) [29] were used in this study: standard type with average density of 660 kg/m^3, low emission type with average density of 630 kg/m^3, and low-density engineered particleboard type with nonstandard density profile and average density of 530 kg/m^3. The same wood material mix consisting mostly of conifers and the same melamine-reinforced urea-formaldehyde glue were used in the production of the particleboard types with different thicknesses (Table 1).

Samples corresponding to different production batches for each of the three particleboard types were acquired from a continuous particleboard manufacturing line and cut directly after production for measuring the formaldehyde emissions and content.

2.2. Chamber Measurements. Formaldehyde emission measurements with 1 m^3 chamber were performed according to the ASTM D6007-14 method (0.43 m^2/m^3 of particleboard at 25°C, 50% relative humidity, and Q/A ratio of 1.173 m/h). Q/A ratio is the ratio of air flow through the chamber (Q) to the sample surface area (A) in m/h that needs to be kept constant when changing chamber size according to ASTM D6007-14. The Q/A ratio equation is presented below:

$$\frac{Q}{A}\text{ratio} = \frac{N}{L} = \frac{Q/V}{A/V}, \quad (1)$$

where N is the air exchange rate, which is the air flow through the chamber (Q) divided by the interior volume of the chamber (V) and L is the loading factor, which is the sample surface area (A) divided by the chamber volume (V).

Formaldehyde emission measurements were also performed with 0.044 m^3 dynamic microchamber (DMC) by keeping the same Q/A ratio, temperature, and relative humidity. In DMC, the area of the three samples is 0.46 m^2 leading to loading factor of 10.39 m^2/m^3 according to equation $L = A/V$. In order to keep the Q/A ratio same, a higher air exchange rate (N) of 12.19 1/h is required following the equation $N = L * (Q/A)$ ratio. The differences between the experimental settings of the 1 m^3 and the 0.044 m^3 DMC chambers are summarized in Table 2.

For the 1 m^3 chamber, two different analysis methods were used: the standard acac method and the method that uses Aero-laser AL4021 analyser (Aero-Laser GmbH, Germisch-Partenkirchen, Germany) with inbuilt acac (Figure 1). Most of the 1 m^3 chamber measurements were done using the standard acac method with spectrophotometer. The Aero-laser was only used to evaluate the emission reduction behaviour of the different particleboard types by taking measurements daily for eight days after production. All DMC measurements were done using the electrochemical sensor.

According to ASTM D6007-14, three full air changes or 15 minutes, whichever is greater, is required before measurements [30]. Measurements with 0.044 m^3 DMC were thus limited by the 15-minute limit, while the 1 m^3 chamber required minimum 6 hours to reach three air changes before measurements. Due to the much faster measurement time, DMC was chosen for the 1-day to 7-day emission measurements. For this evaluation, values were taken at day one and at day seven after the production.

2.3. Perforator Measurements. For perforator measurements, samples (25 × 25 × thickness mm^3) were cut from the cooled particleboards, and approximately 110 g thereof was weighted for the determination of formaldehyde content according ISO 12460-5:2015 [32]. According to this method, samples are boiled for 2 h in toluene under reflux, and the extracted formaldehyde is sampled through perforation in water. The formaldehyde content is analysed photometrically using the acetylacetone (acac) method that is based on the Hantzsch reaction. The aqueous formaldehyde reacts with ammonium ions and acetylacetone to form diacetyldihydrolutidine (DDL) that has absorbance maximum at 412 nm (ISO 12460).

The obtained perforator values were adjusted to moisture content of 6.5% by multiplying with the perforator correction factor F that was calculated from the formula published by Jann and Deppe [33]:

$$F = -0.133u + 1.86, \quad (2)$$

where u is the moisture content of the particleboards. The final results were expressed in milligrams per 100 g of dry board. The calculated formaldehyde value is affected by storage conditions as well as by the panel type [25], which is why all samples were measured for their formaldehyde content within two days after production to minimize the effect of storing.

2.4. Statistical Analysis. To explore the relationships between the formaldehyde control methods (1 m^3 chamber, DMC, and perforator), linear regression analysis was employed. For each data set, upper and lower outlier fences were calculated using the formulae Q1-IQR and Q3 + IQR, respectively, where Q1 is the 1st quartile, Q3 is the 3rd quartile, and IQR is the interquartile range. Based on these outlier fences, outlier values were calculated. Each outlier was evaluated using DFFITS (difference in fits) to evaluate the influence on the model using the given equation:

$$\pm\max\left(1, 3\sqrt{p, n}\right), \quad (3)$$

where n is the number of runs, and p is the number of terms in the model. t-test was also performed to compare the formaldehyde emissions of particleboards measured using DMC after

Table 1: Information on the particleboard types used in the study.

Identifier	Particleboard type	Target density (kg/m^3)	Thickness (mm)
A	Standard	660	18 20
B	Standard, low emission	630	12 15 18 22
C	Engineered, low-density	530	18

Table 2: Differences between the experimental settings of the 1 m^3 and the DMC chamber methods.

Chamber method	Volume (V) (m^3)	Sample area (A) (m^2)	Loading factor (L) (m^2/m^3)	Air exchange rate (N) (1/h)
1 m^3	1.00	0.43	0.43	0.50
DMC	0.044	0.46	10.39	12.19

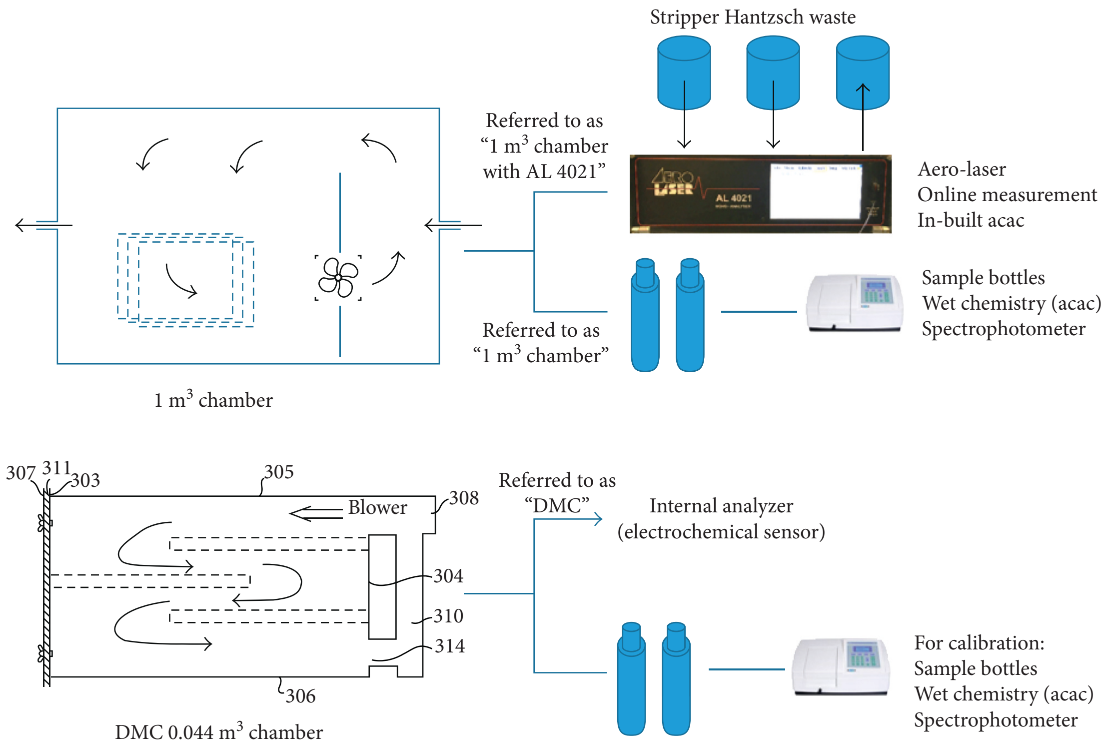

Figure 1: Air flow inside the 1 m^3 chamber and the dynamic microchamber (DMC) [31], the different measurement pathways, and how they are referred to in this article.

one day and seven days from the production. For all statistical analyses, Design-Expert® software version 11 was used.

3. Results and Discussion

For higher emissions (>0.09 ppm), and when using standard particleboards, the perforator gives satisfactory correlation to most chamber methods [34]. As an example, a high correlation ($r^2 = 0.93$) between EN 120 perforator content and ASTM D6007 emission for 16 mm standard particleboards at emission range 0.06–0.24 ppm has been reported [35]. However, it was stated that improvements are required for the perforator method. There are more publications stating nonexistent or poor correlation between perforator and chamber methods for low emission levels [24, 25]. Thus, this unsatisfactory correlation is the main driver for finding an alternative to the perforator formaldehyde content measurement method. The need is also supported by legal requirements for appropriate formaldehyde emission methods at emission values below US EPA TSCA Title VI level (0.09 ppm measured with ASTM E1333 for particleboards) [29, 36].

As a background check, it was studied how the 7-day 1 m^3 chamber emission values and the perforator ISO 12460-5 values relate to each other. The relationship was evaluated statistically based on Pearson correlation. No significant correlation was found for any particleboard type separately nor when all types were evaluated together ($p > 0.05$, Figure 2). The perforator-to-chamber method correlation is sensitive to the density and type of panel in question. Thus, when groupings of panels are done solely by thickness for certification purposes as in Figure 2 (e.g. EPA TSCA Title VI certificate), the correlation, if there is any, will further decrease.

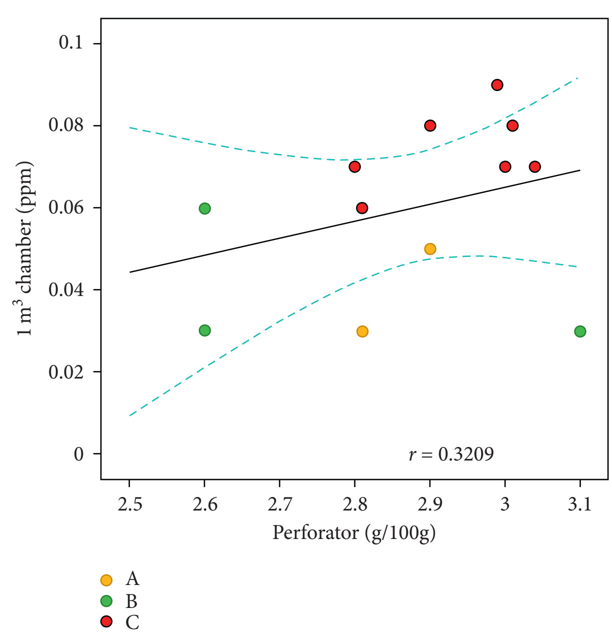

Figure 2: Correlation between 1 m^3 chamber (following ASTM D6007-14) formaldehyde emissions and ISO 12460-5 perforator formaldehyde content for 18 mm particleboards of three types. Circles represent values measured from different production batches, and dotted lines represent confidence bands. No outliers exist. Upper and lower fences are 0.114 and 0.004.

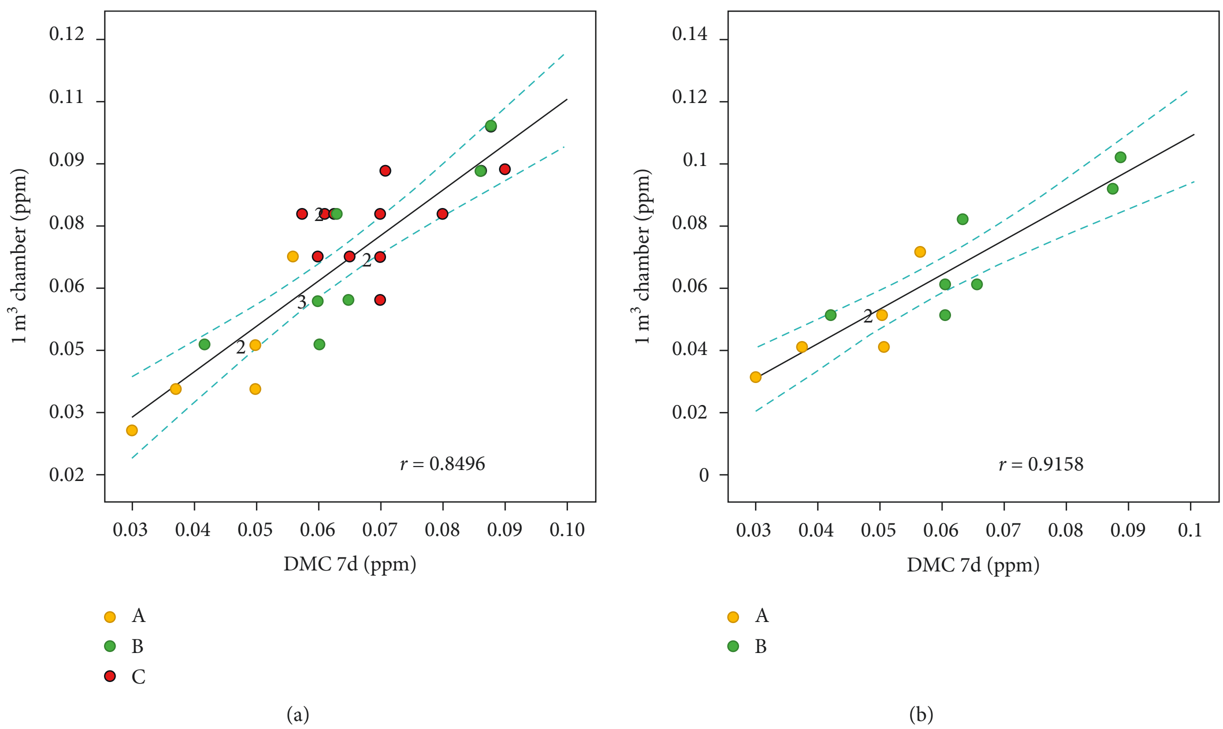

Figure 3: Correlation between the 1 m^3 chamber (following ASTM D6007-14) and DMC chamber formaldehyde measurements (a) for all particleboard types (A, B, and C) and (b) for particleboard types A and B. Circles represent values measured from different production batches, dotted lines represent confidence bands, and the numbers before circles indicate more than one measurement at the same point. No outliers exist for (a) and (b). Upper and lower fences are 0.125 and 0.005 for (a) and 0.10 and 0.02 for (b).

Perforator is still extensively used in particleboard industry, both in Europe and in Asia, since there is a lack of reliable fast formaldehyde measurement method alternatives and due to the high price of chambers.

The small chamber ASTM D6007 and the 0.044 m^3 dynamic microchamber (DMC) are widely accepted (e.g. US EPA TSCA Title VI) and included in the list of approved formaldehyde control test methods to ASTM E1333. The small chamber size and higher air exchange rate however can increase error sourced from the unhomogeneous nature of the particleboards themselves. Inconsistencies caused by sample heterogeneity and chamber conditions have previously been reported by Salem et al. [37]. This hypothesis is supported by the fact that correlation between ASTM and DMC emissions for the most unhomogeneous particleboard type C was poor, lowering the correlation (Figure 3(a)). The combined correlation (positive) for particleboard types A and B was strong ($p < 0.0001, r = 0.9158$, Figure 3(b)).

In industrial practice, the seven-day waiting time of ASTM chambers is too long for an efficient quality control of the production. Because of this, panel manufacturers using DMC or small chambers usually apply correlations between the measurements taken at their internal factory laboratories (after 0–3 days) and the final seventh day emissions, typically done by external laboratories. However, there is no universal way of doing this procedure.

In this study, the correlation between the 1-day emissions and the 7-day emissions using a DMC were evaluated for three different industrially produced particleboard types. No correlation was detected ($p > 0.05$, Figure 4(a)) for the low density particleboards C (530 kg/m^3). However, the emission reduction from day one to day seven was significant ($p < 0.0001$) for this particleboard type as can be seen in Figure 5.

The particleboard types A and B with higher density above 600 kg/m^3 had significant positive correlations between the 1-day and 7-day DMC emissions (A: $r = 0.8721$, $p = 0.0022$; B: $r = 0.8857$, $p < 0.0001$). The particleboard types A and B had similar density and formaldehyde emission profile, and the combined correlation was better than the individual ones between the 1-day and 7-day emissions (AB: $r = 0.9099$, $p < 0.0001$, note two outliers, Figure 4(b)). Based on these results, grouping the A and B particleboard types is acceptable, while the type C particleboard should be evaluated separately.

It should be noted that the samples of particleboard type B originated from three 12 mm- and two 15 mm-thick production batches, while the rest of the samples were between 18 mm and 22 mm in thickness (Figure 5). Thinner particleboards have typically higher emissions [38]. In this case, the thin particleboards (12 mm and 15 mm) had significantly higher final emissions than the thick boards (18–22 mm) ($p < 0.05$), but there was no significant difference in the emission reduction behaviour from day one to day seven ($p > 0.05$). However, evaluating the thin and the thick particleboards separately increased the correlation slightly ($r = 0.9137$ for thicker particleboards, $R^2 = 0.9210$ for thinner particleboards, Figure 6).

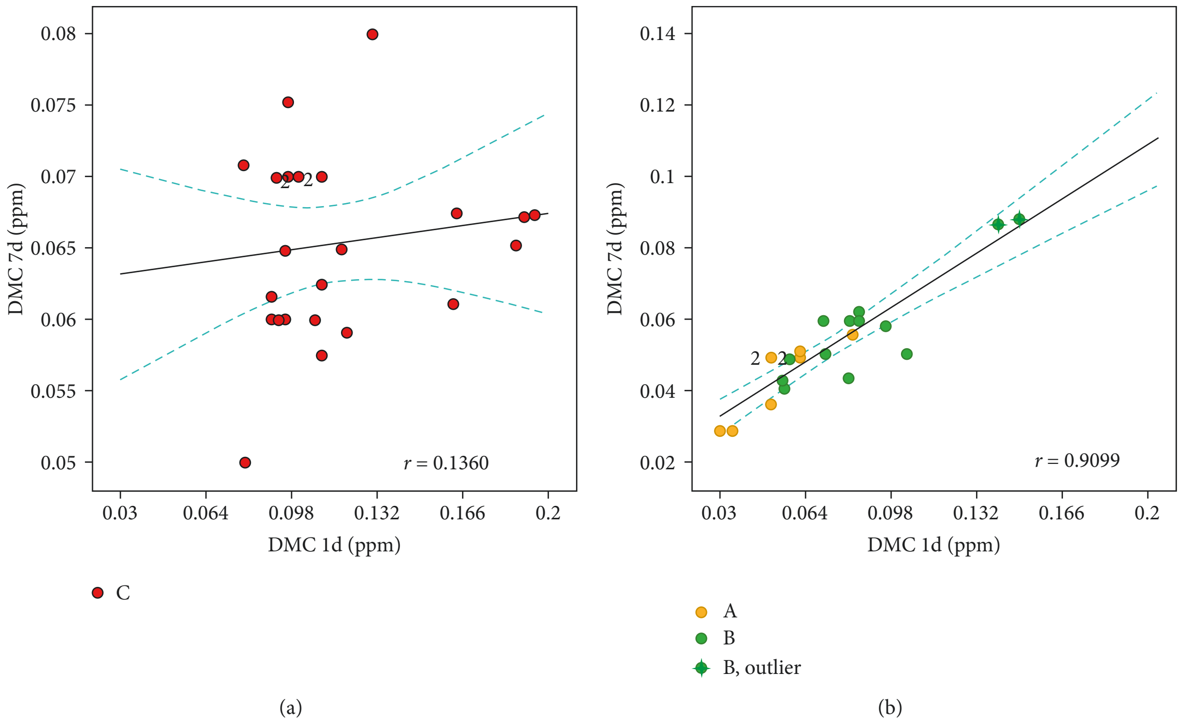

Figure 4: Correlation between the 1-day and 7-day DMC values for (a) particleboard type C (low-density) and (b) particleboard types A (standard) and B (standard, low-emission) combined. Circles represent values measured from different production batches, dotted lines represent confidence bands, and numbers before circles indicate more than one measurement at the same location. No outliers exist for (a), and two values have been marked in the figure as outliers in (b). Upper and lower fences are 0.085 and 0.045 for (a) and 0.081 and 0.024 for (b). The outliers in (b) were not excluded due to low influence on fitted values (DFFITS 0.319 and 0.524).

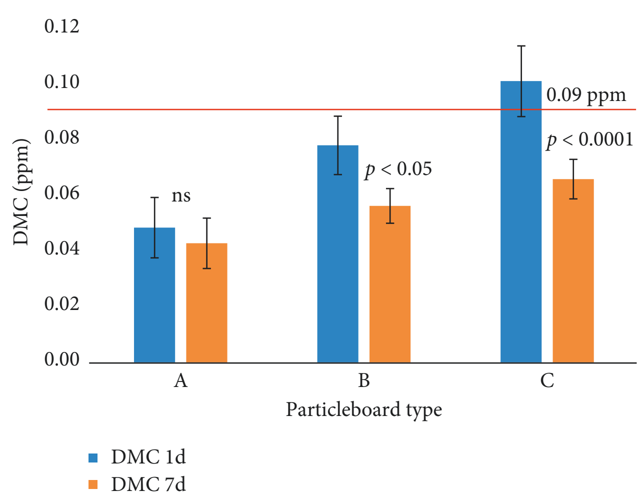

Figure 5: Formaldehyde emissions measured using DMC after one day and seven days from the production of particleboard types (A, B, and C) The EPA TSCA Title VI level of 0.09 ppm for particleboards is marked in the graph. Bars represent mean values of sample determinations for different production batches (7 for A, 8 for B, and 19 for C), and the error bars represent standard deviations. Significance of differences between 1- and 7-day mean values for each board type is marked above the bars.

In evaluation of any technique for measuring the formaldehyde emissions of wood-based panels, the exogenous factors such as temperature, humidity, air change rate, and loading factor need to be considered [24]. These parameters can affect low-density and nonhomogeneous particleboards in a different way than standard particleboards. Less pressure is used in production of the low-density particleboards, which can lead to less free formaldehyde escaping during the pressing. Thus, taking the first measurement already at day 1 might cause error as the emissions for low-density boards are still high, and small changes in the particleboard production can affect the initial level greatly.

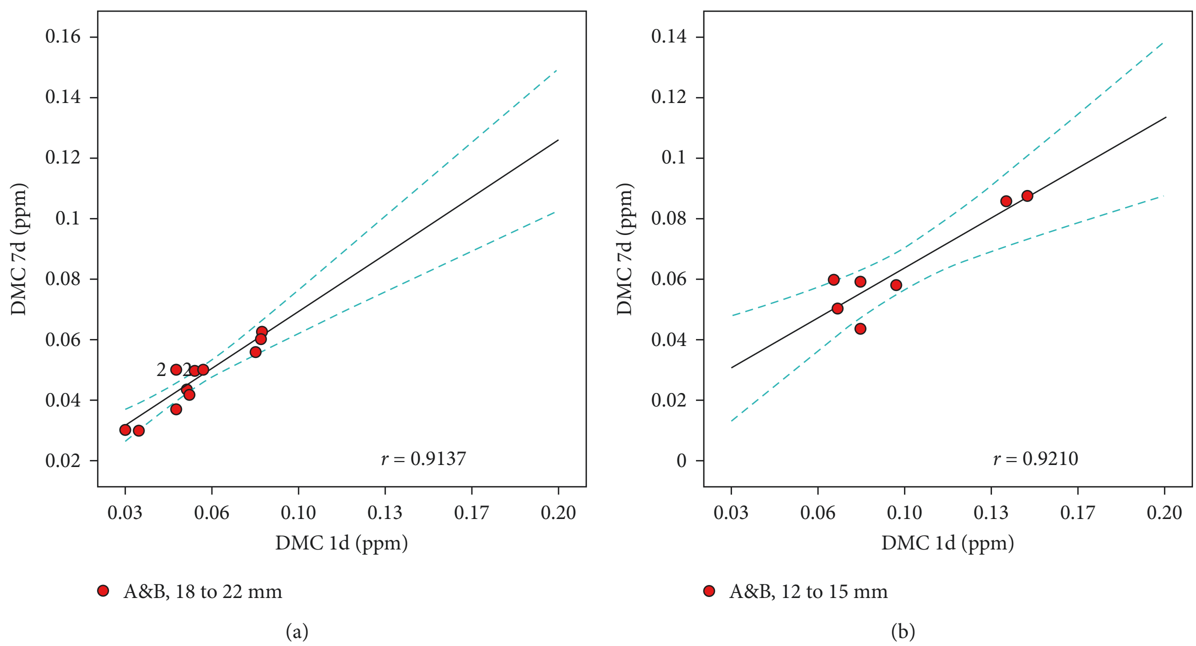

Figure 6: Correlation between the 1-day and 7-day DMC emissions of the particleboard types A and B with thickness of (a) 18 to 22 mm and (b) 12 to 15 mm. Circles represent values measured from different production batches, dotted lines represent confidence bands, and numbers before circles indicate more than one measurement at the same location. No outliers exist for (a) and (b). Upper and lower fences are 0.064 and 0.030 for (a) and 0.101 and 0.027 for (b).

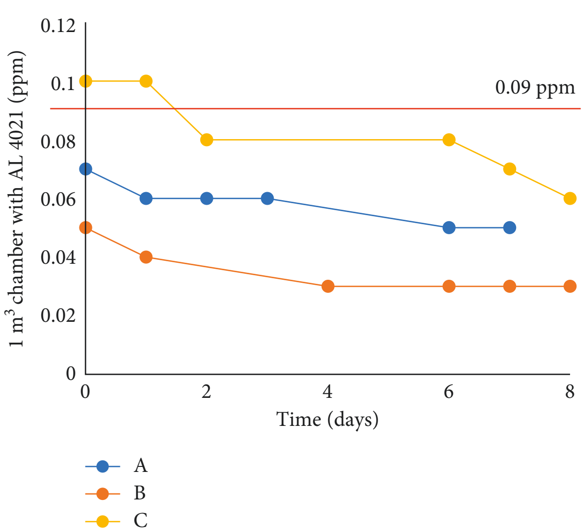

Figure 7: Formaldehyde emission (measured with the 1 m^3 chamber using the Aero-laser AL4021) reduction with time (days) after the production for the particleboard types A (standard), B (standard low-emission), and C (low-density). Day 0 is after 2 h conditioning from the production. The EPA TSCA Title VI level of 0.09 ppm for particleboards is marked in the graph.

To investigate the difference in emission reduction behaviour, formaldehyde emission measurements for each particleboard type using the Aero-laser AL4021 connected to the 1 m^3 chamber were taken regularly after the production till day 8 (Figure 7). Getting information on the emissions related to the production time is important since in some cases, it has been reported that highest emissions appear on the second or the third day after starting the measurements [4]. This phenomenon can be caused by the increase of moisture content at the start of the measurement as the particleboards are very dry when coming directly out of the production. The low-density particleboard C had higher initial emission than the high-density standard particleboard A, and standard low-emission board B. The higher 1-day emission was also noted for the DMC measurements (Figure 5). The C type particleboards seemed to hold a high emission level longer than the other two types (day 2 to day 6 after the production) before the emissions decline sharply. A more gradual reduction of formaldehyde emissions was observed for the A and B particleboard types. The board types A and B were already below the permitted emission limit two hours after production. More continuous emission measurements are required to elucidate the time-dependent formaldehyde emission inclination after the particleboard types' production.

4. Conclusions

The poor correlation between the perforator content value and the ASTM chamber methods at formaldehyde emission rates below 0.09 ppm is known. It is thus of high interest to evaluate alternatives to the perforator method for European and Asian markets. In this study, the common practice of creating correlations between fast formaldehyde emission measurements and final seven-day emission values were evaluated using a fast DMC-based method for three different particleboard types. The following could be concluded:

(1) There was a significant correlation between the DMC and ASTM D6007 emissions, both measured at day seven after the production.

(a) The correlation was better for the particleboard types A and B ($r = 0.9158$).

(2) For some board types the 1-day emission values provide an effective formaldehyde control method:

(a) Two of the higher density particleboard types (A: 660 kg/m^3 and B: 630 kg/m^3) were already below the permitted emission limit one day after the production.

(b) For the same particleboard types, there was also a high correlation between the 1-day and 7-day DMC emissions enabling a good prediction of the final emissions. In such cases the 1-day emissions do not necessarily have to be below the permitted emission limit.

(3) The correlation between the 1-day and 7-day DMC emissions is not universal and needs to be investigated for each particleboard type individually. Grouping of particleboards cannot be solely based on their thickness:

(a) For the third particleboard type C with same thickness as the other two types but with lower density (C: 530 kg/m^3) and nonhomogeneous density profile, there was no correlation between the 1-day and 7-day emissions

(b) The formaldehyde emission reduction was significant for the particleboard type C after seven days from the production

The lack of correlation for particleboard type C needs to be further investigated. As the 1-day DSC emissions were high, conditioning with different air exchange rates should be evaluated for faster aging of the particleboards. This could stabilize the 1-day emission values. Further measurements using Aero-laser to evaluate the emission reduction behaviour of particleboards soon after their production until a steady state is reached with more frequent measurement intervals could be utilized in this work.

Overall, the method of using the 1-day DMC emission values for a quick formaldehyde control at the industry seemed to be suitable for some particleboard types. However, the significance of evaluating each particleboard type separately was highlighted in this study by the varying results of the low-density particleboards.

Data Availability

The data used to support the findings of this study were provided by the particleboard manufacturer in question under license, and so cannot be made freely available. Access to these data will be considered by the author upon request, with permission of the particleboard manufacturer.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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