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THE EFFECT OF METAL NANOPARTICLES ON FORMALDEHYDE EMISSION FROM WOOD BASED MATERIALS

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ABSTRACT

Considering the significant production volume of wood composite materials, the search for ecological solutions to reduce formaldehyde emissions during their pressing and further operation is urgent. The effect of nanoparticles of zinc, aluminium, and iron metals on formaldehyde emission from particleboard samples during their manufacture and subsequent exposure for three weeks is examined in the paper. Nanoparticles of metals were produced by the electro-spark method. In order to determine the priority metal, the method of analysis of hierarchies and search experiments was used. Samples of particleboard were made with the addition of a modified glue based on UF resin. Nanoparticles of the metals Zn, Al, and Fe were used in concentrations of 2% and 8%, respectively, to modify the adhesive resin. Measurements of the level of free formaldehyde were carried out on the third and seventeenth days after the pressing of particleboard samples. The results of experimental studies confirmed the theoretical calculation of priority based on the selected assessment criteria. The best results were observed when zinc was used as a filler at a concentration of 8%.

Keywords: particleboard; formaldehyde; metal nanoparticles; urea-formaldehyde resin; adhesive modification.

INTRODUCTION

One of the most important factors in the production of wood composite materials is their environmental friendliness, both during manufacture and in further operation. According to the Food and Agriculture Organisation (2020), the industrial production of wooden boards in the world reached 250 million m^3, of which the production of fibreboard, particleboard, and oriented strand board (OSB) amounted to 250 million m^3. Phenol-formaldehyde (PF) and urea-formaldehyde (UF) resins are mainly used for their production. According to the Fraunhofer Institute (2012), 80-85% of particleboard is manufactured using adhesives containing formaldehyde. Despite this, particleboard furniture is the most popular. Since wood particles are poor conductors of heat, during hot pressing of particleboard, it is distributed unevenly (Bolton *et al.*, 1989). The core of the composite reaches the curing temperature later than the outer part. Unreacted methyl (hydroxymethyl) groups and methylene ether groups remain in the hardened resins. Over time, they diffuse to the surface and slowly degrade, releasing formaldehyde into the environment. Changes in temperature and air humidity affect the acceleration of the degradation process. The release of formaldehyde and volatile organic compounds from new furniture was revealed after several months (Sherzad and Jung 2022, Ghani *et al.*, 2018).

The danger of formaldehyde is related to its effect on the human body as a whole, its primary emissions, at a concentration of 0.1-5 ppm, can contribute to respiratory tract irritation, headache, dizziness, and lacrimation. According to research by the National Academies of Sciences, Washington (1980), the negative impact of formaldehyde on the lymphatic system of the human body was revealed, which leads to myeloid leukemia, cancer of the nasal cavity, damage to bone marrow function (Lv *et al*., 2020, Wei *et al.,* 2016, Wei *et al*., 2017, Kang *et al.*, 2021), etc.

The issue of modifying polycondensation resins with various fillers, both organic and inorganic, is relevant. Among the inorganic fillers, the use of nanoparticles of various metals deserves attention (Lopatko *et al*., 2013), because they have little effect on the viscosity of the glue, but they close the vessels of the wood well, preventing the adhesive from penetrating its thin layers. Adsorption of glue and the occurrence of the "starved gluing" defect are prevented (Bekhta and Bits 2008).

It was found (Gul *et al.,* 2017) that the influence of Fe_2O_3 nanoparticles as a filler improved the resistance to swelling across the thickness of medium-density fibreboard (MDF). At the same time, the pressing time of the panels was shorter due to the increase in thermal conductivity. Iron nanoparticles are able to bind free formaldehyde and react with it, releasing CO_2, water, and ferrous ions. In the presence of iron with a low degree of oxidation and in a small concentration, when interacting with lignin (Wan and Frazier 2017), a Fenton reaction occurs, during which new formaldehyde molecules are formed. It is believed that the mechanism of formaldehyde reduction is the ability of metal nanoparticles to accelerate the oxidation of formaldehyde and the formation of less harmful products. Additionally, iron oxide nanoparticles can absorb formaldehyde by adsorption and then slowly release it over time. The use of zinc in the manufacture of MDF boards (Gul *et al*., 2017) showed that mechanical properties and moisture resistance of the panels were improved. According to Tian *et al.* (2017) and Salem *et al.* (2013), ZnO is quite sensitive to formaldehyde molecules, it is used as part of analysers to determine free formaldehyde in the air. Moreover, when the humidity of the material increases, the adsorption of formaldehyde molecules shortens the bond distance between water molecules and zinc oxide, thereby increasing the average bond energy of the entire system. At the same time, formaldehyde is adsorbed by the ZnO surface (Jin *et al.,* 2017, Giroto *et al.,* 2021, Gul *et al.*, 2021, Schmidt-Mende and Macmnus-Driscol 2007).

The ability of aluminium (Alabduljabbar *et al*., 2020, Kumar *et al.,* 2013a, Shukla and Parameswrn 2007) to improve the physical and mechanical properties of UF resin and to adsorb free formaldehyde contributed to the use of aluminium oxide (Al_2O_3) in various thermosetting polymers. Al^{3+} ions have the property of reducing the duration of gluing due to the filling of micropores in wood without changing the structural structure of glue based on urea formaldehyde resins (Cademartori *et al.*, 2018, Kumar *et al*., 2013a). At the same time, during the hydrolysis of aluminium salts, a certain amount of H^+ ions are released, which neutralize the excess alkalinity of the resin. Therefore, the use of complexing agents with aluminium to modify urea-formaldehyde resins deepens the hardening process and increases the water resistance of the glue. In addition, aluminium, like zinc, is a catalyst for the polycondensation process (Du *et al.,* 2019).

Despite a significant amount of research, the impact of the metal form of the nanoparticles remains open, as it is more active and potentially has the ability to bind formaldehyde better. Oxide, sulphide, or other forms of metals are usually used because they are easier to obtain (Gul *et al.,* 2020, Tian *et al.,* 2017, Cademartori *et al*., 2019, Kumar *et al.,* 2013b, Chotikhun *et al.,* 2018). As a result, the study of the influence of the metal form of nanoparticles on the ability to bind formaldehyde was not conducted, however, there is a method that allows obtaining nanoparticles with a reduced content of oxide forms in an affordable way (Olishevskyi *et al.*, 2018). In addition, nanoparticles can have a fairly wide range of sizes from 1 to 100 nm, and it is appropriate to determine their rational size for reducing formaldehyde emissions.

The aim of the research is to identify a priority nano-sized metal that would ensure the binding of formaldehyde in the process of manufacturing wood composite materials.

MATERIAL AND METODS

For conducting exploratory experimental studies, pine wood (*Pinus sylvestris*) crushed into particles of the size generally used for the middle layer of particleboard with a moisture content of 4% was used. Urea-formaldehyde resin Unicol RESIN 474 (solid content 65%, gel time 50 s) was used for the bonding of particleboard samples. Nanoparticles Fe^0, Al^0 and Zn^0 (metal forms of nanoparticles) were used as fillers. In parallel, samples were made from disintegrated wood without adding the above fillers. The number of pressed samples with a thickness of 9 mm was 12 pieces.

Metal nanoparticles (Figure 1a) were produced by the electro-spark method in the form of colloids according to a patented method (Olishevskyi *et al.*, 2018). Colloids were evaporated to powders in a laboratory drying oven, then homogenized in a mortar and dispersed in distillate to concentrations of 2% and 8%. Dispersed solutions were introduced into the adhesive mass in a ratio of 1:2, respectively, and thoroughly mixed for 20 minutes until complete homogeneity.

The mass of the adhesive was 10% of the mass of the wood chips. Resinated chips were placed in a mould and pressed in one cycle in a laboratory press for 180 seconds under the following regime: temperature t = 130 °C, pressure p = 2.9 MPa. After that, the samples were left under the press to cool down to a temperature of t = 40 °C. The finished samples were kept for 48 hours in the room at an air temperature of t = 20 °C and relative humidity of φ = 60 ± 5%. The dimensions of the samples were d = 41 mm, h = 9 mm (Figures 1b, c). The density of the pressed samples was 800 kg/m^3 ± 10%.

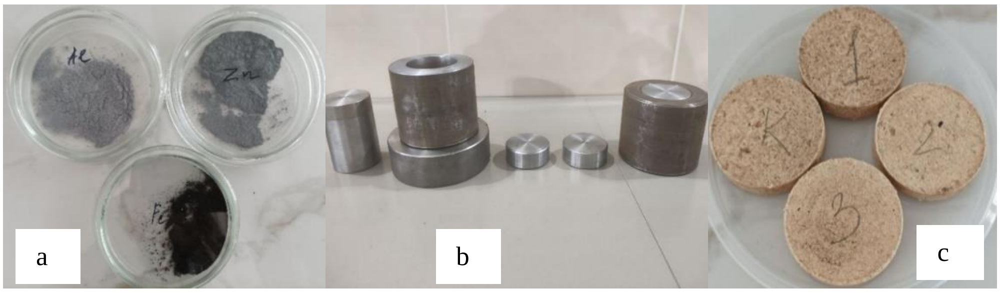

Fig. 1 Visualisation of components and equipment for making samples: a) nanoparticles of the studied metals Al^0, Zn^0, and Fe^0 dried to powders; b) moulds for making samples with internal d = 41 mm; c) ready-made samples using 8% of Al nanoparticles to modify UF adhesive in three repetitions and control.

To select a prioritised nanosized metal, one of the methods of fuzzy logic was used, the analytic hierarchy process (AHP), which consists of decomposing the problem into simpler component parts and gradually establishing the priorities of the evaluated components using pairwise comparisons (Burak 2013). For this reason, the selection of a priority nanosized metal, alternative options for its achievement, and criteria for evaluating the quality of alternatives are set as a goal. Nanoparticles of the following metals were selected as alternatives: A_1 – Fe, A_2 – Al, A_3 – Zn; as criteria: Cr_1 – price, Cr_2 – production time, l/min, Cr_3 – magnetism, Cr_4 – particle size, nm; Cr_5 – adsorption capacity, mg/g.

After determining the priorities of all elements of the hierarchy, the method of paired comparisons is used using Saaty's scale (Burak 2013). The matrix of pairwise comparisons (MPC) of the criteria is built with respect to the goal, and the MPC of the alternatives is built with respect to each criterion.

For each matrix, the geometric mean, *Gi*, and the local priority, LPr_n, of each row of the matrix are calculated:

$$G_i(a_{i1}, a_{i2}, \ldots a_{is}) = (a_{i1} * a_{i2} * \ldots * a_{is})^{\frac{1}{s}} \quad (1)$$

Where: *i* – the row number of the matrix,
s – the number of elements in the *i*-th row of the matrix.

$$a_{i1} = w_1/w_1;\ a_{i2} = w_1/w2;\ \ldots\ a_{is} = w_1/w_s \quad (2)$$

w – the accepted numerical value on Saaty's scale.

$$LPr_n = \frac{[(w_n/w_1)(w_n/w_2)\ldots(w_n/w_n)]}{(G_1+G_2+\ldots+G_n)} \quad (3)$$

Where: n – the MPC line number.

To control the consistency of expert assessments, two related characteristics are used - the consistency index (CI) and the consistency ratio (CR):

$$CI = \frac{\lambda_{max}}{m-1} \quad (4)$$

$$CR = \frac{CI}{P_m} \quad (5)$$

Where: m – the size of the matrix,
Pm – the consistency index for the positive inverse symmetric matrix of random estimates of size *m x m*,
λ_{max} – the maximum eigenvalue of MPC:

$$\lambda \sum a_{1i_1} \sum a_{2i_2} \sum a_{ni_{n_{max}}} \quad (6)$$

Where: $\sum a_{1i}$ – the sum of the values of the first column of the MPC,
LPr_1 – the value of the local priority of the first line of the MPC,
When *CR* < 0.1 ... 0.2, the calculations are considered satisfactory.

The solution to the multi-criteria ranking problem is presented in the form of a global priority vector (GlPr) of alternatives in relation to the goal.

The Temtop M2000 device was used to determine the formaldehyde emission. This is a non-standard method, but it is suitable for comparing the efficiency of used formaldehyde scavengers. The resulting values of formaldehyde emission as a result of the measurement (mg/m^3) were converted into ppm according to the formula (Saltzman 2013):

$$C_{ppm} = \frac{C_{mg/m^3} \times 24.45}{M.W.} \quad (7)$$

Where: C_{ppm} – the concentration of free formaldehyde expressed in ppm (million),
$C_{mg/m3}$ – concentration of free formaldehyde in mg/m^3,
$M.W.$ – molecular weight of formaldehyde (g/mol),
24.45 – the molar volume of any gas or vapour under normal conditions.

RESULTS AND DISCUSSION

According to the calculation according to equations (1 – 6) with the use of AHP, a matrix of alternatives was built for each of the criteria, and the priorities of the criteria in relation to the aim are presented in Table 1. It can be seen that the results of the calculations are consistent, as they are within the required limits of $CR < 0.1 ... 0.2$.

The results of determining the global priority metal from the accepted alternatives are given in Table 2.

Tab. 1 Criterion priority matrix with respect to the objective and alternatives to each criterion.

Criteria	Criterion priority	Alternatives		
		A_1	A_2	A_3
Cr_1	0.042	0.04	0.31	0.49
Cr_2	0.068	0.07	0.23	0.70
Cr_3	0.137	0.08	0.23	0.69
Cr_4	0.152	0.10	0.41	0.49
Cr_5	0.600	0.11	0.00	0.51
Consistency of calculations				
CR	0.152	to Cr_1 –0.03; to Cr_2 –0.128; to Cr_3 –0.164; to Cr_4 –0.150; to Cr_5 – 0.09;		

Tab. 2 Global priorities of accepted alternatives.

Alternatives	A_1	A_2	A_3
GlPr	0.101	0.123	0.542

Therefore, the highest priority was given to the alternative 3 – nanoparticles of zinc. To determine the formaldehyde emission, the samples together with the Temtop M2000 device were placed under a hermetic transparent glass cover with a volume of 12 litres, and the indicators were taken after 15 minutes (Figure 2). After each sample, the room and glass cover were ventilated, and the device was calibrated.

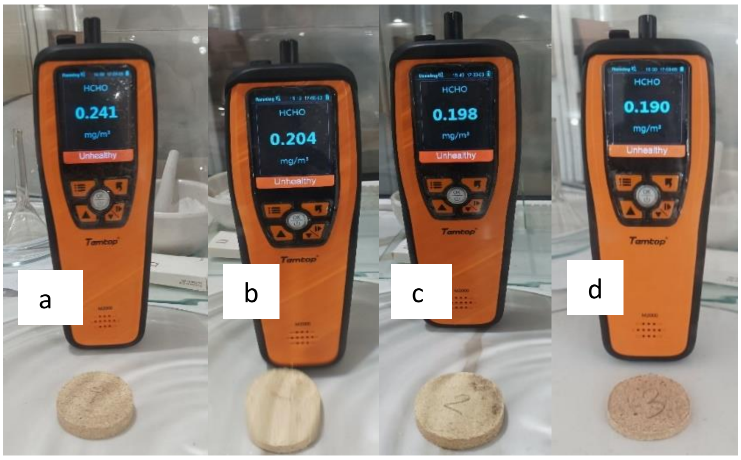

Fig. 2 Indicators of the level of free formaldehyde in the control sample (a) and samples of the modified glue based on UF with the addition of 8% Al^0 nanoparticles in three replicates (b, c, d) after 15 min of measurement using the Temtop M2000 device.

The background value of formaldehyde when measuring the state of native chips was 0.034 ppm. The obtained particleboard samples with the addition of unmodified glue had an average of 0.188 ppm, which was 5.5 times higher than the background value.

The results of measurements after 48 hours of exposure are presented in Table 3. Samples modified with 8% Zn^0 showed the best result. On the third day after pressing, the formaldehyde level was 48% lower than the control and was 0.098 ppm. The addition of 2% Zn^0 reduces free formaldehyde emissions by 27% to 0.140 ppm.

A reduction in formaldehyde emission was also observed when Al^0 nanoparticles were used. The best result was obtained with the addition of 2% Al^0 – the emission decreased by 29% compared to the control sample. At the same time, the addition of the metallic form of Al^0 at a concentration of 8% reduced the level of free formaldehyde emission by only 18%.

Tab. 3 Average indicators of the level of free formaldehyde in the study samples on the third day after pressing, ppm.

Metal	Concentration, %	Var. 1	Var. 2	Var. 3	Mean ppm
Control	0	0.193	0.188	0.184	0.188
Al^0	2	0.134	0.137	0.129	0.133
	8	0.170	0.147	0.146	0.154
Zn^0	2	0.145	0.141	0.133	0.140
	8	0.096	0.101	0.098	0.098
Fe^0	2	0.446	0.429	0.432	0.436
	8	0.186	0.216	0.227	0.210

The results of measuring the formaldehyde level of the samples modified with Zn^0 nanoparticles on the seventeenth day showed that they were almost no different from the measurements on the third day.

In the samples using the adhesive modified with aluminium nanoparticles on the seventeenth day, a slight increase in the level of formaldehyde emission was observed compared to the previous measurement on the third day. Samples modified with 2% Al^0 showed a 15% increase in free formaldehyde, and samples modified with 8% Al^0 showed a 5% decrease in formaldehyde.

Tab. 4 Average indicators of the level of free formaldehyde in the study samples on the seventeenth day after pressing, ppm.

Metal	Concentration, %	Var. 1	Var. 2	Var. 3	Mean ppm
Control	0	0.182	0.186	0.189	0.186
Al^0	2	0.162	0.157	0.155	0.158
	8	0.178	0.177	0.177	0.177
Zn^0	2	0.124	0.125	0.124	0.124
	8	0.113	0.112	0.112	0.112
Fe^0	2	0.356	0.344	0.351	0.350
	8	0.120	0.121	0.118	0.120

The results of measuring the level of formaldehyde in samples modified with iron nanoparticles showed a significant decrease compared to the measurements obtained on the third day after pressing, by an average of 30%.

The conducted experimental studies confirmed the priority calculations for the use of nanoparticles of various metals, proving the superiority of Zn^0. In addition, zinc in any form can act as a catalyst for the urea polycondensation reaction with the formation of H-bonds between C=O and N–H groups, accelerating crystallisation while at the same time forming linear oligomers of different lengths (Du *et al*., 2019), which may affect further formaldehyde degradation. Considering the negative effect of zinc on the physical and mechanical properties of wood composite materials, namely the promotion of brittleness of the final product (Gul *et al*., 2021), further research is necessary, for example, on its use in mixtures with other metals. Thus, the use of nanoparticles of Al^0, despite the slight reduction in formaldehyde emissions (Kumar *et al*., 2013a), improves the physical and mechanical properties of wood composites.

The use of iron nanoparticles during the repeated measurement showed a positive result, which may indicate the safe use of such wood composites indoors. However, the increased toxicity of production can negatively affect the health of workers involved in their production and can also indicate the danger of their production process for the environment.

CONCLUSION

Nanoparticles in metallic form were proposed to reduce formaldehyde emissions in particleboard manufacturing using an adhesive based on UF resin. The results obtained from the research experiment confirmed the effectiveness of using Zn^0 and Al^0 nanoparticles as modifiers of UF resin to reduce formaldehyde emissions. The application of 8% zinc showed the best result – a reduction in formaldehyde emission by an average of 44%. In order to ensure the necessary physical and mechanical properties of particleboards, further studies of the influence of the mixture of the metallic form of Zn^0/Al^0 nanoparticles in different concentrations on the release of formaldehyde from wood composite materials will continue.

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