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Effect of magnesium nanoparticles on formaldehyde emissions from wood composite materials

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Abstract. For the production of wood composite materials, adhesives based on cheap and affordable, but harmful urea-formaldehyde resins are mainly used. Given the substantial production volumes of such materials, it is important to find environmental solutions to reduce formaldehyde emissions during their pressing and subsequent operation. The purpose of the study was to present the results of a study on the use of magnesium oxide nanoparticles to bind unreacted formaldehyde in wood composite materials. Analysis of methods for manufacturing metal nanoparticles allowed determining a priority method that allows obtaining ultrafine structures with a size not exceeding 100 nm, namely, the method of volumetric electric spark dispersion of metals in a liquid.

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Investigating the morphology of magnesium nanoparticles allowed determining that they have an almost crystalline form formed from the vapour phase, with an average particle length not exceeding 100 nm. The results of spectral analysis of the element composition in the nanophase, specifically magnesium and oxygen, demonstrated that the magnesium content does not exceed 32.2%, while oxygen constitutes 67.78%. This indicates that divalent magnesium oxide does not have a pronounced metallic phase, which would interfere with the sorption processes of formaldehyde. The conducted examinations of formaldehyde emission of samples of particle boards with modified magnesium oxide nanoparticles in concentrations of 2% and 8% glue based on urea-formaldehyde resin showed mixed results. Compared to the control samples, the formaldehyde level remained almost unchanged on the second day of follow-up, and for a concentration of 2%, it even increased by 6%. However, by the sixteenth and ninetieth day, a reduction in the level of free formaldehyde emissions was observed at 19% and 22% respectively. The results obtained can be used to improve the production of non-harmful particle boards with improved properties

Keywords: ultrafine structures; metal oxide; production method; samples; harmfuly reduction

Introduction

With the development of nanotechnologies, a substantial body of findings has been accumulated regarding the usage of metal nanoparticles across various fields, ranging from medicine to agricultural production. The usage of nanometals as fillers in adhesives contributes to the enhancement of physical and mechanical properties of medium-density fiberboard (MDF) wood composite panels. A. Pizzi *et al*. (2020) noted that a special feature of the technology of manufacturing wood composite materials is the combination of crushed wood particles with a binder, followed by pressing under the influence of elevated temperature. O. Bekhta & T. Krystofiak (2023) indicated that urea-formaldehyde (UF) resins are predominantly used as binders, which are harmful to human health both during the manufacturing process of MDF and particleboard (PB) panels and their subsequent use. There is experience in reducing unreacted formaldehyde emissions by adsorbing it with nanoscale metals.

In the nanoscale state, there are a large number of atoms with uncompensated bonds on the metal surface, which leads to increased surface energy and their intense interaction with the environment (Lopatko *et al.*, 2020; Murmantsev *et al.,* 2022). The effectiveness of the use of nanomaterials is associated with the appropriate form of preparation and the possibility of long-term storage of chemical and biological activity (Aftandilyants & Lopatko, 2019).

Metals such as iron, zinc, and aluminium are most commonly used. H. Alabduljabbar *et al.* (2020) established that iron nanoparticles are able to bind free formaldehyde and react with it, with the release of CO_2, water on low valence iron. The ability of aluminium to improve the physical and mechanical properties of UF resin to adsorb free formaldehyde contributed to the use of aluminium oxide (Al_2O_3) in various thermosetting polymers. According to X. Tian *et al.* (2017), ZnO is quite sensitive to formaldehyde molecules and is used as part of analysers to detect free formaldehyde in the air.

There are several methods for producing metal nanoparticles: explosion of conductors, electron beam and gas-thermal, mechanical or chemical dispersion, and evaporation-condensation of materials. K.V. Vynarchuk *et al.* (2021) proved that the method of volumetric electric spark dispersion of metals in a liquid has an advantage in obtaining an ultrafine structure not exceeding one hundred nanometers with the specified characteristics. The experience of using this method for processing aluminium granules in organic matter has shown that 92.81% of aluminium nanoparticles with a length of 36.6 nm and a width of 35 nm were formed. Notably, by reducing the grains of metallic materials in the nanoscale range, more effective strengthening of the particle structure is observed (Aftandilyants & Lopatko, 2018). Therewith, the average temperature of the working fluid may be low, but in microplasma volumes of short-term sparks, ultra-high temperatures from 10,000 °C to 12,000 °C occur during 10-100 microseconds. Such flares allow obtaining particles from materials with substantially different melting points. High cooling rates in the liquid of dispersed spark-erosion particles lead to a substantial modification of their structure and the formation of materials with unique properties.

Magnesium oxide MgO, which has a high specific surface area and high absorption efficiency of toxic heavy metal ions and organic pollutants, was examined in a study by Z. Fusheng *et al.* (2022). Experience of using MgO nanoparticles for the destruction of organochlorine compounds, adsorption of large amounts of SO_2, CO_2 HCl, HBr, and other gases allows expecting the binding of unreacted formaldehyde in wood composite materials. This issue could be addressed by incorporating magnesium nanoparticles obtained through the electrospark method into the adhesive.

The purpose of the study was to investigate the use of magnesium nanoparticles as a potential adsorbent of unreacted formaldehyde in wood composite materials.

Materials and Methods

For the study, crushed pine wood with a humidity of 6%, an adhesive based on urea-formaldehyde resin Unicol RESIN 474 (Italy) were used. As a filler, magnesium nanoparticles were used, which were added to the resin solution in the amount of 2% and 8%. For comparison, control samples were made without a nanofiller.

A total of 9 samples with a thickness of 10 mm and a diameter of 41 mm were produced. The Temtop M2000 device (China) was used to measure free formaldehyde values. Measurements were conducted on the second, sixteenth, and ninetieth days after the production of prototypes. To determine the formaldehyde emission experimentally, obtained as a result of measurement (mg/m$^{3)}$ converted to ppm by the formula (Villanueva *et al.*, 2021):

$$C_{ppm} = \frac{C_{mg/m^3} \times 24.45}{M.W.}, \quad (1)$$

where C_{ppm} – the concentration of free formaldehyde is expressed in ppm (mln^{-1}, or a×10^6); $C_{mg/m}{}^3$ – concentration of free formaldehyde in mg/m^3; *M.W.* – molecular weight of the pollutant (g/mol); 24.45 – molar volume of any gas or vapour under normal conditions.

Magnesium nanoparticles were obtained using the method of volumetric spark dispersion (VSD) of metals in liquid (Patent No. 130939..., 2018). The essence of the VSD method is as follows: metal magnesium granules are placed in an aqueous medium in a special reaction chamber, where a voltage pulse passes through a freely enclosed layer of granules, causing current switching in the electrically conductive layer of granules. As a result of this process, there is electrical erosion of the surface of metal granules and the formation of a nanodispersed fraction of magnesium through the condensation of the vapour phase. When all parameters of the discharge circuit are properly coordinated, this nanodispersed fraction constitutes a significant portion of the erosion products. By adjusting the parameters of the discharge circuit (charging voltage of the capacitor and its capacitance), the frequency of pulse passage, their duration, and the initial resistance of the reaction chamber, it is possible to obtain suspensions with varying concentrations of magnesium nanoparticles.

A layer of conductive magnesium granules (5-10 mm) was used in the discharge chamber (Fig. 1) to ensure spark-erosion dispersion, made of a dielectric. For the volumetric electrospark dispersion of metal in liquid, discharge pulses (DP) were generated with currents ranging from 1 to 10 kA, which is 5 to 500 times greater than the average current of the power grid. The electrical resistance of the granule layer (0.1-1 Ohm) depends on the voltage and frequency of the discharge pulses and can stochastically vary within the range of 0.01-10 Ohms with an increase in discharge current by 1.5-5 times (without sparks between the granules), or a decrease by 2-100 times (with a corresponding increase in the duration of the discharge pulses, also without electrical sparks between the granule layer). The parameters of the discharge circuit are subject to changes within the following limits: working capacitor capacity C = 25-200 uF, capacitor charge voltage U_C = 50 - 250 V, and the inductance of the discharge circuit L did not exceed 1 μH. The dispersion process was conducted in a discharge chamber filled with deionised water.

Figure 1. Device for producing metal nanoparticles
Note: 1 – electric spark generator; 2 – discharge chambers; 3 – discharge pulse control unit

The morphology of the dispersed phase was determined using a Zeiss SUPRA 40VP electron microscope (SEM) (Germany).

A colloidal metal solution dried in a Labexpert thermal chamber (Ukraine) and ground to a powdery form was dispersed in distillate to 2% and 8% concentrations to examine the effect of nanomagnum oxide on formaldehyde emission. Adhesive mass according to recipe of A. Kumar *et al.* (2018) was introduced into pre-prepared chips in a ratio of 1:2, respectively. The adhesive weight was 10% of the wood chip weight.

Tarred chips were pressed in a laboratory press in one cycle for 180 seconds, under the schedule: temperature t = 130°C, pressure p = 2.9 MPA. The compressed samples were left under the press to cool to a temperature of t = 40°C. After that, they were kept for 48 hours in a room at an air temperature of t = 20°C and a relative humidity of φ = 60 ± 5% (Fig. 2). The density of the pressed samples was 780 kg/m^3 ± 10% (Mantau, 2012).

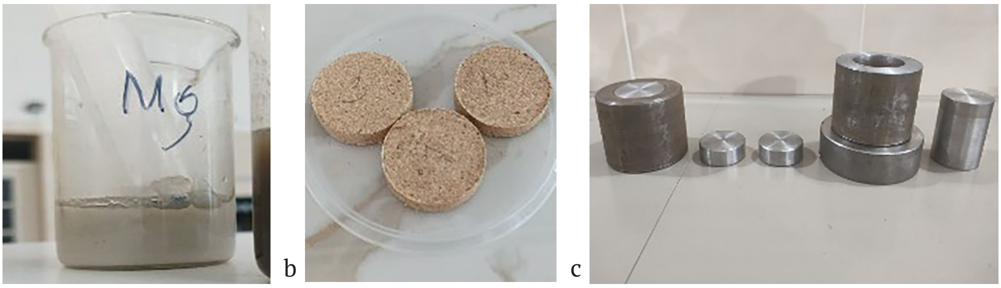

Figure 2. Visualisation of components and accessories for sample production

Note: a – colloidal magnesium solution; b – finished samples using an 8% UF resin-based adhesive modified with Mg nanoparticles in three repetitions; c – moulds for manufacturing samples with an internal diameter of d = 41 mm

The samples together with the Temtop M2000 device (China) were placed under a sealed transparent cap with a volume of 12 litres and the indicators were taken after 15 minutes on the second, sixteenth and ninety days to determine the free formaldehyde emission index. Determination of the formaldehyde emission value was calculated using formula (1).

Results and Discussion

The result of determining the morphology of the dispersed phase is shown in Figure 3.

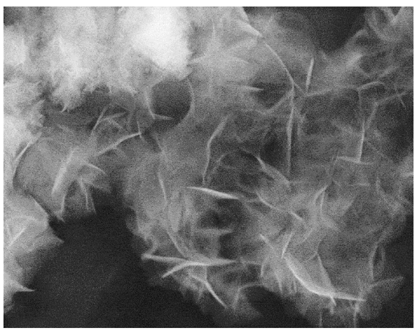

Figure 3. View of magnesium nanoparticles through a Zeiss SUPRA 40VP electron microscope (SEM)

Source: compiled by the authors

Particles that approach the crystalline shape were formed from the vapour phase. The presence of spherical particles in the total mass is the result of spraying the liquid phase. It was established that the average particle size in cross-section does not exceed 10 nm when they are about 60-100 nm long. Therewith, spectral measurements of the quantitative composition of the elements that formed the Nanophase were performed (Fig. 4).

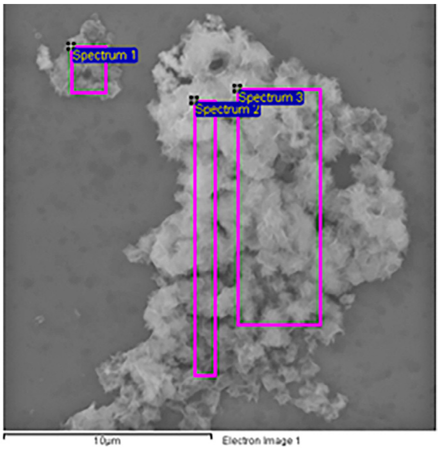

Figure 4. Result of spectral studies of the composition of nanophase elements
Source: compiled by the authors

The general structure of metal nanoparticles obtained by electric spark dispersion in water implies the presence of an oxide layer on the surface and a metal core. According to the results of microrentgenospectral analysis (Table 1), it can be seen that the amount of magnesium in the total mass of the particle varies from 14.5% to 32.2%, the rest is oxygen. Considering the atomic mass of magnesium and its total amount in the examined phase, it can be assumed that almost all magnesium forms a MgO compound, and magnesium nanoparticles do not have a pronounced metallic phase. Thus, two-valence magnesium oxide MgO is mainly involved in sorption and other processes of interaction between the Nanophase and the medium.

The results of experimental studies are shown in Figure 5 and Figure 6.

Table 1. Results of microrentgenospectral analysis of magnesium nanoparticles

Spectrum	In stats.	O	Mg	Total
Spectrum 1	Yes	85.55	14.45	100
Spectrum 2	Yes	70.73	29.27	100
Spectrum 3	Yes	67.78	32.22	100
Spectrum	**O**	**Mg**		
Max.	85.55	14.45		
Min.	67.78	32.22		

Source: compiled by the authors

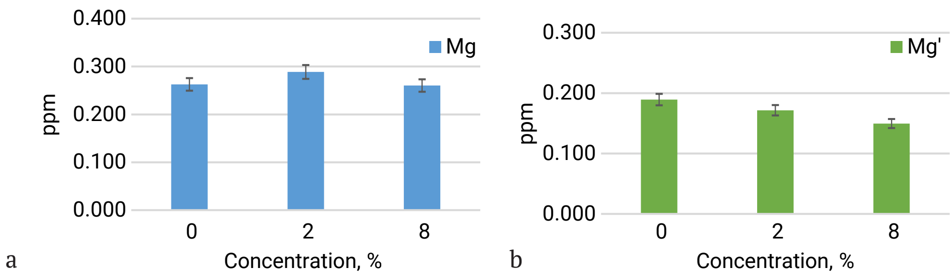

Figure 5. The level of free formaldehyde emission when using an adhesive based on UF resin modified with magnesium nanoparticles in various concentrations

Note: a – on the second; b – on the sixteenth day after pressing

Source: compiled by the authors

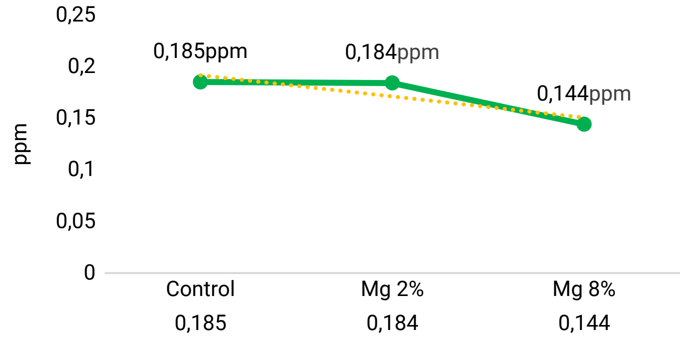

Figure 6. Emission of free formaldehyde when using an adhesive based on UF resin modified with magnesium nanoparticles in various concentrations of 2% and 8% on the ninetieth day

Source: compiled by the authors

On the second day, when using magnesium, the value of the isolated formaldehyde was on average 0.263 ppm for samples modified with a 2% solution and 0.260 ppm for 8%, while the control indicators were 0.188 ppm. On the sixteenth day, the control remained at a constant level of 0.186 ppm, while Mg 2% was 0.189 ppm and 0.150 ppm for 8%, which is 19% lower than the control.

On the ninetieth day, repeated measurements were made to understand the effect of nanoscale magnesium on the formaldehyde binding process in the long-term use of chipboard. The following results were obtained: for samples modified with a 2% colloidal solution of magnesium nanoparticles – 0.184 ppm, for 8% – 0.144 ppm, which is a 22% reduction in the emission of the test gas relative to the control. In turn, the level of formaldehyde emission in samples not modified with a nanofiller fluctuated within the standard error and amounted to 0.185 ppm.

Similar research results were obtained by G. Paul *et al.* (2021) when using magnesium lignosulfonate in the adhesive formulation. However, an increase in the content of lignosulfonate solution in the adhesive formulation, according to the authors, can lead to an increase in moisture and the content of the combined-cycle gas mixture during the pressing process, and an increase in the brittleness of the plates (Hu *et al.,* 2015; Bekhta *et al.*, 2021). Studies by E. Athanassiadou *et. al.* (2009), A. Pizzi (2013), and S. Costa *et al.* (2019) showed a reduction in formaldehyde emissions (up to the production of panel class – E1) when using magnesium lignosulfonates in the case of wood composite boards such as MDF or plywood, using a filler content of more than 20% (Alonso *et.al.*, 2005).

The results obtained show that magnesium has the ability to act as an adsorbent due to the fact that a strong four-membered ring involving a carbonyl group (CO) is formed on its surface links. This results in the weakening of the carbonyl bond to nearly a single bond. Nanoscale magnesium can be considered as a stoichiometric chemical reagent. Its high surface area means that 30-40% of magnesium fragments are located on the surface, which allows adsorption reactions to occur in the stoichiometric range, and its electronic structure involves only s-p electrons.

According to M. Nagpal & R. Kakkar (2020), there are several ways to adsorb formaldehyde onto MgO nanostructures. Depending on the coordination and type of defect site, different adsorption products are formed, and all reactions are exothermic. Low-coordination magnesium centres have high reactivity and adsorb the formaldehyde molecule. Oxygen from the carbonyl group coordinates with the surface centre of magnesium, which leads to the formation of a four-coordinate complex, in which the carbonyl bond is substantially weakened, which leads to the destruction of formaldehyde.

In the works of researchers P. Cademartori *et al.* (2018), W. Gul *et al.* (2021), it is established that the experience of using magnesium oxide for formaldehyde adsorption is still insubstantial, but there is interest in using nanoparticles of other metals to reduce the toxicity of particle boards. The positive effect of aluminium, iron, and zinc oxide nanoparticles on the binding of free formaldehyde when the latter were introduced into the UF resin solution was observed in the studies by M. Salem *et al*. (2013), S. Dinesh Ram *et al*. (2022).

Considering the surface feature of magnesium nanoparticles, P. Gabriela *et al.* (2022), A.E. Elsayed *et al.* (2023) consider it appropriate to use them for binding unreacted formaldehyde in the manufacture of wood composite materials, which will contribute to the environmental friendliness of both the technological process and products during operation. This modification of wood-composite materials makes it possible to obtain materials with additional characteristics and added value (M. Shvets *et al.*, 2020). Moreover, the issue of reducing the toxicity of finished products made of wood composite materials is a priority for the furniture industry (Pinchevska & Šmidriaková, 2016).

Conclusions

Analysis of studies on the use of metal nanoparticles – iron, zinc, and aluminium as a modifier of UF resin to reduce formaldehyde emissions showed the possibility of reducing free formaldehyde emissions into the air during the long-term operation of wood composites. The experience of using magnesium oxide, which has a high specific surface area and high absorption efficiency of hamful heavy metal ions and organic pollutants, allowed determining the possibility of using nanomagnet as an adsorbent for binding unreacted formaldehyde in wood composite materials. The scientific originality lies in the production of magnesium oxide nanoparticles using the electric spark method, which made it possible to achieve average particle sizes in the range of 5-60 nm. Experimental studies of the production of magnesium nanoparticles by the electric spark method and their subsequent use as a binder filler for the production of chipboard samples confirmed the hypothesis of reducing formaldehyde emission. Further research is necessary to determine the optimal concentration of nanometals in the adhesive used for the manufacture of wood composite materials.

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Conflict of Interest

The authors declare no conflict of interest.

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Вплив наночастинок магнію на показники викидів формальдегіду з деревино композиційних матеріалів

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Анотація. Для виробництва деревних композиційних матеріалів переважно використовують клеї на основі дешевих та доступних, проте токсичних карбамідоформальдегідних смол. Враховуючи значні обсяги виробництва таких матеріалів, актуальним є пошук екологічних рішень щодо зменшення викидів формальдегіду під час їх пресування та подальшої експлуатації. Метою статті було представити результати дослідження з використання наночастинок оксиду магнію для зв'язування непрореагованого формальдегіду у деревинокомпозиційних матеріалах. Аналіз способів виготовлення наночастинок металів дозволив визначити пріоритетний метод, що дозволяє отримати ультрадисперсні структури, розмір яких не перевищує 100 нм, а саме метод об'ємного електроіскрового диспергування металів у рідині. Вивчення морфології наночастинок магнію дозволило визначити, що вони мають майже кристалічну форм яка утворилася з парової фази, та середня довжина частинок не перевершує 100 нм. Результати спектральних досліджень складу елементів у нанофазі а саме магнію і кисню, показали, що кількість магнію не перевищує 32,2 %, а 67,78 % займає кисень. Це свідчить про те, що двохвалентний оксид магнію не має вираженої металевої фази, яка б заважала процесам сорбції формальдегіду. Проведені дослідження емісії формальдегіду зразків деревностружкових плит із модифікованим наночастинками оксиду магнію в концентраціях 2 % і 8 % клеєм на основі карбамідоформальдегідної смоли показали неоднозначні результати. У порівнянні з контрольними зразками на другу добу спостереження рівень формальдегіду майже не змінився, а для концентрації 2 % навіть збільшився на 6 % . Проте, вже на шістнадцяту і дев'яносту добу спостерігалося зниження рівня виділення вільного формальдегіду на 19 % та 22 % відповідно. Отримані результати можуть бути використані у вдосконаленні виробництва нетоксичних деревностружкових плит з покращеними властивостями

Ключові слова: ультрадисперсні структури; оксид металів; спосіб отримання; зразки; зниження токсичності