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# INFLUENCE OF PARTICULATE MIXTURE CONTAINING STABILISED WOOD ON LONG-TERM BEHAVIOUR OF WOOD-CEMENT COMPOSITES. CASE STUDY

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## ABSTRACT

This paper presents research on the changes in the properties of cement-bonded particleboard modified with particulate mixture (PM). PM replaced 4% of the binder (cement) and 4% of the filler (spruce chips). The cement-bonded particleboards were tested for physical (bulk density, swelling, linear expansion due to relative humidity changes) and mechanical properties (modulus of rupture and modulus of elasticity). Development of phase composition and microstructure by XRD and SEM were also analysed. The long-term behaviour of wood-cement composites was studied over a period of 2 years. The physical, mechanical properties and microstructure of the modified particleboards were compared with commercially produced cement-bonded particleboards from CIDEM Hranice, Inc. There is no intentional change in properties when using PM compared to the reference boards and the values reached the EN 634-2: 2007.

KEYWORDS: Wood-cement composite, stabilised wood, by-product, long-term behaviour, microstructure.

## INTRODUCTION

Cement-bonded particleboard (CBP) is composed mainly of 63% wood chips and 25% cement by volume, however by weight, cement accounts approximately 50%. The modification of the composition of wood-cement composites has been ongoing for several decades (de Lima et al., 2011; de Lima et al., 2020; Frybort et al., 2008; Fuwape et al., 2007; Gigar et al., 2023; Hou et al., 2022; Kumar and Kesavan, 2020; Loganayagan et al., 2021; Ranjan et al., 2024; Vu et al., 2019; Yel, 2022; Zhou and Kamdem, 2002). The potential use of contaminated wood from formwork in combination with a $CO_2$ cured magnesium cement-based matrix was investigated by Wang et al. (Wang et al., 2016). (Hossain et al., 2018) recycled wood formwork (waste wood) into CBP using magnesium cement with a lower production temperature <750°C while achieving physical and mechanical properties that meet the requirements of relevant standards. Based on Karade (Karade, 2010), lignocellulosic waste appears to be one of the potentially suitable alternative raw materials. However, these wastes need to be pretreated or chemical accelerators need to be used. The use of rice husk has been shown to be a potentially suitable alternative raw material for the production of CBP (Sarkar et al., 2012), where an increase in strength and resistance to sulphate has been demonstrated (Hu et al., 2020). The use of babaçu (Orbignya sp.) coconut shells had a positive effect on achieving good physical and mechanical properties (Almeida et al., 2002). The use of pulverized fly ash and incinerated sewage sludge ash for the production of CBP resulted (under air curing) in increased flexural strength and dimensional stability (Chen et al., 2020). These improvements are attributed to the formation of an amorphous M-S-H gel within the pozzolanic reaction. Stabilisation of waste wood by $CO_2$ is also a very interesting possibility. The cement reaction is accelerated by the $CO_2$ directly reacting with the different phases of the cement to form C-S-H gels and $CaCO_3$. This process accelerates hardening in the microstructure, improving mechanical properties and producing stable carbonates for long-term stability. Better compatibility between cement and wood can be achieved by $CO_2$ stabilisation. Furthermore, mechanical properties, bulk stability and segregation of contaminated material are improved (Wang et al., 2017). The addition of nano-slag proved to be a potentially raw material for modification, which had a beneficial effect on the physical, mechanical and microstructural properties of particleboard. However, higher density did not necessarily translate into higher flexural strength values (Ali et al., 2020). The stabilisation of wood in matrix composition is described in detail in (Melichar et al., 2024).

Based on the above, it is evident that a wide range of different alternatives in the production of wood-cement composites have been investigated and are still the subject of research. However, virtually none of the authors have addressed the recovery of the particulate mixture from CBP production in terms of the long-term behaviour of these composites. The effect of moisture on the behaviour of CBPs is analysed by Melichar et al. (Melichar et al., 2021). However, this is a study of the properties of modified boards in contact with water in the liquid state.

## MATERIALS AND METHODS

### Production process

The production of test samples of wood-cement composites was carried out on the continuous production line of the CIDEM Hranice, Inc. Spruce chips, blended cement 42.5 R, water, aluminum sulphate, water glass and particulate mixture (PM) were used for production. The boards were made in two formulations – reference and modified. A total amount of 8% PM replaced cement and spruce chips in an amount of 4% each. The composition of the recipes (Tab. 1) was designed taking into account the findings of previous research (Melichar and Bydzovsky, 2019; Melichar et al., 2019; Melichar et al., 2021).

*Tab. 1: Composition of the cement-bonded particleboards (mass %).*

| Component | Control mixture – RE [%] | Modified mixture – PA [%] |
|---|---|---|
| Cement CEM II/B-S 42,5 R | 50 | 46 |
| Particulate mixture (PM) | 0 | 8 |
| Spruce chips | 18 | 14 |
| $Al_2(SO_4)_3.18H_2O$ $Na_2SiO_3$ | 2 | 2 |
| Water | 30 | 30 |

## Particulate mixture

The PM was collected directly in the cement-bonded particleboard (CBP) production. These are already mixed ingredients from the production of CBP – cement, spruce chips, water and hydration additives, which are collected and then transported to the outdoor landfill.

The PM properties were analysed in detail, and the phase composition was also examined within the time-lapse sampling to detect any fluctuations in terms of individual mineral content. Furthermore, the microstructure was analysed by both optical and scanning electron microscopy including chemical composition and particle size distribution.

## Methods of testing

The test pieces 290 × 50 × 12 mm for determination of the flexural strength and modulus of elasticity and 50 × 50 × 12 mm for determination of the density and tensile strength perpendicular to the plane of the board were selected. The pieces were placed in a climate chamber with an ambient temperature of (20 ± 2)°C and a relative humidity of (75 ± 3)%. Each measurement was always taken after climatization at (65 ± 5)% relative humidity and a temperature of (20 ± 2)°C. The properties of the wood-cement composites were evaluated over a period of 2 years, first after 28 days and later after half a year, i.e. at (183, 365, 548 and 730) days. One of the two sets tested was always subjected to cyclic freezing and thawing according to EN 1328 (1996). Compared to the standard recommended procedure, a slight modification was made by increasing the minimum number of cycles required from 50 to 100. The second set was used as a comparison. The properties were determined according to EN 323 (1993), EN 310 (1993), EN 319 (1993), including analysis of mineralogical composition (XRD) and microstructure (SEM). The set of test pieces consisted of 6 test pieces for the determination of one specific parameter in one specific environment and age. A total of 120 test pieces (EN 310 and EN 1328) and another 120 test pieces (EN 323 and EN 319) were prepared.

Separately, a hygroscopicity test was performed where the effect of cyclic freezing and thawing. The test was based on EN 318 ("EN 318," 2002), while the procedures were modified to describe the hygroscopic behaviour of the boards in more detail. Dimensional and mass changes were monitored on specimens with dimension of 350 × 150 × 12 mm in intervals of 10% ambient relative humidity changes (i.e. 0, 10, 20, 30, ..., 90, 96)%. The dimensions correlated to the larger format boards actually produced. The sorption isotherms for each of the monitored parameters are obtained. The set of test specimens consisted of 6 pieces, i.e. a total of 12 pieces were made.

The tempered test specimens were weighed with accuracy of 0.01 g and the dimensions were measured with a digital calliper with a resolution of 0.01 mm. Bending strength and modulus of elasticity were tested on a device with a 20 kN load cell and accuracy of ± 0.5%. Phase composition was evaluated by using X-ray diffractometer Empyrean Panalytical (CuKα radiation) with angular resolution 0.026°. Microstructure was analysed by a scanning electron microscope TESCAN MIRA3 XMU with resolution 1.2 to 1.5 nm at 30 kV in SE mode and 2 nm at 30 kV in BSE mode.

## RESULTS AND DISCUSSION

### Particulate mixture's analysis

The sieve analysis indicates a different grain size compared to cement and spruce chips. However, a similarity in the grain size curve of spruce chips and PM can be observed (Fig. 1). PM contains a slightly higher percentage of smaller particles, which is determined by the presence of cement or cement matrix.

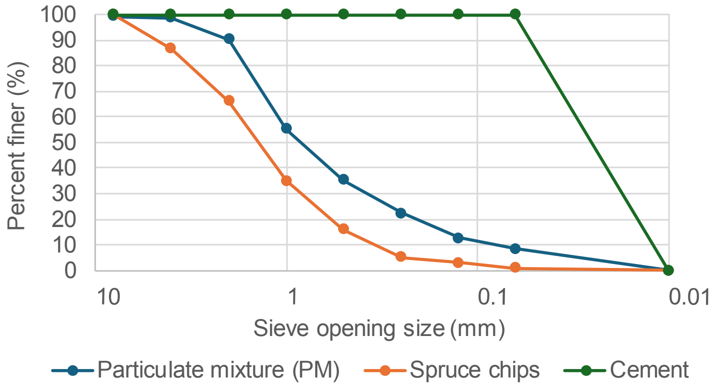
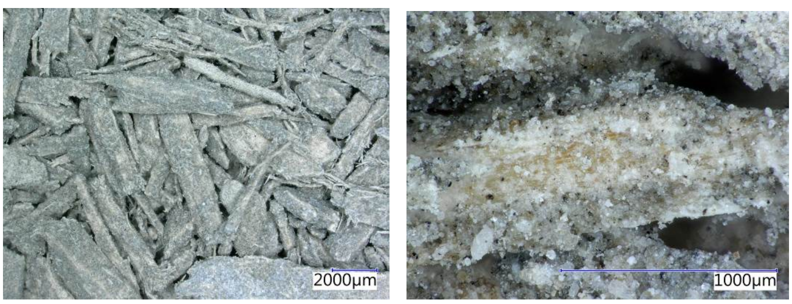



*Fig. 1: Distribution and particle size of used cement, spruce chips and PM.*

Analysis by optical microscopy shows that the PM is composed mainly of spruce chips with cement matrix adhering to their surface (Fig. 2). At higher magnification, the cement matrix coating of the chips appears to be imperfect (Fig. 2b). However, microstructural analysis by SEM in combination with EDX shows a relatively closed structure of the spruce chips surface just with respect to the cement matrix (Figs. 3, 4).



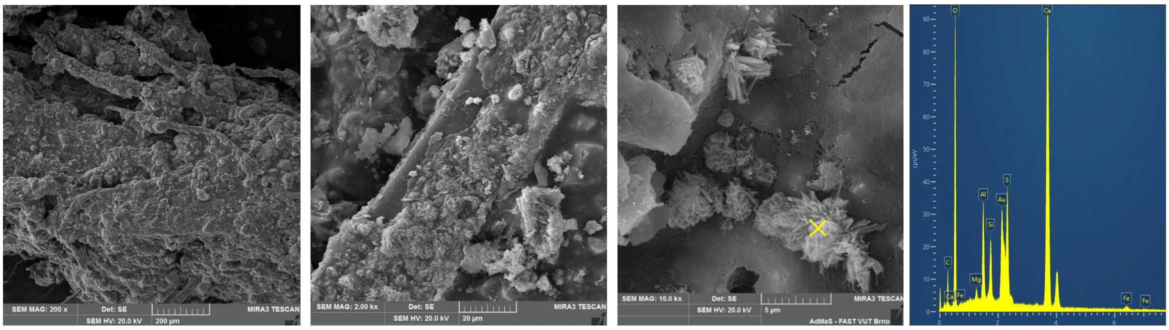

a) b)

*Fig. 2: Detail of PM structure; optical microscope images; mag. (a) 20× (b) 200×.*

In some cases, sites where the wood surface was exposed, i.e. without the presence of cement matrix, were identified on the surface of spruce chips contained in PM (Fig. 3b). However, these were rather isolated occurrences, indicating a rather closed structure of the PM. The fact that the PM chips are relatively well enveloped and thus mineralized by the cement matrix is also evidenced by the results of water absorption and bulk density (Tab. 2).

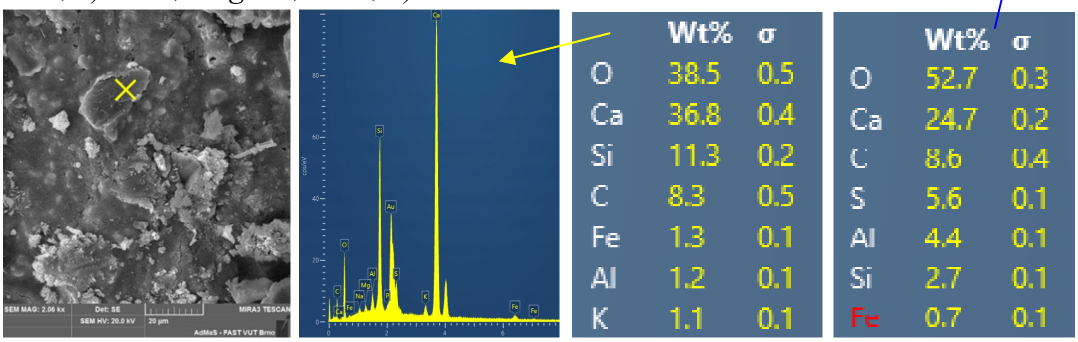
*Tab. 2: Properties of PM and primary spruce chips.*

| **Material** | **Bulk density (kg/m$^3$)** | **Loose bulk density (kg/m$^3$)** | **Water absorption (%)** |
|---|---|---|---|
| Spruce chips (Melichar et al., 2022) | 420 | 90 | 196.3 |
| Particulate mixture (PM) | 1230 | 560 | 28.4 |



a) b) c) d)

*Fig. 3: Microstructure and elemental analysis of PM – detail of surface; mag. a) 200×; b) 2,000×; c) SEM, mag. 10,000×; d) EDAX.*




a) b) c) d)

*Fig. 4: PM – detail of surface; a) SEM, mag. 2,000×; b), c) and d) EDAX.*

From the mineralogical analysis (Fig. 5) and elemental analysis (Figs. 3d, 4b) it is evident that the surface of the spruce chips contained in the PM is covered by a continuous layer of cement matrix except for isolated locations.

The matrix contains the standard hydration products (CSH gels, portlandite), as well as calcite (a consequence of the carbonation of portlandite), sulphate phases (ettringite) and also residues of non-hydrated clinker minerals (larnite – β-$C_2S$). It is therefore evident that the PM is relatively well mineralised for the production of wood-cement composites and is characterised by a high degree of compatibility with the mixture for the production of CBP.

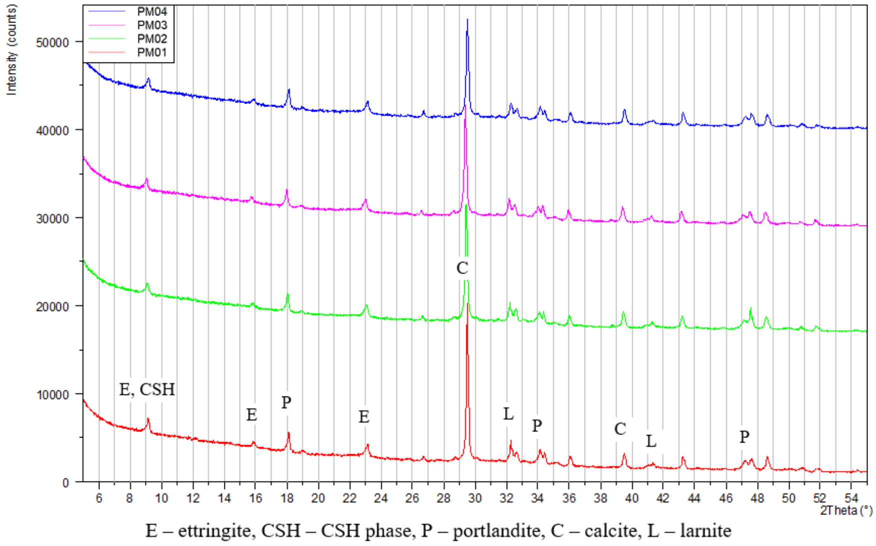
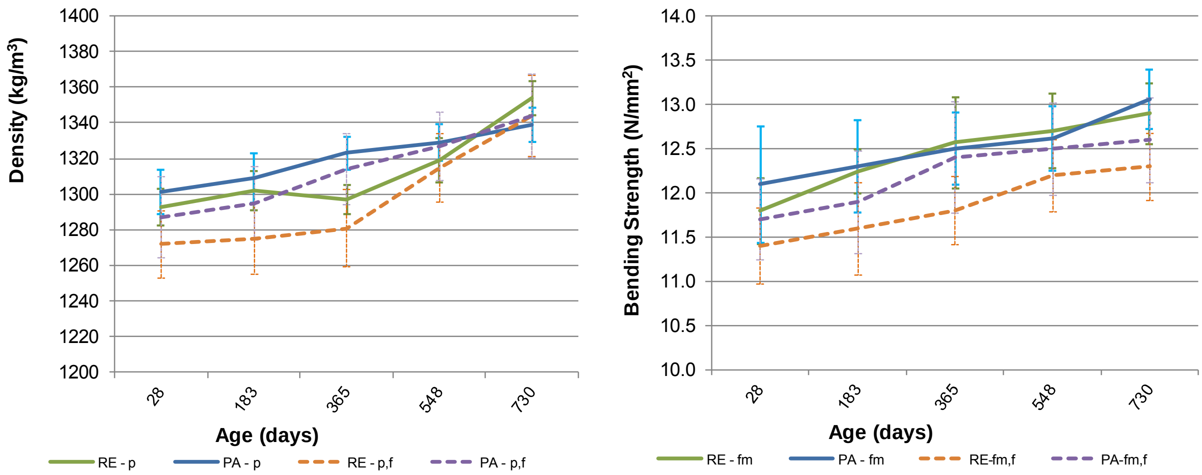



*Fig. 5: XRD diagram – PM from cement-bonded particleboard production (4 samples collected after 2 months each due to variability analysis).*

## Properties of the modified CBP

The modification of the CBP composition by the PM does not have a very significant effect on density (Fig. 6). Only minor differences in material composition of the boards and their exposure to cyclic freezing/thawing.



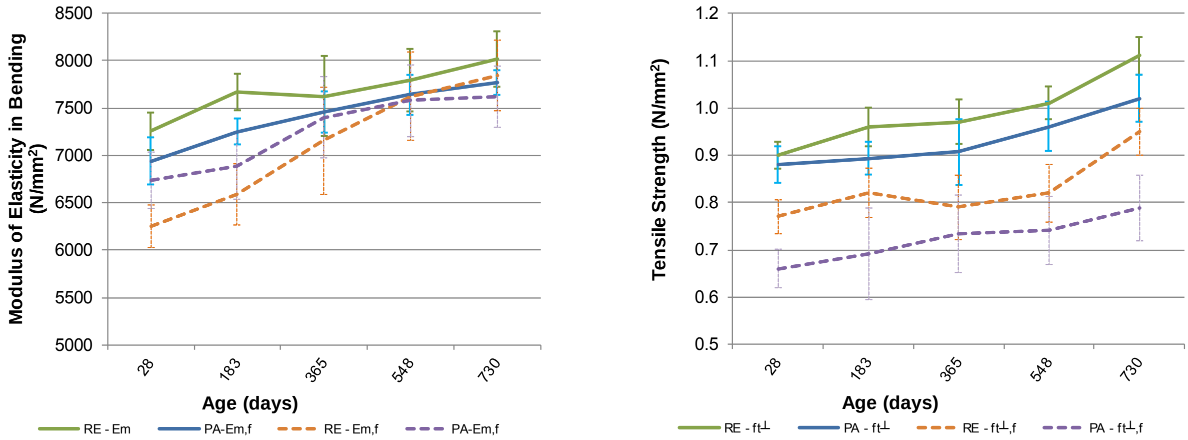

(a) (b)

*Fig. 6: a) density – p, change of density – Δp, b) bending strength – $f_m$, change of strength – $Δf_m$*

The density increases with age for both types of boards – RE and PA. The cyclical effect of freezing and water is reflected in terms of variability or deviation of the determined values from the average. More noticeable dispersions (error bars, Fig. 6a) can be observed in the case of boards exposed to frost. Variability decreases approximately with increasing age. However, these differences are not significant. Slightly higher values are obtained for PA boards. The effect of freezing is also less pronounced in the case of PA boards. The modification of the boards with a particle mixture containing stabilised chips has a slightly favourable effect on the density values. The density differences (due to board composition and frost cycling) decrease with increasing age.

The bending strength $f_m$ is a key parameter of the performance of the CBP. The bending strength results (Fig. 6b) already indicate a slightly more significant difference between the analysed board types. The effect of cyclic freezing and thawing is more pronounced, with PA boards being better evaluated. This finding is in contrast to the results of (Zhou and Kamdem, 2002), where a noticeable decrease in flexural strength also occurs with respect to the lower cement dosage. Reason is that PA boards contain significantly reduced amount of spruce chips by a much higher quality alternative raw material (already mineralized or stabilised wood particles). Alternative PM component, which has a much lower water absorption and a higher bulk density (Tab. 2), has had a positive effect. The strength increases slightly over time, both for RE and PA boards. After 730 days, the PA boards have a slightly higher strength (6.8%). The RE boards at 730 days show a decrease (8.5%) due to exposure to cyclic freezing and thawing, when the PA boards show a decrease of 3.8 %. In terms of the $f_m$ variability, the variance of the individual strength values increases slightly due to the modification of the composition by the alternative PM component. The PA boards show the relative error values in the range 2.6–5.5%. The strong dependence of the strength values variability is also confirmed by the results of (Fuwape et al., 2007) –an average flexural strength of 8.1 $N/mm^2$ with a relative error of 25.2% was determined (boards containing waste paper). Both RE and PA boards are more resistant to the effects of cyclic freezing and thawing over time. Similar to density, the effect of freezing on the variability of flexural strength was evident. Boards subjected to alternating freezing and thawing have slightly higher absolute errors.

RE boards stored in the laboratory environment show higher values of modulus of elasticity in bending (Fig. 7a). In case of cyclic frost exposition, PA boards can be better evaluated. $E_m$ values increase with increasing age. Between age 548 and 730 days RE and PA boards show a very low decrease in modulus of elasticity due to frost exposure. After 28 days of ageing the modification of composition by the PM is more pronounced than after 2 years of ageing (values of 28 and 730). The variability of the modulus of elasticity values is slightly lower compared to the variability of the bending strength.




(a) (b)

*Fig. 7: (a) modulus of elasticity in bending – $E_m$, change of modulus – $\Delta E_m$, (b) transverse tensile strength perpendicular to the plane of the board – $f_t\perp$, change of strength - $\Delta f_t\perp$.*

The tensile strength perpendicular to the plane of the board $f_{t\perp}$ (hereafter also "tensile strength"; Fig. 7b) was also evaluated. A decrease in tensile strength of up to 17% was observed due to the modification of the board composition. The variability of the tensile strength values is the highest of the parameters analysed (up to 13.9%). This is closely related to the fact that the largest volume changes are due to the orientation of the spruce chips in the boards in the thickness direction (i.e. perpendicular to the plane of the board). This is because the pressing of the boards takes place perpendicular to the plane of the board during the production process. Therefore, stresses are introduced into the boards, which are released when the boards subsequently come into contact with water. Then the introduced stresses then have a negative effect on the cohesion of the board structure in the thickness direction. This phenomenon is supported by the findings of other authors (Fuwape et al., 2007; Melichar et al., 2021) dealing with similar issues. Due to volume changes of the spruce chips, the adverse effect of induced stresses in the boards will then be compounded by possible disturbances in the cohesion of the matrix. One of such factors is, among others, the modification of the matrix composition (substitution of cement by PM). The achieved values of the modified PA boards can be evaluated very positively, where e.g. in (Okino et al., 2004) values in the range of 0.19 to 0.40 N/mm$^2$ are reported for CBP.

The mass (Fig. 8a) was the most affected by changes in ambient humidity. The effect of composition is not negligible in terms of hygroscopicity.

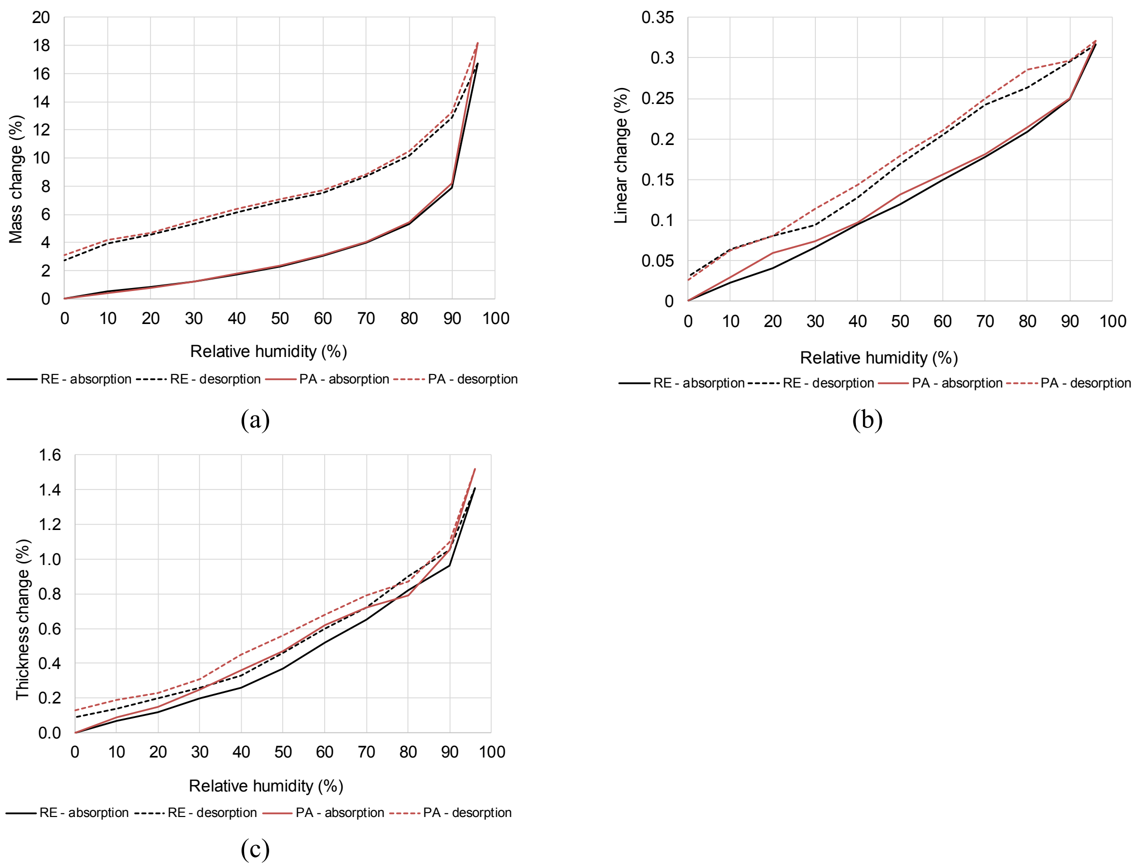



*Fig. 8: Mass (a), linear (b) and thickness (c) change due to hygroscopicity of CBP.*

Fluctuating relative humidity (RH) was more pronounced in the case of the modified PA boards (18.2 %). The reference RE boards showed a maximum mass change of 16.7%. The graph (Fig. 8a) also shows that the highest mass increase occurred at RH in range of 90%–96%. The sorption isotherms are similar, but some slight differences can be observed. Irreversible change – hysteresis is characterised by a certain amount of water from air moisture bound into the CBP structure. Hysteresis was determined 2.3% (RE) and 3.1% (PA).

The linear (dimensional) changes of the boards were also analysed (Fig. 8b). The RE boards can be better evaluated with respect to the maximum linear change. However, when comparing the trends of the absorption and desorption isotherms of RE and PA, only slight differences can be observed. The maximum linear changes are 0.30% (RE) and 0.32% (PA) respectively. The trend of the sorption isotherms is relatively smooth. The hysteresis was determined to be 0.03 %.

The dimensional changes in the direction perpendicular to the plane of the board are noticeably more noticeable (Fig. 8c) than the linear changes. This is connected to the spatial arrangement – the orientation of the spruce chips and the direction of compaction during board production. The characterization of the spruce chips distribution in the board is presented in (Melichar et al., 2021). The authors evaluate the structure of the boards based on CT images. The orientation of the radial and tangential direction of the chips is dominantly perpendicular to the plane of the board. This may justify the largest dimensional changes in this direction. The more significant dimensional changes of wood in the radial and tangential direction are also confirmed by (Fu et al., 2019; Melichar et al., 2021). The changes in the direction perpendicular to the plane of the board are also related to the release of residual pressures and the action of water-saturated chips by pressure on the adjacent cement matrix, which is supported by (Rowell, 2004). The greatest change can be observed between relative humidity of 90% and 96%. The maximum thickness change determined was 1.5% (PA boards). In contrast, standard RE boards are characterized by a lower change (1.4%). The irreversible change was determined to be 0.13% for PA boards and 0.09% for RE boards.

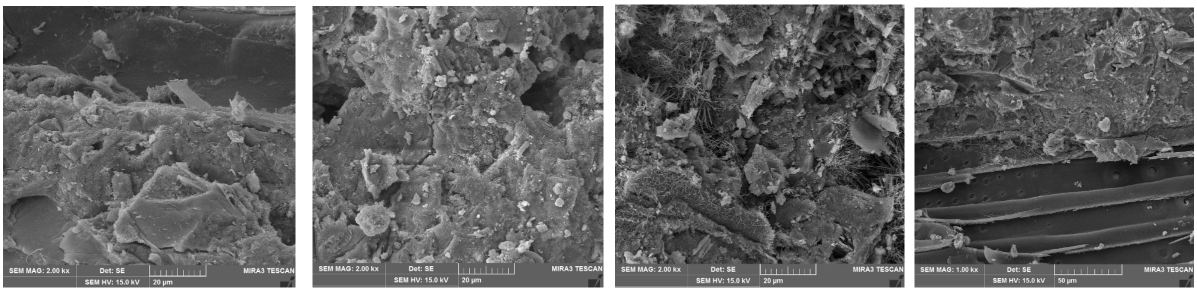
Due to increasing age and different material composition, the phase composition of the RE and PA boards is constant at each age. PM was therefore confirmed as highly compatible for the purpose of modifying the CBP composition.

Similarly to the XRD analysis of the PM raw material, virtually identical phases or gel structures were identified in the PA boards (Tab. 3). The only difference was in the evolution of the diffraction lines. An increase in the intensity of the $CaCO_3$ peaks could be observed with increasing age. Conversely, a decrease in the intensity of the $\beta$-$C_2S$ diffraction lines is evident. Thus the compact structure of the cement matrix is still gradually formed with increasing age.

*Tab. 3: Mineralogical composition of analysed CBP.*

| Boards | Identified minerals |
|---|---|
| RE | CSH phase, ettringite, portlandite, calcite, larnite |
| PA | CSH phase, ettringite, portlandite, calcite, larnite |

The microstructure of the CBP can be assessed as compact, without the presence of disturbances between the individual phases of the cement matrix (Fig. 9).



a) b) c) d)

*Fig. 9: Microstructure of RE wood-cement composites after 730 days of ageing and exposed to 100 frost/defrost cycles according to EN 1328; a) detail of matrix-chip ITZ; b) detail of matrix structure; PA – c) detail of matrix structure; d) detail of matrix-chip ITZ.*

The alternative component PM interacts very well with the hydration products and is highly compatible with the structure of the wood-cement composite. From the point of view of the identification of β-$C_2S$ in the PM and also in the PA boards, where the intensity of the diffraction lines of this mineral decreased with age. Thus PM partially (but to a very limited extent) participates in the formation of the cement matrix structure and slightly participates in the cohesion improvement of the composite.

With increasing age there is a gradual growth of identified crystalline phases of the matrix (especially $CaCO_3$). A certain percentage of these minerals growing into the cellular structure of spruce chips. The interfacial zone (ITZ) of the matrix and spruce chips shows no failures or defects. Hydration products penetrate into the cellular structure of the chips (Fig. 9d). The penetration of cement matrix ions into the wood structure was confirmed by Capari et al. (2018). The strengthening of the bond between the matrix and the wood chips is, among other things, partially ensured by the exchange of ions between the wood and the cement matrix (Frybort et al., 2008). Microstructure analysis confirmed a high degree of PM compatibility in the wood-cement composite structure. It is virtually impossible to distinguish individual PM particles in the wood-cement composite (PA boards).

## CONCLUSIONS

Modification of the CBP composition with a particulate mixture (by-product of CBP production) shows a high potential in terms of industrial production of CBP. The substitution of 4% cement within the production of CIDEM Hranice, Inc. can save about 3 thousand tons of cement annually. This aspect will be reflected both from the environmental and economic point of view.

The tested boards meet the requirements of EN 634-2 (2007). With increasing age, the difference in mechanical properties between the reference RE and the modified PA boards gradually decreases. PM is highly compatible with respect to a longer time horizon and can be partially involved in the cement matrix structure. The hygroscopic behaviour of the RE and PA boards is very similar. PA boards being slightly more subject to dimensional and mass changes due to fluctuating relative humidity. The phase composition of PM and the analysed CBP confirms the high degree of compatibility and stabilisation. Thus synergistic interaction of PM with the components of the mixture for the production of CBP is assumed. Slight changes in the diffraction lines of $CaCO_3$, $Ca(OH)_2$ and β-$C_2S$ were identified with increasing age. The intensity of the calcite lines showed an increase, whereas the intensity of the diffraction lines of portlandite and larnite decreased. SEM and EDX analysis confirmed the findings of XRD analysis. The microstructure of the PA boards is compact and practically indistinguishable from that of the RE boards. The contact zone of the cement matrix and spruce chips does not show disturbances. The hydration products of the matrix penetrate the cellular structure of the spruce chips.

Currently, data from long-term exposure of the boards in a real environment and also from exposure in significantly accelerated aggressive laboratory environments –$CO_2$, $SO_2$, shock changes in temperature and humidity according to EN 321 (2001) and presented by (Melichar et al., 2022) are being analysed. This follow-up research will allow future refinement and extension of the existing results and findings. Superabsorbent polymers in combination with fly ash for the modification of the wood-cement composites´ matrix will be also studied in scope of the follow-up research (Melichar et al., 2023). Attention will also be paid to increasing the activity of the alternative component PM by mechanical ways using the findings presented in (Ravaszová and Dvorák, 2021; Ravaszová et al., 2022).

## ACKNOWLEDGMENTS

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