Melichar *et al. Journal of Wood Science* (2022) 68:3
https://doi.org/10.1186/s10086-021-02004-3

Journal of Wood Science

ORIGINAL ARTICLE

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Structural, physical and mechanical changes of cement-bonded particleboards during sudden fluctuations in temperature and moisture

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Abstract

The paper presents research focused on behaviour of cement-bonded particleboards with modified composition during sudden changes of temperature and humidity. Four types of boards were made—one control and three modified ones. Finely ground limestone was used as a modifying component in binder. Secondary wood particles made from crushing cuttings of cement-bonded particleboards were used as chips substituent. Two sets of test specimens (1 set = 6 test specimens) were manufactured. The first set was stored in laboratory conditions. The second set was subjected to 10 cycles of sudden changes of temperature (− 20 °C to + 70 °C) and humidity in accordance with EN 321 (further in the paper referred to as "wet–frost–dry cycle".) After each cycle, dimensions and mass of the test specimens as well as ultrasonic pulse velocity were determined. A detailed analysis of structural changes in boards during cycling was carried out by an optical microscope. After 10 wet–frost–dry cycles were completed, bending strength and modulus of elasticity in bending were determined. The analysis of test results implies a very good relation between change of ultrasonic pulse velocity and width of cracks in the area of interfacial zone between cement matrix and wood particles. This finding also corresponds with dimensional and volumetric changes of the boards. Dependence of bending strength and modulus of elasticity in bending on composition of boards is apparent. Positive influence of secondary spruce chips on dimensional changes of cement-bonded particleboards caused by sudden changes of temperature and humidity was proved. Finely ground limestone contributes to more resistant structure of boards which leads to improved bending properties. Adverse conditions had more considerable influence on bending strength (decrease by 21% to 26%) than on modulus of elasticity in bending (decrease by 12% to 19%).

Keywords: Cement-bonded particleboard, Finely ground limestone, Cuttings, Secondary spruce chips, Sudden change, Temperature, Water, Ultrasonic, Microstructure

Introduction

Resistance of cement-bonded particleboards to action of sudden changes of temperature and humidity is limited by material composition of the boards. From this point of view, properties of spruce chips are very important [1]. By adjusting properties of wood in cement-bonded composite materials it is possible to achieve better properties of the final composite material [2–8]. Composition of cement-bonded particleboards can be modified by both matrix and filler [9–15]. Possibility of using waste from cement-bonded particleboards is presented only in [16]. However, the authors analysed the waste as an alternative component for concrete.

Cuttings without further use are produced during cement-bonded particleboards manufacture. Hence this

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by-product could be characterized as waste. The Company CIDEM Hranice, a.s. (Czech Republic) produces around 5000 t of these cuttings per year [17]. After appropriate treatment of properties of cuttings, this alternative component could be re-used for manufacture of cement-bonded particleboards [17, 18]. Use of these suitably treated cuttings can improve durability of cement-bonded particleboards. Especially, use of secondary chips separated from cuttings could have advantages. Secondary spruce chips had already once gone through the manufacturing process of cement-bonded particleboards. This process includes mineralization by Ca ions from cement, Na_2SiO_3 water glass and $Al_2(SO_4)_3.18H_2O$ with elevated temperature and pressure. Cell structure of secondary chips is therefore mostly filled by components of cement matrix (portlandite, C–S–H, ettringite, etc.). Presence of hydration products of cement matrix penetrated inside lumens and gel formation on surface of wood was proved [19]. Sodium silicate also penetrates to the lumina of the wood cells, which leads to the improved flexural modulus of rupture and decrease in moisture sorption of the treated wood. Sodium silicate located in wood structure blocks the pits connecting the two wood cells. This phenomenon significantly contributes to decrease in moisture migration into the wood structure [20]. Thus the structure of the secondary chips, particularly their surface, is denser with decreased absorption and less susceptible to volume changes caused by increased humidity. Furthermore, content of hemicellulose is lower than in case of primary chips. It is caused by dissolubility of hemicellulose in alkaline environment of cement matrix. The most of the hydroxyl groups in hemicellulose is responsible for approximately 37% of total water sorption in wood [21]. Compared to primarily used chips, properties of such secondary chips are considerably stabilized. Improvement of properties of raw and stabilized wood through various methods is also proved [22–28]. Therefore, the secondary spruce chips are expected to show better resistance to changes of temperature and humidity.

Properties and behaviour of cement-bonded particleboards in adverse conditions have been studied by many authors, for example [29–34]. Nevertheless, none of the authors has dealt with resistance of cement-bonded particleboards containing secondary spruce chips from cuttings of these boards. Most publications present research focused mainly on fluctuation of air humidity or its combination with increased temperature. It also means that boards were subjected to different course of cycles compared to procedure described in EN 321. The standard describes relatively aggressive method with sudden change of temperature $\Delta\Theta = 90$ °C and change of humidity from water bath to dry environment. Particleboards containing wood change their structure and parameters considerably during such fluctuations of environment.

In terms of characterizing behaviour of cement-bonded particleboards in adverse environment in detail, it is appropriate to support standard methods by non-destructive techniques. Ultrasonic pulse method is used mainly for evaluation of changes in inner structure of some building materials. This technique is mostly applied for tests of freeze–thaw resistance or for evaluation of quality—for determination of dynamic Young's modulus of elasticity or compressive strength of concrete. The procedure is codified in Czech and foreign technical standards for concrete and some building materials (natural stone, concrete, refractory materials, bricks, etc.). However, currently there is no technical standard describing application of ultrasonic pulse method for evaluation of properties of cement-bonded particleboards. Problems of using this method for examination of properties of wood based panels and particleboards are presented [35–49]. The surface density can be used to quality control of particleboards with different structures [47]. However, the authors did not analyse changes of properties of boards caused by wet–frost–dry cycles described in EN 321.

The objective of the research presented in this paper is the analysis of cement-bonded particleboards during sudden changes in temperature and humidity using conventional test techniques supported by non-destructive ultrasonic pulse method. Attention is paid to assessing changes of dimensions, mass, volume, mechanical properties and structure of cement-bonded particleboards with modified composition exposed to wet–frost–dry cycles (according to EN 321).

Materials

Cement-bonded particleboards were made on the production line of the company CIDEM Hranice, a.s. The process was in accordance with standard conditions of industrial manufacture. Manufacturing process used standard mixture for boards (only REF further in the text). Composition of this mixture is as follows: 50% of cement, 18% of spruce chips, 30% of water and 2% of hydration additions (sodium water grass and aluminium sulphate). This composition is commonly used by CIDEM Hranice, a.s. Considering results of current and previous research [17, 18, 50], three modified compositions were designed:

- REF—reference mixture, standard production of cement-bonded particleboard.
- LI10—10% of cement was replaced with finely ground limestone.
- SW07—7% of spruce chips was replaced with secondary chips

- L10/S07—10% of cement was substituted with limestone and 7% of primary chips with secondary chips (Table 1)

Compatibility of spruce wood and cement matrix is at high level. Spruce usually does not cause problems during cement hydration [51]. The ions exchange between cement and wood in presence of water can lead to a strong bond between the two materials due to the improved anchoring of the binder [19, 51]. Mechanical bonding and mechanical interlocking [52] such as chemical bonding [53] of wood and cement matrix occur. The interlocking is caused by growing crystals of cement matrix into cell structure of wood. Hydroxyl bridges and hydrogen bonding are significant during bonding cement matrix and wood when the matrix is bonded to the cellulose fibres [54]. Diffusion of cement molecules (based on Ca, Si, etc.) into the cell wall of wood chips was proved [55]. Mineral components of matrix penetrate deeply into the wooden structure when lumens, rays, etc., are partially filled by these hydration products [56]. However, the compatibility of the cement matrix and spruce chips is partially limited by some inhibitors contained in spruce wood. The main problem could be presence of hemicellulose which is dissoluble in alkaline environment, i.e. cement matrix (pH = 12.5). Hemicellulose diffuses into the cement paste [51, 57]. This leads to significantly reduced forming of the main products of cement hydration (portlandite and C–S–H phases and gels) [58, 59]. Unreacted cement grains are surrounded by acicular hydrates in presence of hemicellulose [60]. Especially, reaction of C_3A and C_3S is inhibited. Amount of unreacted cement clinker grows with increasing extractive content. This leads to strength reduction of the cement–wood composite [61]. The composition of cement is also important due to the compatibility between wood and cement with an effect on mechanical properties [62, 63]. Several procedures were used to eliminate aforementioned adverse effect of hemicellulose during cement-bonded particleboards:

- pre-treatment of spruce chips;
- use of chemical substances;
- treatment of the particleboards during manufacturing process.

Pre-treatment of wood is important to reduce the content of extractives in chips. Therefore, open air drying was applied to the used spruce chips when hemicellulose amount was reduced to range of 0.15–0.25%. Acceptable content of hemicellulose in wood particles for cement-bonded particleboards production is up to 0.5% [64, 65]. Sodium water glass and aluminium sulphate were used to regulate the hydration process of cement in spruce chips presence. These chemical substances accelerate forming of cement matrix, which partially eliminate negative effect of hemicellulose. Also these substances contribute to mineralization process of spruce chips (penetrate to cell structure of wood). Positive effect of curing cement-bonded particleboards (with spruce chips) by elevated temperature (40–60 °C) during production is obvious [66]. Therefore, the particleboards were thermally treated at elevated temperature, 45 °C.

Properties of the cement used are presented in Table 2. Figure 1 shows the curve of distribution and particle size of the cement.

Chemical composition and other significant properties of the limestone Kotouč Štramberk VMV15-F are shown in Table 3. Dominant component in crystalline phase is calcite, further aragonite, traces of magnesite and silica. The curve of distribution and particle size of finely ground limestone is presented in Fig. 1.

Table 1 Composition (%) of designed mixtures—reference and modified by limestone and secondary spruce chips

Component	REF	LI10	SW07	L10/S07
Cement	50	45	50	45
Limestone	–	5	–	5
Spruce chips—primary	18	18	16.74	16.74
Spruce chips—secondary	–	–	3.53	3.53
Water	30	30	30	30
Admixtures	2	2	2	2

Table 2 Properties of the cement CEM II 42,5 R

Chemical composition	CaO	SiO_2	Al_2O_3	Fe_2O_3	Alkali	MgO
[mass %]	58.9	19.2	5.5	3.2	< 1	1.7
Specific surface area		Specific weight		Compressive strength		
419 m^2/kg		3172 kg/m^3		59 MPa		
Initial setting time		Granulometric composition				
155–187 min		Shown in Fig. 1				

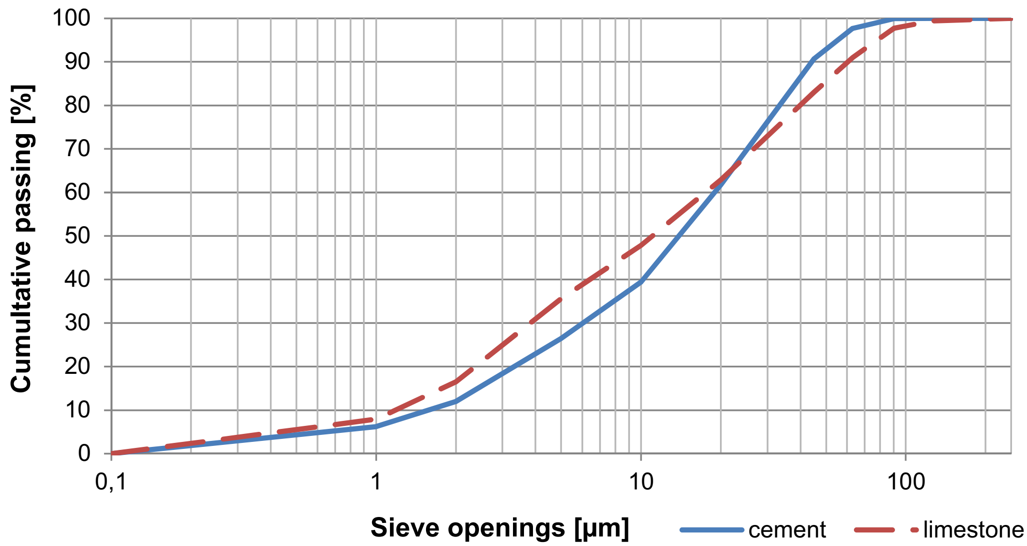

Fig. 1 Granulometric curve of cement CEM II/A-S 42,5 R and limestone VMV15-F

Table 3 Properties of the limestone VMV15-F

Chemical composition	CaO	CO_2	SiO_2	Al_2O_3	Fe_2O_3	Alkali	MgO
[mass %]	52.4	41.6	1.2	0.3	0.1	0.06	0.6
Specific surface area		Specific weight			Granulometric curve		
487 m^2/kg		2697 kg/m^3			see Fig. 1		
Mineralogic composition (XRD analysis)							
Calcite, aragonite, traces of magnesite and quartz							

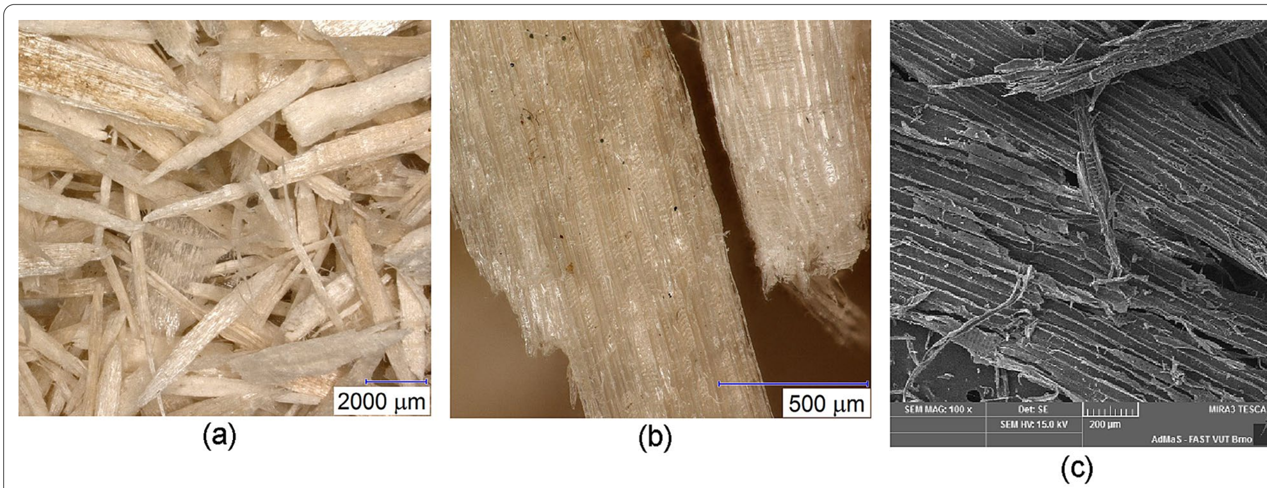

Fig. 2 Structure of primary spruce chips for manufacture of cement-bonded particleboards: **a** magnification 10× and **b** magnification 70× images from optical microscope Keyence VHX-950F; **c** image from electron microscope Tescan MIRA3 XMU (magnification 100×)

Table 4 Properties of the primary and secondary spruce chips

Particle size [mm]	Density [kg/m^3]	Bulk density [kg/m^3]	Water absorption [%]
0–8.0	420	90	196.3
0.5–1.0	1160	460	54.7
1.0–2.0	1190	480	49.3

Spruce chips contain dominant proportion of particles within the interval 0.25 mm to 4 mm, in total 79%. Particles below 0.25 mm are in the proportion of 7%. The rest are particles from 4 to 8 mm, i.e. 14%. Figure 2 shows structure of primary chips used for manufacture of cement-bonded particleboards. Properties of the primary spruce chips are shown in Table 4.

Cuttings (Fig. 3a) are produced during adjusting particleboards to the required shape and size. Production of cuttings has a rising trend which Fig. 3b shows. Particle size distribution was modified in a jaw crusher. Required groups of particles were separated. Separation was carried out on the basis of results presented in [17]. Particles selected for further research were those with the highest contents of TOC (total organic carbon, see [17]), i.e. content of wood. Particles with dimensions from 0.5 to 2 mm were used. In this way, secondary spruce chips (Fig. 4) were gained. Table 4 shows properties of secondary spruce chips. Specific surface area of the chips was not considered regarding to small amount of substitution.

Density of secondary spruce chips is higher than that of primary chips. Relatively small amount of the primary chips will be substituted—7% of the primary chips, which is approx. 3.5% of the whole particleboard mixture.

Fig. 3 Cuttings (by-product) from manufacture of cement-bonded particleboards: **a** sample taken in the manufacturing plant; **b** production of cuttings by CIDEM Hranice a.s. in the years 2010 to 2019

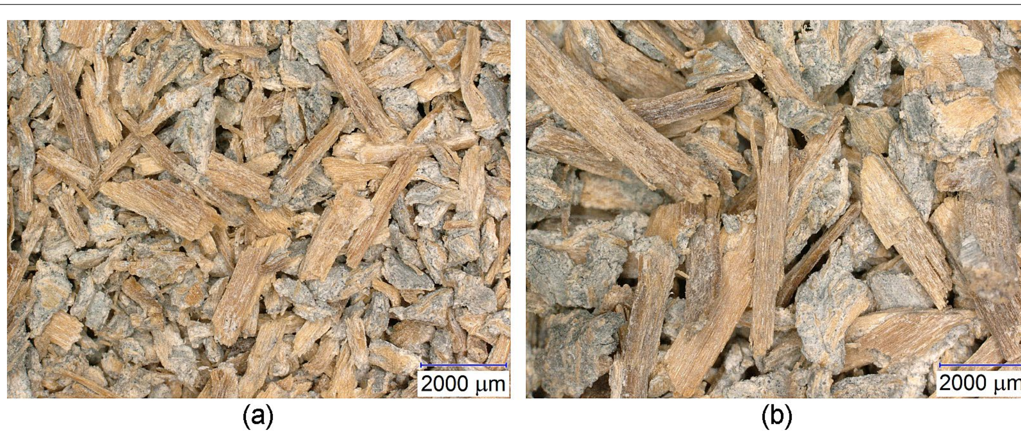

Fig. 4 Crushed cuttings from production of cement-bonded particleboards: separated secondary chips **a** 0.5 to 1.0 mm (magnification 10x) **b** 1.0 to 2.0 mm (magnification 10x)

Therefore, a significant effect on the final density of produced particleboards is not expected.

Comparison of parameters of primary and secondary chips implies that primary spruce chips have considerably higher water absorbing capacity and lower density compared to secondary chips. Lower water absorption of the secondary chips indicates better resistance to volume changes related to humidity and temperature fluctuations.

Results of the analyses stated in [17], including assessment of structure (Figs. 4 and 5), show that secondary chips contain residua of cement matrix. Products of cement matrix are bonded to the surface of the secondary chips, which Fig. 5 shows.

Grains from 1 to 2 mm contain even a certain number of small clusters of matrix and chips, which are presented in Fig. 4b. This can lead to enhanced chips resistance to humidity and temperature changes. On the other hand, negative impact could be proved during manufacturing process of cement-bonded particleboards. Secondary chips with residua of cement matrix are less squeezable during pressing compared to primary chips. Different flexibility of secondary chips could result in less compact or dense structure and even slight decreased strength of boards. Figure 6 shows microstructure of secondary chips. Cell structure of wood is partially filled with products of cement hydration.

Secondary chips dose added into raw material was 1:2—fraction 0.5 mm to 1.0 mm: 1.0 mm to 2.0 mm. To achieve required "moist" consistence (suitable for pressing), water dose was around 30% (by weight). However, amount of water was modified according to measured moisture of spruce chips dosed into the mix for production of boards. To achieve stabilization of wood chips and improve hydration processes, doses of additions based on sulphates and soluble glass were added into the mix.

Methods

Cement-bonded particleboards REF, LI10, SW07 and L10/S07 were made with standard manufacturing process in the plant CIDEM Hranice, a.s. in the Czech Republic. After 21 days of maturing, test specimens were prepared in accordance with EN 634-1 [67], EN 634-2 [68], EN 323 [69] and EN 310 [70]. Based on the analysis with ultrasonic pulse method, longer size of test specimen was selected—380 mm × 50 mm × 12 mm. For each individual set, 12 test specimens were made—6 control specimens for comparison and 6 specimens for cycling in accordance with EN 321 [71]. After 28 days of maturing in the production conditions in CIDEM Hranice, a.s., test specimens were transported to the Institute of Technology of Building Materials and Components, Faculty of Civil Engineering, Brno University of Technology. Test specimens matured further in the environment with 75% relative humidity and constant temperature (20 ± 2) °C. Under such conditions, the reference test specimens were matured for 11 months and specimens intended for wet–frost–dry for about 9 months. Test specimens intended for exposition in adverse conditions were then subjected to cycling in accordance with [71]. All specimens were then placed in the environment with relative humidity (65 ± 5)% and temperature (20 ± 2) °C. Testing of all physical–mechanical properties of all test specimens of cement-bonded particleboards were carried out at the age of 12 months. Sequence of individual steps and times, i.e. from manufacture of boards to final testing, is shown in Fig. 7.

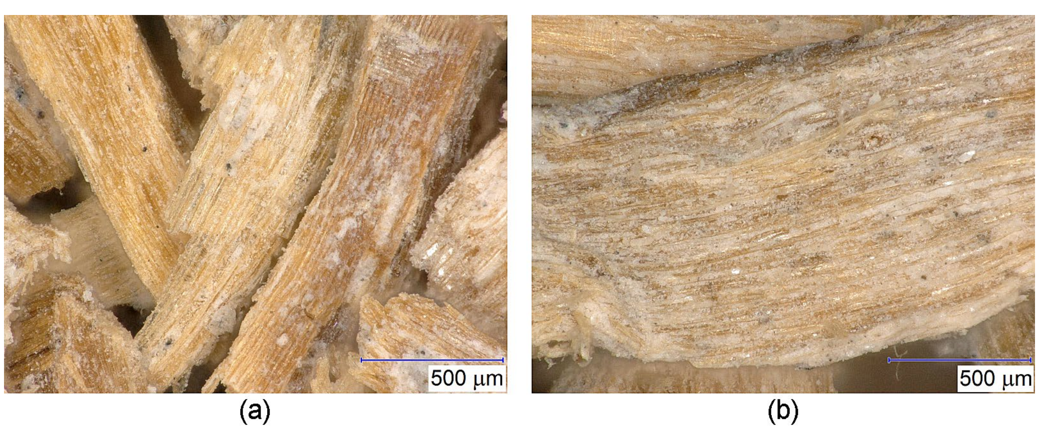

Fig. 5 Structure of separated chips from crushed cuttings from production of cement-bonded particleboards (images from optical microscope): **a** 0.5 to 1.0 mm (magnification 70x) **b** 1.0 to 2.0 mm (magnification 70x)

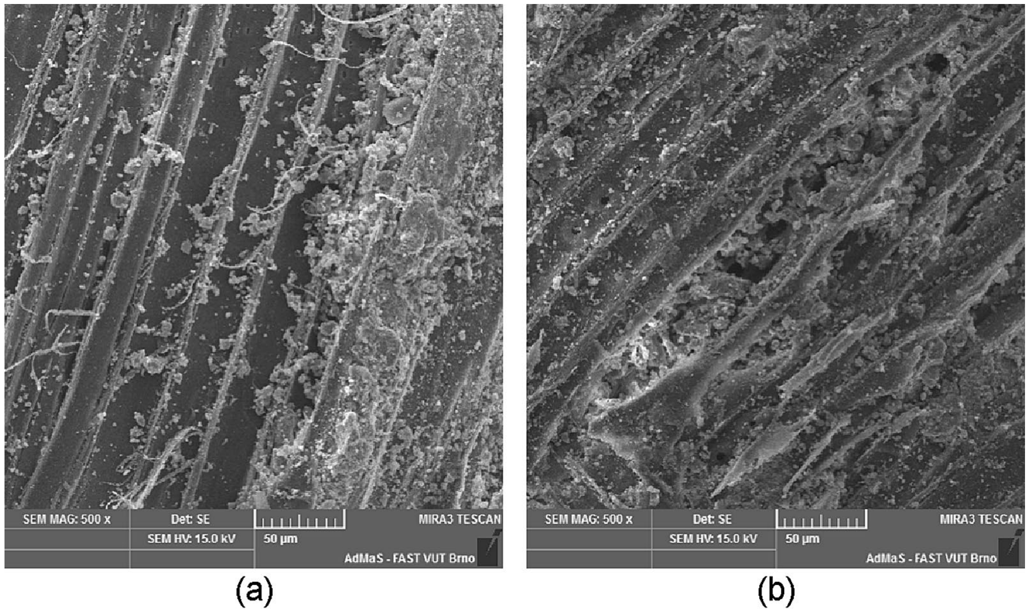

Fig. 6 Detail of chips separated from crushed cuttings of cement-bonded particleboards (images from electron microscope): **a** 0.5 mm to 1.0 mm; **b** 1.0 tot 2.0 mm

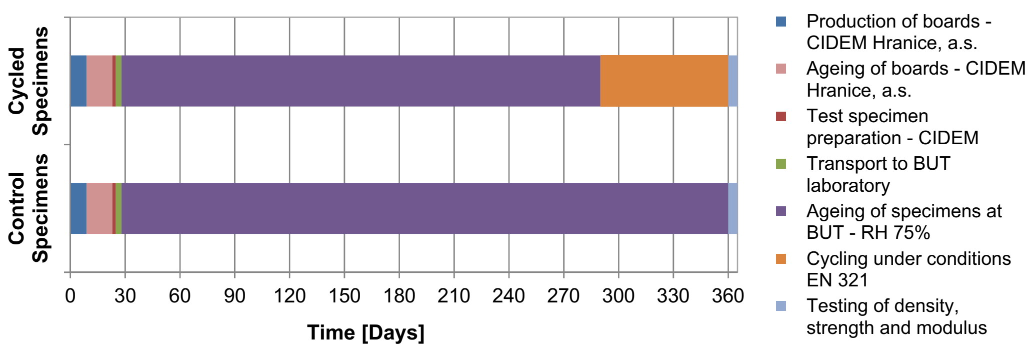

Fig. 7 Timeline of presented research

Shock action of temperature and humidity

To assess long-term durability from the point of view of sudden changes of temperature and humidity, environment defined in EN 321 [71] was selected. Course of the cycles is shown in Fig. 8.

All phases of the cycle follow each other without a break. Freezing and drying apparatuses with sufficient volume are set to the required temperature in advance so that placing of test specimens does not change set conditions. In this way, sudden changes of exposition environment are assured (of both temperature and humidity). Thermal gradient between the phase of freezing and drying reaches 90 °C, which is a relatively aggressive change of environment if presence of frozen water is considered. In total, 10 wet–frost–dry cycles were repeated.

Monitoring of properties during cycles

After each test cycle was completed (Fig. 8), dimensions and weight were determined and changes in the structure of cement-bonded particleboards were observed. Changes in the structure were determined with ultrasonic pulse method—process of measurement was in accordance with EN 12504-4 [72]. Ultrasonic pulse velocity (in accordance with [72]) and dynamic Young's modulus of elasticity (in accordance with ČSN 731371 [73]) were determined.

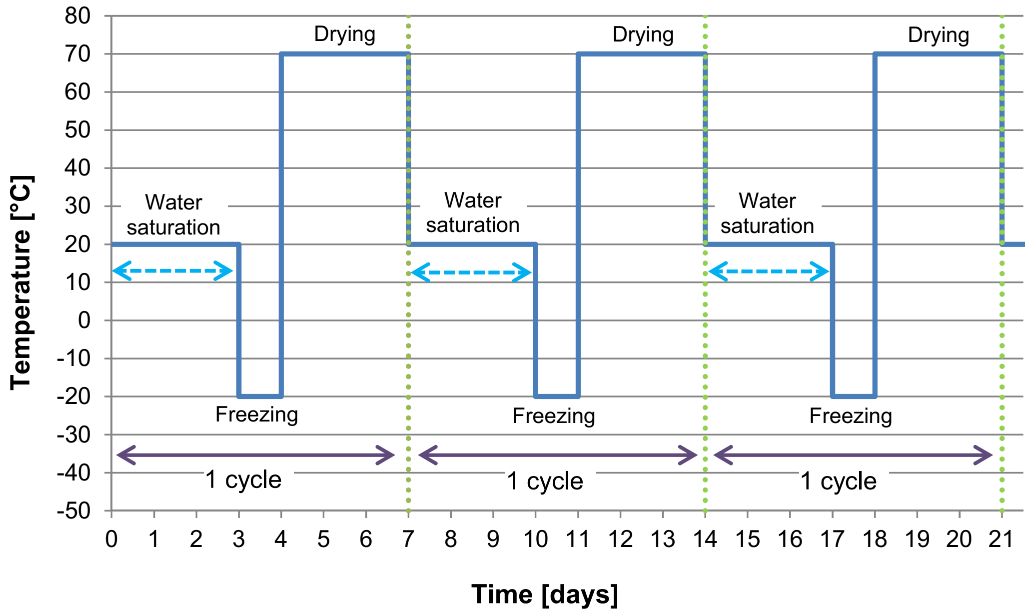

Fig. 8 Course of sudden changes of temperature and humidity (wet–frost–dry cycles)

Measurements were taken with the apparatus for determination of arrival time of the pulse PUNDIT LAB with accuracy 0.1 μs and natural frequency of the transducers 150 kHz. The boards were tested with ultrasonic pulse always after drying phase. For acoustic bond, silicon binding agent with constant elasticity was used because of high water absorbing capacity of the surface of the analysed material. Using materials stated in ČSN EN 12504-4 [72] could have a negative impact on repeated measurements taken at identical points.

Measurements of test specimens with dimensions 380 mm × 50 mm × 12 mm were carried out by semi-direct transmission. Direct transmission in longitudinal and transversal direction of samples with standardly used sounders is problematic (natural frequency is from 54 to 150 kHz) because thickness of the sample is 12 mm and minimal diameter of the sounder is 25 mm; moreover, surface on these areas is not compact. When sounding is carried out in the direction of thickness of a test specimen, the sounder touches tested surface by all its area. However, it is problematic to achieve reliable results at such a short distance.

Determination of the interval of passage of ultrasonic pulse was carried out on two diagonally located symmetrical measuring bases. The reason for this arrangement was capturing changes in the whole volume of the test specimen. Figure 9 presents scheme of transmission of the test specimens. Positions for attaching actuator and sensor on test specimens were marked with accuracy 0.5 mm. Locations of attachment are shown in Fig. 10. Before each ultrasonic measurement, length of measuring base d_1, d_2 were determined. Subsequently, transit times T_1, T_2 were determined.

Ultrasonic pulse velocity was calculated in accordance with relation (1) [72]:

$$V = L/T, \tag{1}$$

where V—ultrasonic pulse velocity [km/s]; L—length of measuring base [mm]; T—transit time [μs].

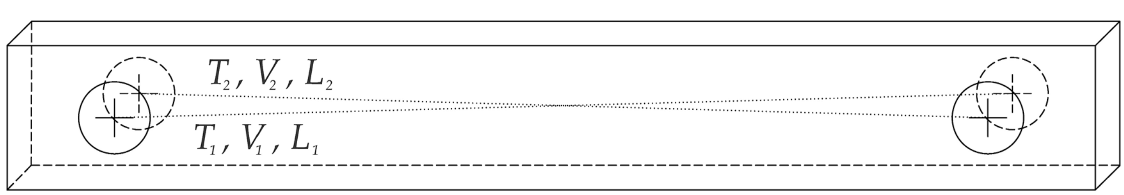

Fig. 9 Scheme of determination of length of measuring basis (L_1, L_2), time (T_1, T_2) and velocity (V_1, V_2) of passage of ultrasonic pulses through the test specimens

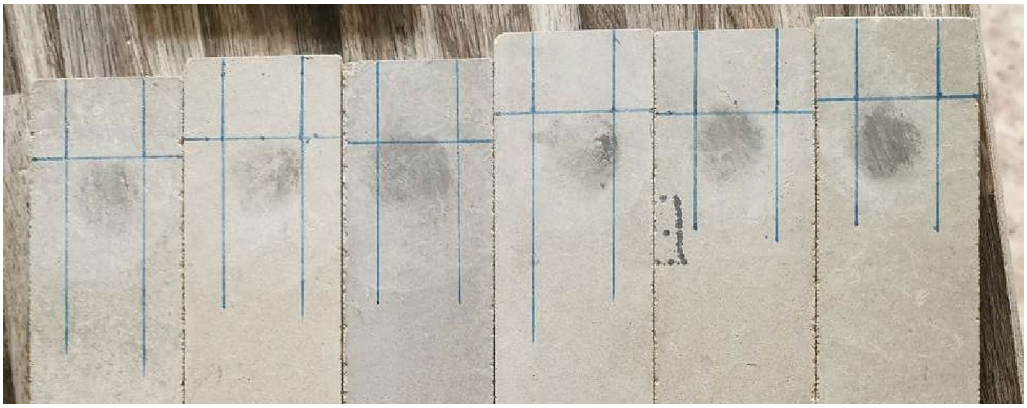

Fig. 10 Representative SE of test specimens after finishing the 4th cycle in accordance with EN321 and after determination of time of passage of ultrasonic pulse

Dynamic Youngs modulus of elasticity E_U was calculated in accordance with the relation (2) [73]:

$$E_U = V^2 \times D, \quad (2)$$

where E_U—dynamic Young's modulus of elasticity from ultrasonic pulse method [MPa]; D—density [kg/m^3].

Further, after the end of each cycle, changes of structure of cement-bonded particleboards were analysed in detail with the optical microscope Keyence VHX-950F. Attention was paid to areas of the boards which can be described as those with the highest probability of failure occurrence due to sudden changes of environment.

Testing of parameters after finished cycles

After 10 wet–frost–dry cycles were completed, both sets of specimens were placed in identical conditions in accordance with EN 310 [70] and EN 323 [69] so that weight and dimensions were stabilized. Density in accordance with EN 323 [69], bending strength and modulus of elasticity in bending according to EN 310 [70] were determined. Arrangement of the test of strength and modulus of elasticity corresponds to three-point flexure. For each set (=6 specimens), average value of analysed parameter was determined.

Results and discussion

Dimensional, volume and mass stability

Based on determined dimensions, shown in Fig. 11, it can be stated that changes in longitudinal direction (length) and transversal direction (width) are smaller than changes of thickness.

Length is characterized by contraction due to sudden changes of temperature and humidity. Contraction in range of 0.14% (SW07) to 0.17% (LI10) after 10 cycles was determined. The most significant decrease of length can be observed as early as after the first cycle, when the contraction is between 0.13% and 0.18%. Boards with secondary chips (SW07 and L10/S07) showed the best results. Development of changes roughly corresponds with findings in [31]. The authors determined contraction of cement-bonded particleboards (at the age of 360 days) due to changes of humidity by 0.16% to 0.17%. Nevertheless, development of measured values is slightly different. This is caused by differences of conditions. Changes of relative humidity at constant temperature can be characterized as slower compared to wet–frost–dry cycling.

Changes in the transversal direction of the particleboards are shown in Fig. 11b. Width reduction was determined between − 0.11 and − 0.18% up to the fifth cycle. Then, more steep growth followed and values are stabilized at 0.08% to 0.19%. Expansion in the direction of width was observed after 10 cycles. Changes in transversal direction are smallest in case of boards SW07. Data of relevant research (technical literature, etc.) were not found. Thus, comparing the results with findings of other authors is not possible.

The most marked changes due to humidity and temperature fluctuations shown in Fig. 11c occur in the direction of thickness. Fibres orientation in wood material and the chips themselves in the cement matrix play a significant role. Changes of thickness of boards can also be influenced by tension in spruce chips from manufacture of cement-bonded particleboards (pressing and elevated temperature). This residual tension in wood (contained in the composite) originates in the method of manufacturing process of composites [74]. After the first cycle, thickness was reduced by 0.19% to 0.31% The third wet–frost–dry cycle is characterized by changes of thickness close to 0%. Then, with following cycles, thickness

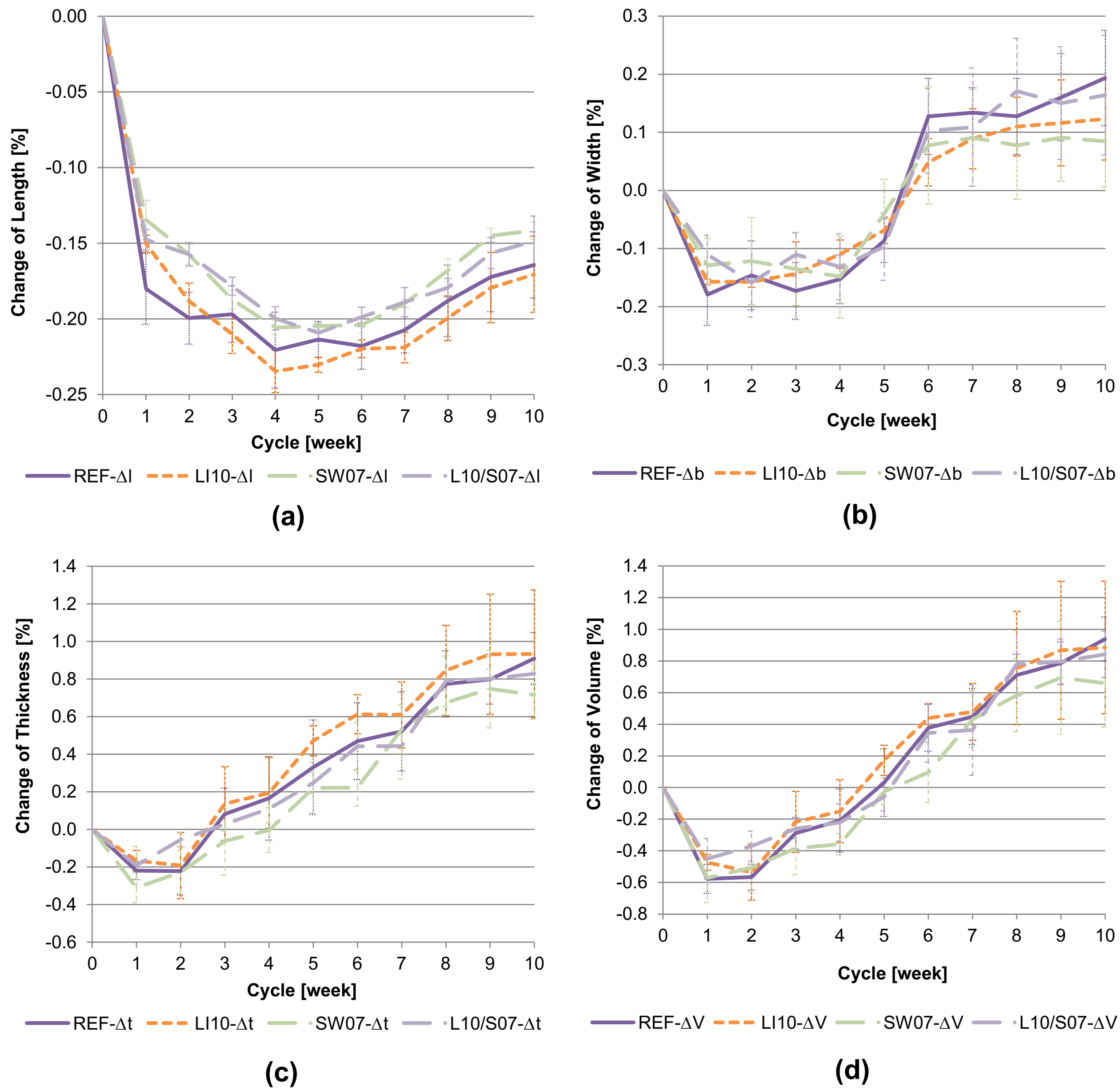

Fig. 11 Comparison of **a** length, **b** width, **c** thickness and d) volume changes of cement-bonded particleboards exposed to wet–frost–dry cycling according to EN 321

grew to the level of 0.72% to 0.93%. Boards SW07 showed the smallest change of thickness.

Comparison of the results shown in Fig. 11c with findings [34] implies that development of thickness changes is similar. However, final values are considerably higher. Irreversible growth of thickness by approx. 15% was determined after the seventh wet–dry cycle [34]. Course of cycling and composition of the particleboards (waste-paper and saw dust) are the reason for such a significant difference. That is why contraction did not occur [34]. Growth of thickness up to the fifth wet–dry cycle is inter alia caused by loss of coherence of cement matrix and chips due to shrinking of wood material during drying [34]. Changes of thickness practically stabilize after the fifth wet–dry cycle [34]. On the contrary, thickness shown in Fig. 11c keeps growing up to the tenth wet–frost–dry cycle.

The trend of volume curves is shown in Fig. 11d. This trend is practically an identical copy of thickness changes. During the first and second cycle, volumetric

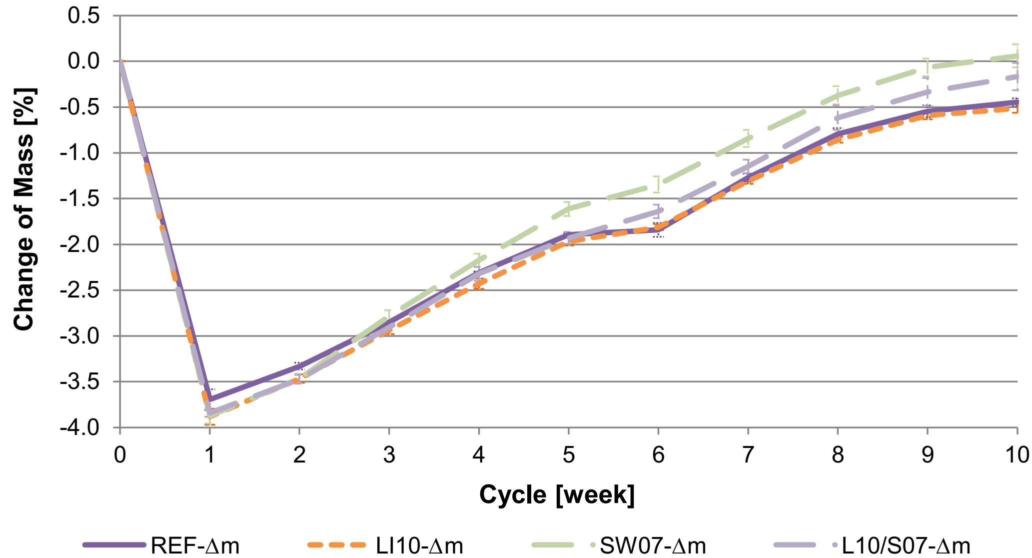

Fig. 12 Comparison of mass changes of cement-bonded particleboards exposed to wet–frost–dry cycling according to EN 321

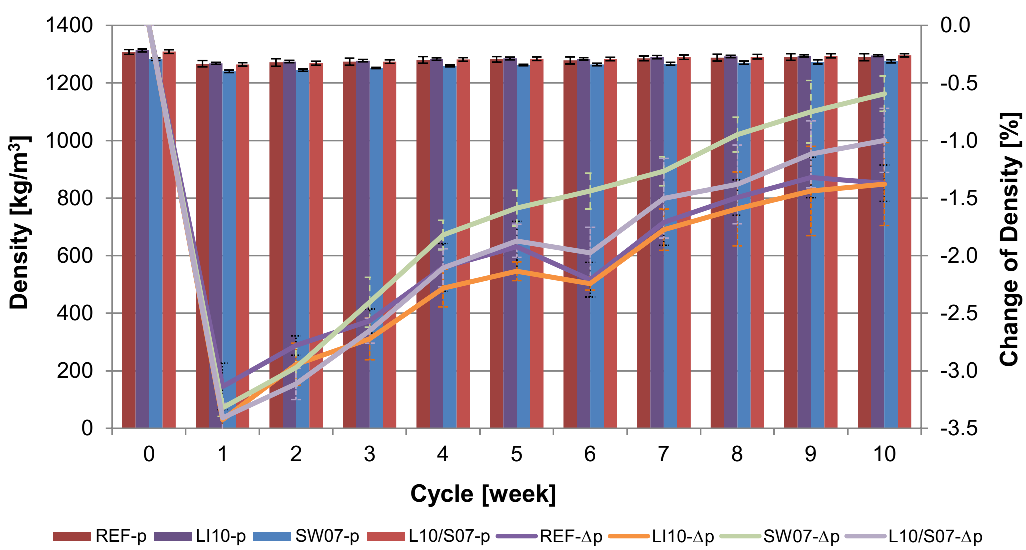

Fig. 13 Comparison of density changes of cement-bonded particleboards exposed to wet–frost–dry cycling according to EN 321

shrinkage is up to 0.6%. After the tenth cycle, volume is characterized by expansion by 0.61% to 0.91%. Boards SW07 show the least volumetric changes. Better resistance to volumetric changes of boards SW07 and L10/S7 can be inter alia explained by different releasing of water soluble carbohydrates and leachable substances. Secondary chips had already been stabilized and smaller amount of leachable substances can be expected [75]. Influence of dry–wet cycles on volumetric changes of wood is presented in [24]. The authors confirm that gradual growth of volume of aspen and birch wood occurs as a consequence of wet–dry cycles. No relevant findings of other authors concerning volumetric changes of cement-bonded particleboards due to wet–dry cycles or wet–frost–dry cycles were found. An interesting possibility of enhancing dimensional and volumetric stability could present partial substitution of primary chips with inorganic materials—for example aggregate based on sintered fly ash, which is analysed in detail in [76]. Volumetric stability could also be improved by materials with similar base, as presented in [77].

Better resistance of boards SW07 to wet–frost–dry cycling is related to different mechanism between cement matrix and secondary or primary chips. The first phase of wet–frost–dry cycle is characterized by water saturation of the particleboards. Secondary chips are more stabilized with lower water absorption as presented in Table 4. Therefore, smaller pressure acts to the cement matrix which surrounds the secondary chips. Particleboards are subjected to frost during the second phase of wet–frost–dry cycle. Thus, the water absorbed in the chips increases volume and pressure on the surrounding matrix. Primary chips contain larger amount of water and higher pressure on adjacent matrix occurs. Last phase of wet–frost–dry cycle is characterized by a rapid increase in temperature from – 20 °C to 70 °C. The pressures are released and volume of the chips decreases. Due to the amount of absorbed water, shrinkage of the secondary chips is lower. Primary chips are subjected to more pronounced contraction. Therefore, more frequent failures are formed in ITZ (internal transition zone) of primary chips and cement matrix.

Resistance of the particleboards to wet–frost–dry cycles is also affected by surface structure of the chips. Secondary chips have more closed surface, or cell structure. Therefore, products of cement matrix do not penetrate into the cell structure of secondary chips in the same way as that of primary chips. Bond of secondary chips with matrix could be negatively affected, i.e. weakened. Regarding better volume stability of secondary chips, slightly weakened bond may not be reflected in dimensional or volume changes. However, due to wet–frost–dry exposure, more noticeable decrease could occur in case of strength characteristics.

Mass changes are shown in Fig. 12. Due to wet–frost–dry cycles, weight decreases by 3.7% to 3.8% straight after the first cycle. After the tenth cycle, changes of weight are close to 0%. SW07 boards show slight growth of weight by 0.06%. Other types of boards show slight reduction of weight from 0.2 to 0.5%. Considerable decrease in weight in the initial phase of wet–frost–dry cycles can be ascribed to elimination of leachable substances from wood material [75]. Development of weight approximately corresponds with results of the research presented in [31]. Compared results show certain similarities in spite of difference in cycle characteristics. Cement-bonded particleboards showed decrease in weight in the first phase by approx. 2.1%, while after cycling, the boards showed increase in weight by approx. 2.7% [31]. Rapid decrease in weight and subsequent gradual growth can be ascribed to the combination of several phenomena taking place at the same time. First, elimination of leachable substances takes place [24, 75]. Then, hysteresis grows gradually [31]. Growth of weight could also be supported by crystallization of some of the products of cement matrix with gradual growing of these new forms.

Changes of density shown in Fig. 13 could also be expressed by determination of dimensions and weight. Trend of density changes is very similar to trend of mass changes. Boards SW07 showed the least reduction of weight after 10 wet–frost–dry cycles; by 0.05%. Boards REF and LI10 can be characterized by decrease by approx. 1.3%.

Secondary chips are characterized by higher density when compared to primary chips. However, this factor did not affect the final density of the particleboards with modified composition. This is due to the smaller amount of secondary chips. 7% of the primary chips were substituted, which is 3.53% of the total composition of the particleboard mixture.

Ultrasonic impulse analysis

Ultrasonic pulse velocity

Results of determining propagation of ultrasonic pulse velocity from beginning to the end of wet–frost–dry cycles of individual types of boards are shown in Fig. 14. Ultrasonic pulse velocity before beginning of wet–frost–dry cycles was between 2.401 km/s and 2.438 km/s and corresponds roughly with the results in [42]. However, boards with considerably lower density, 713 kg/m^3 and 718 kg/m^3, were studied. Ultrasonic pulse velocity after the 10th wet–frost–dry cycle was between 2.351 km/s and 2.385 km/s.

Based on the results presented in Fig. 14, it is obvious that in case of semi-direct transmission method, particleboard analysing in various (symmetrical) directions is important. Thus, the whole volume of the particleboard can be analysed exactly and in detail. Velocity can change in different directions because the board structure may not be homogeneous. All non-homogeneous areas and defects of the structure may become worse because of exposing boards to the wet–frost–dry cycles. An interesting fact is that slightly more distinct differences between velocities V1 and V2 shown in Fig. 14a, b can be observed on boards REF and LI10. This confirmed the assumption of different development of ultrasonic pulse velocity on individual measuring bases.

Results shown in Fig. 15 imply that all types of the analysed boards show considerable decrease in ultrasonic pulse velocity after the first wet–frost–dry cycle, which corresponds to decrease in density shown in Fig. 13 and which is confirmed by changes of dimensions and weight. This decrease can be explained by more significant changes in the structure of the cement-bonded particleboards. After the first wet–frost–dry cycle, differences in

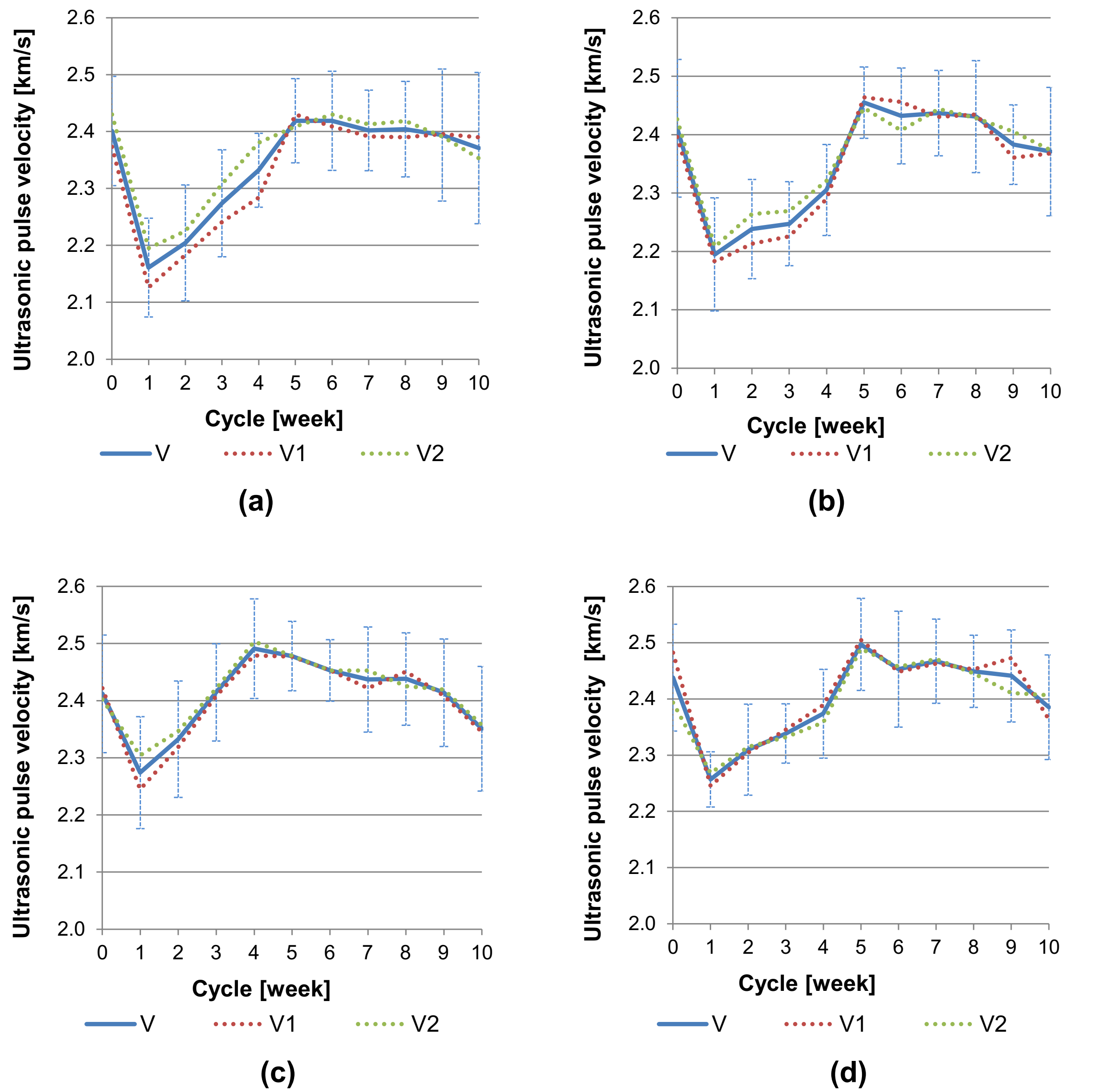

Fig. 14 Ultrasonic pulse velocity in cement-bonded particleboards exposed to environment according to EN 321 **a** REF; **b** LI10; **c** SW07; **d** L10/S07

velocity of individual types of boards are more distinct. Decrease in velocity after the first wet–frost–dry cycle is less noticeable on boards SW07 and L10/S07.

From 1st to 5th wet–frost–dry cycle, the boards show increase in ultrasonic pulse velocity. Most of test specimens achieved maximal ultrasonic pulse velocity after 5th cycle. The highest velocities after 4th or 5th cycle were observed on boards SW07 and L10/S07. Maximal values of velocities are roughly comparable to values before the wet–frost–dry cycling. Increase in velocity from 2nd to 5th cycle can be explained by compaction of the board structure. This is in relation with the fact that most of decomposable substances had already been leached from chips, and with the hysteresis of wood. Denser structure also relates to leaching of ions from cement matrix, while the crystalline phases gradually grow during further cycling. During compaction of the board structure, no detectable failures of bond on the interface between cement matrix and wood chips occur, neither in the structure of individual components. Compaction of the structure is confirmed by increasing of density, as shown in Fig. 13.

From 5 to 10th wet–frost–dry cycle, the boards show constant decrease in ultrasonic pulse velocity. This

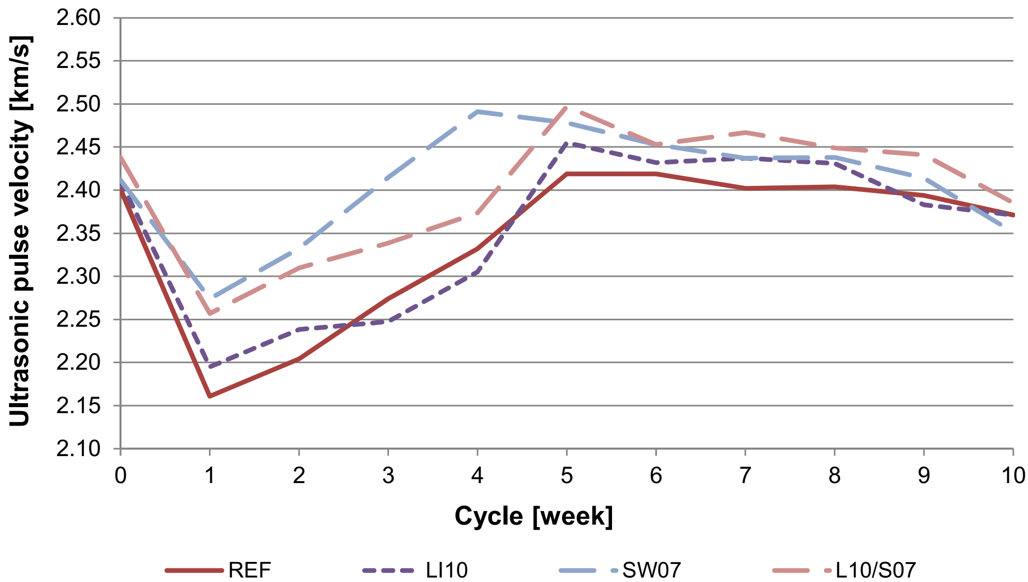

Fig. 15 Comparison of ultrasonic pulse velocity in cement-bonded particleboards exposed to wet–frost–dry cycles in accordance with EN321

decrease can be observed in spite of compaction of the structure and constantly increasing density shown in Fig. 13. Decrease in ultrasonic speed velocity is related to damage to inner structure of cement-bonded particleboard as a consequence of ongoing wet–frost–dry cycles. Gradually, coherence and bond of the interface between cement matrix and wood chips or in the structure of individual components is deteriorated. During this phase of exposition, influence of structural damage is more apparent during sudden changes of temperature and humidity than the aspect of gradual compacting of the structure. Ultrasonic pulse velocity between 5 and 10th wet–frost–dry cycle presented in Fig. 15 shows smaller differences between the analysed types of boards (compared to velocity between 1st and 5th cycle).

Decrease in ultrasonic pulse velocity after 10 wet–frost–dry cycles (related to ultrasonic pulse velocity before cycling) was between − 1.2 and − 2.5%. If decrease in ultrasonic pulse velocity after finished cycling is related to maximal value (i.e. after 4th of 5th wet–frost–dry cycle), the value of decrease is in the interval from − 2.0 to − 5.6%.

Initial ultrasonic pulse velocity (before exposition) is nearly identical for boards REF, LI10 and SW07. Boards L10/S07 showed slightly higher velocities. Boards SW07 and L10/S07 (with secondary chips) showed slightly more distinct decrease in ultrasonic pulse velocity (− 2.5%, and − 2.2%) after all 10 wet–frost–dry cycles compared to board REF and LI10 (− 1.2% and − 1.7%).

It was found out that density changes shown in Fig. 13 and ultrasonic pulse velocity, presented in Fig. 15, correspond only partially. It is an important finding, because some authors studied relation between density and ultrasonic pulse velocity in cement-bonded particleboards. Some scientists [40] found very strong dependencies. Effect of particle size of the analysed material is significant when density is low. However, none of the authors used ultrasonic pulse method for boards exposed to wet–frost–dry cycles. Ultrasonic pulse method was used for evaluation of only those boards which were stored in standard laboratory environment. The findings of research presented in this paper imply that cement-bonded particleboards show different behaviour (during exposure in various environments) with regard to relation between conventional and non-destructive test method.

Dynamic Young´s modulus of elasticity

Values of dynamic Young's elasticity modulus presented in Fig. 16 were between 7.46 GPa and 7.78 GPa before wet–frost–dry cycling. After finishing 10th cycle, average values of dynamic Young's modulus of elasticity were determined, in the interval from 7.05 GPa to 7.38 GPa.

Decrease in dynamic Young's modulus of elasticity after 10 wet–frost–dry cycles was determined in the range of − 3.8% to − 5.5%. If decrease in ultrasonic pulse velocity after finished cycling is related to maximum (achieved after 4th or 5th wet–frost–dry cycle), the values of decrease are in the range of − 3.8% to − 9.9%. Boards REF showed nearly identical values of dynamic Young's elasticity modulus before cycling and after finishing 5th wet–frost–dry cycle.

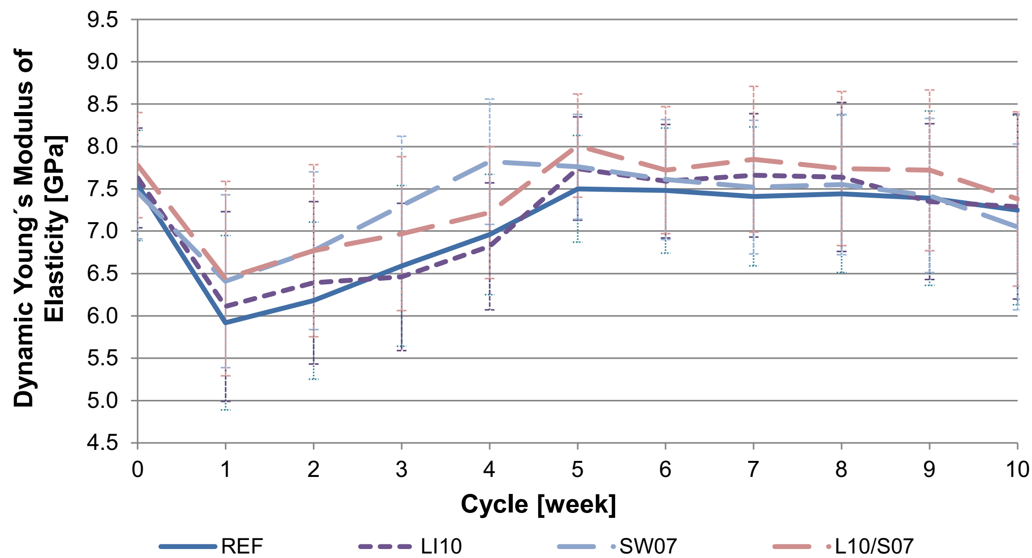

Fig. 16 Comparison of dynamic Young's elasticity modulus of cement-bonded particleboards exposed to wet–frost–dry cycling in accordance with EN321

Development of dynamic Young's elasticity modulus reflects development of ultrasonic pulse velocity. However, slight differences can be found in development of velocity and modulus shown in Figs. 15 and 16. Density is considered for calculation of dynamic Young's elasticity modulus. Therefore, density affects final values of modules and thus smaller variations between curves of individual types of boards are observed. This is valid for development of curves between 1st and 5th wet–frost–dry cycle.

Mechanical parameters

Modification of composition of cement-bonded particleboards had positive effect on bending strength. Comparison of bending strength is presented in Fig. 17. Boards REF showed 11.5 N/mm^2 and modified boards are characterized by strength from 12.2 to 12.7 N/mm^2. There are two reasons for the positive effect. Finely ground limestone contributes to hydration reaction of cement when nucleation and growth of C–S–H crystals occur [78–80]. Boards modified by limestone are characterized

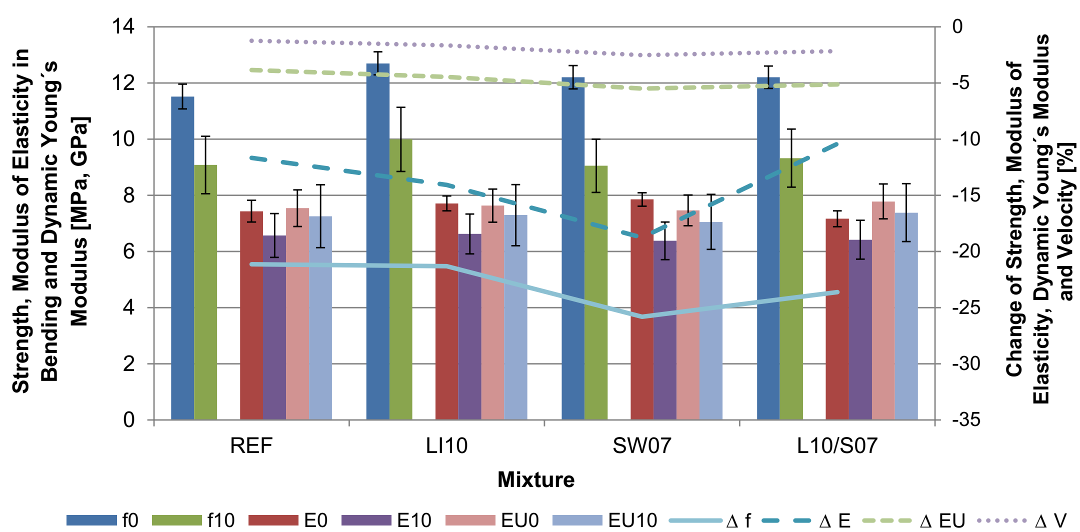

Fig. 17 Comparison of changes of bending strength, modulus of elasticity and dynamic Young´s modulus of elasticity before and after 10 wet–frost–dry cycles

by a slightly higher density, which is obvious from Fig. 13. Denser and more cohesive structure of cement matrix modified by limestone causes higher strength of boards LI10 and L10/SW07. Furthermore, boards SW07 contain once already mineralized secondary chips. These secondary chips are almost three times denser than the primary chips. Structure of the secondary chips is stabilized more effectively. Therefore, the secondary chips are characterized by higher strength, which positively affects strength characteristics of the modified boards.

Wet–frost–dry cycling had more negative effect in spite of higher initial bending strength of boards with modified composition. Bending strength of cement-bonded particleboards decreased by 21.3% to 25.8% by cycles. Bending strength of REF boards decreased by 21.1%. The considerable positive effect of finely ground limestone on properties of cement-bonded particleboards was confirmed by determined values of bending strength and modulus of elasticity in bending.

Secondary chips have a positive effect on dimensional or volumetric stability and changes of mass or density shown in Figs. 11, 12, and 13. However, slightly more distinctive reductions of strength and modulus of elasticity were observed. It is important that strength and modulus of elasticity in bending of all the tested boards after 10 wet–frost–dry cycles are above 9 N/mm^2. All boards fulfil requirements of EN 634-2 as regards minimal bending strength and modulus of elasticity in bending even after exposition to wet–frost–dry cycling.

The results show a need for complex approach to solving problems of explanation of phenomena taking place in cement-bonded particleboards during their exposition to wet–frost–dry cycles. Even though boards SW07 and L10/S07 showed better stability of dimensions and volume (as confirmed by the ultrasonic pulse method), their resistance to bending strain is slightly lower. Length of wood chips affects resistance of boards to wet–frost–dry cycling. Secondary chips have smaller dimensions and different shape compared to primary chips, which are shown in Figs. 2, 4 and 5. However, this factor had no effect on the values of strength and modulus of control specimens (without wet–frost–dry cycling). Secondary chips are gained through treatment in a jaw crusher, where bond of individual particles is damaged. Possibility that the structure of some particles used in the boards was already slightly damaged cannot be excluded. Their structure could be further damaged during board pressing (as a part of the production process). This may not have shown on dimensional and volumetric changes. However, failures could gradually grow due to wet–frost–dry cycles and this could explain decrease in bending properties.

Evaluation of relation between non-destructive analysis and mechanical properties

Decrease in ultrasonic pulse velocity, or dynamic Young's elasticity modulus, which is calculated from the values before and after cycling, characterizes development of changes in the structure of cement-bonded particleboards. However, these values differ from the trend of decrease in strength and modulus of elasticity shown in Fig. 17.

More appropriate (and more objective) way is assessment of final changes in the structure compared to maximal value after exposition to wet–frost–dry cycles, which is presented in Fig. 18. In this particular case, it is

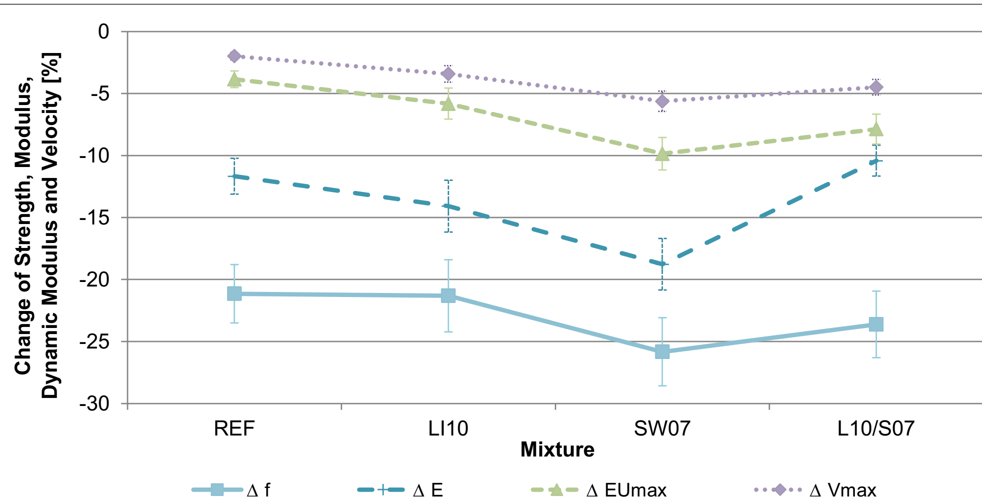

Fig. 18 Changes of parameters from measurement of ultrasonic pulse method and mechanical characteristics in cement-bonded particleboards during exposition to conditions in accordance with EN 321

determination of percentage differences based on reduction of ultrasonic pulse velocity of dynamic Young's elasticity modulus between 4th or 5th and 10th cycle. The development of changes of parameters from ultrasonic pulse method is quite similar to development found by means of destructive testing of bending strength and modulus of elasticity in bending. This method of evaluating by ultrasonic pulse method gives a better characterization of current condition of cement-bonded particleboards during adverse exposition (wet–frost–dry cycles).

Detailed analysis of structure

Analysis of structure with an optical microscope supports mainly development of ultrasonic pulse velocity. Figures 19 and 20 show that most significant opening of cracks in the contact zone of cement matrix were observed in the direction of thickness after first

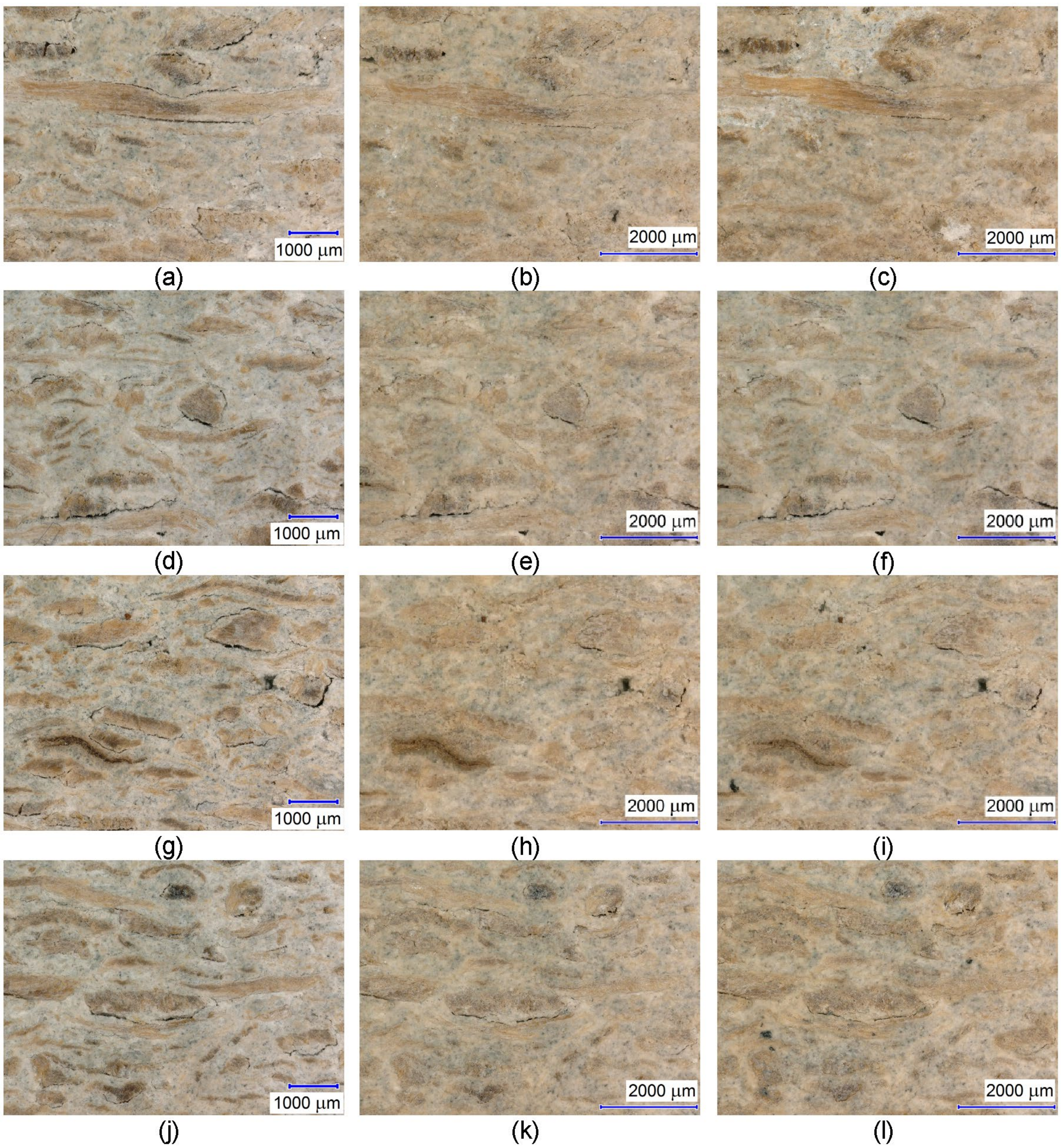

Fig. 19 Structure of boards REF: after **a** 1, **b** 5, **c** 10 cycles; LI10: after **d** 1, **e** 5, **f** 10 cycles; SW07: after **g** 1, **h** 5, **i** 10 cycles; L10/S07: after **j** 1, **k** 5, **l** 10 cycles in accordance with EN 321 (edge of the board, i.e. surface in the direction of thickness)

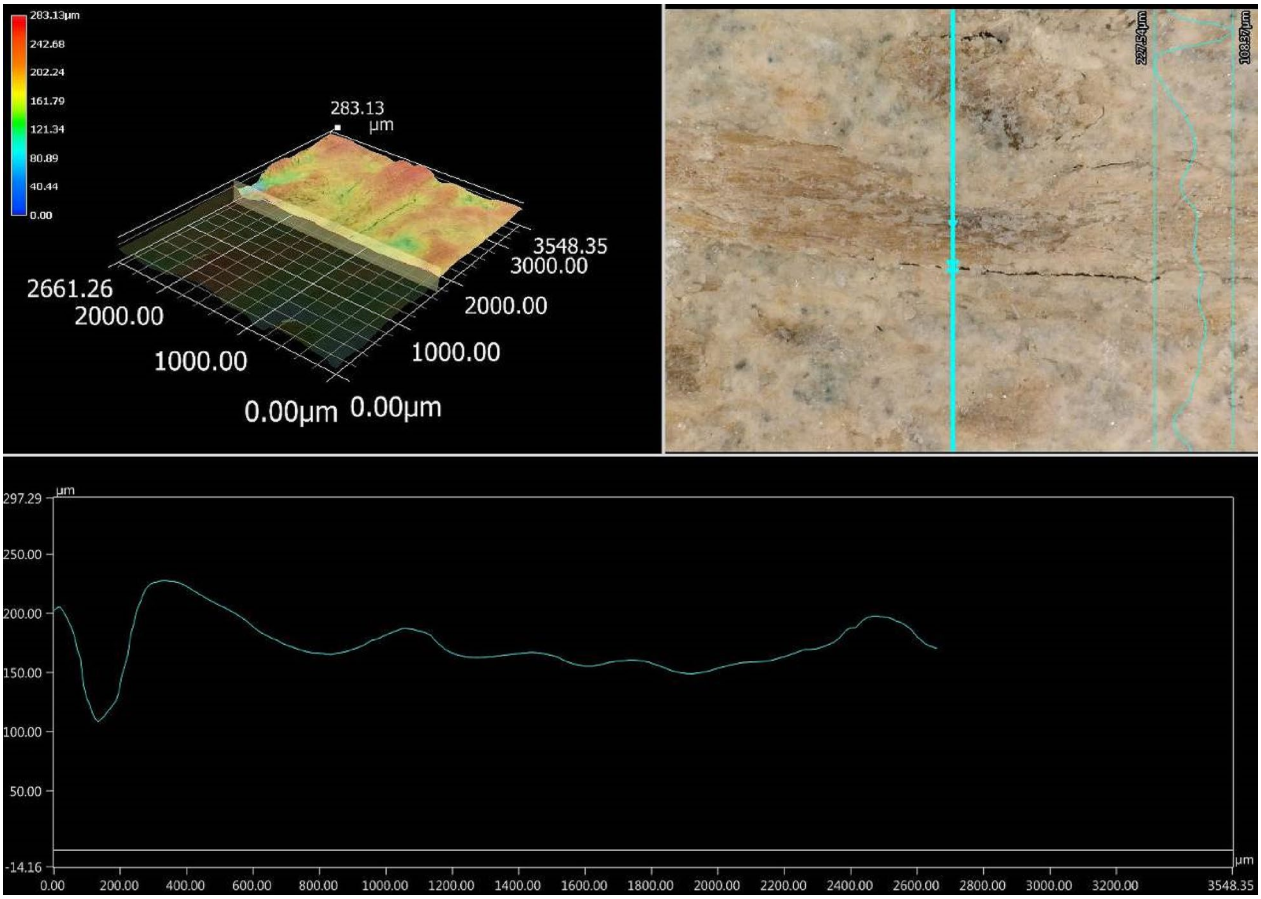

Fig. 20 Structure of REF boards—detailed evaluation of structure with analysis of relief of the surface after 5th wet–frost–dry cycle

wet–frost–dry cycle. Subsequently, influence of hysteresis of chips and crystallization of some ions contained in the cement matrix (for example Ca) show. This gradually closes the structure of the boards and makes it more compact. Observed cracks are practically closed. However, after the 5th wet–frost–dry cycle, aggressiveness of environment prevails—sudden changes of temperature and humidity. Gradually, the contact zone of cement matrix and spruce chips is damaged. Failures are observed in the chips too. These facts cause slight decrease in ultrasonic pulse velocity from the 5th to 10th wet–frost–dry cycle. Width of cracks gradually increases. Differences in the changes of structure of individual types of boards visible in the optical microscope images are not significant. This confirms minimal differences in development of changes in individual dimensions and volume of cement-bonded particleboards REF, LI10 and L10/S07.

Selected areas were analysed in 3D mode shown in Fig. 20, which makes it possible to evaluate structure of the relief; in this case it is related to changes in the direction of width of test specimens. Changes identified in the images confirm the results of dimensional and volumetric stability.

Figure 21 shows selected areas of top surface of the tested boards after finishing 10 wet–frost–dry cycles. Areas with cracks around the edges of the boards were selected intentionally. Identified cracks presented in Fig. 21 are beyond observation with the naked eye. Close observation shows only slight changes of shade. The microscope reveals presence of cracks. Leached or crystalized products were found in the cracks. This confirms the above-mentioned hypothesis that some substances leached from cement matrix form new phases. These new phases fill up the cracks formed by thermal and volumetric expansion/contraction, which creates more compact structure at first. Increasing expansion pressures then cause new failures, i.e. loss of bond between cement matrix and spruce chips. Swelling of wood can cause expansion pressures [81] which contribute to deterioration of the structure of cement-bonded particleboards.

An optical microscope can confirm or explain some phenomena which take place during exposure

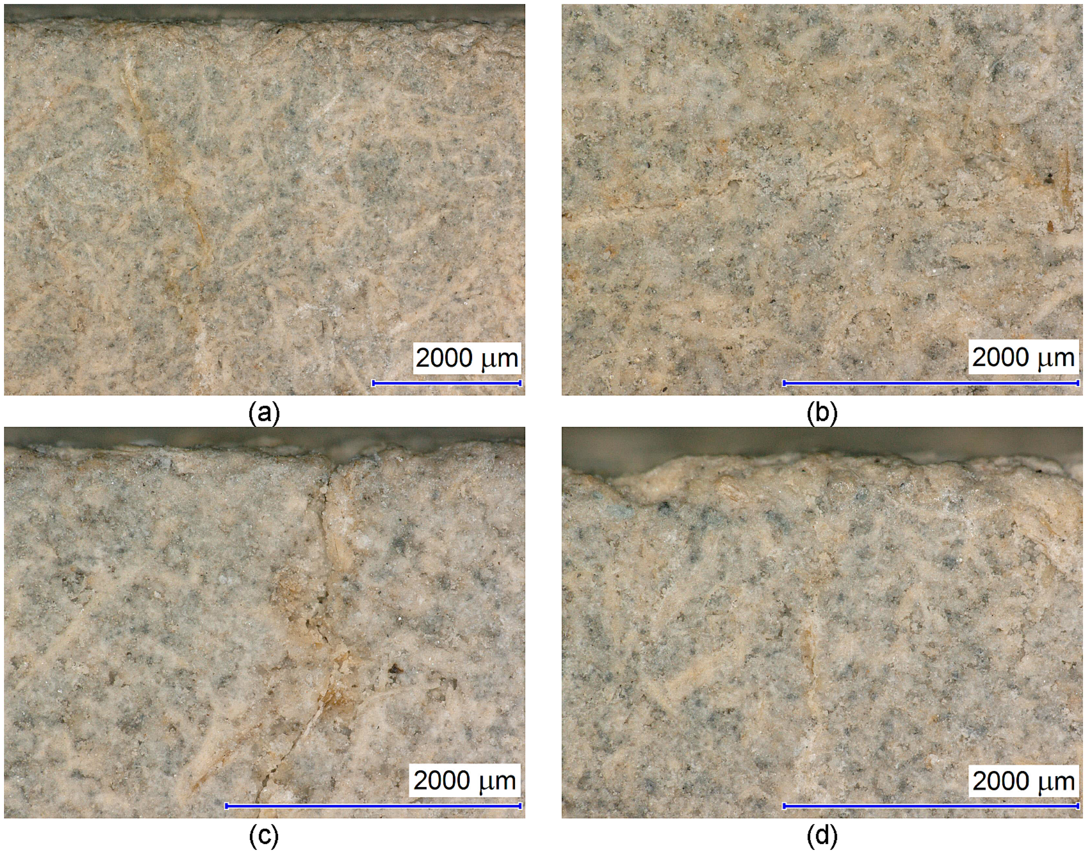

Fig. 21 Structure of boards after 10th cycle according to EN 321: **a** REF, **b** LI10, **c** SW07 and **d** L10/S07

of cement-bonded particleboards to wet–frost–dry cycling. This method is capable of analysing contraction/expansion of wood chips which affect considerably dimensional and volumetric changes of cement-bonded particleboards.

Conclusions

Based on the results and findings including comparison with findings of other authors, it can be stated that wet–frost–dry cycling in accordance with EN 321 is reasonable for verification of cement-bonded particleboards durability. The exposition environment of this technical standard covers real conditions even though conditions described in EN 321 are more aggressive. Thus effect of adverse conditions on cement-bonded particleboards is significantly accelerated.

High level of relation was found between changes of ultrasonic pulse velocity and width of crack in the interface between cement matrix and wood chips. This finding also corresponds to dimensional or volumetric changes of the boards. Strength and modulus of elasticity strongly depend on composition of the boards. Positive effect of chips used as a secondary raw material on dimensional changes of cement-bonded particleboards caused by sudden changes of temperature and humidity was proved. Finely ground limestone contributes to more resistant structure of boards which leads to improved bending properties. Wet–frost–dry cycling had more influence on bending strength (decrease by 21% to 29%) than on modulus of elasticity in bending (decrease by 12% to 19%). Assumed possibility of using ultrasonic pulse method combined with an optical microscope for evaluation of changes of cement-bonded particleboards inner structure during sudden fluctuations in humidity and temperature was confirmed. Further research should focus on changes of structure during wet–frost–dry cycles also by other non-destructive methods—for example resonance (or impact-echo) method. Measuring values of electric impedance for determination of moist distribution in analysed material (during wet–frost–dry cycling), as presented in [82] could also bring crucial results and findings.

It was proved that modification of composition of cement-bonded particleboards with finely ground limestone and secondary chips is suitable from the point of view of resistance to adverse conditions. The cuttings could be re-used (partially or totally) for production of the boards. Simplicity of the cuttings modification (crushing, grain selection) creates good conditions for increasing economical effectiveness of production and follows current trends of environmental protection, i.e. re-using wastes from production as secondary raw materials.

Abbreviations

REF: Reference cement-bonded particleboards; LI10: Cement-bonded particleboards modified with finely ground limestone; SW07: Cement-bonded particleboards modified with secondary spruce chips; L10/S07: Cement-bonded particleboards modified both limestone and secondary chips; TOC: Total organic carbon; V1, V2: Ultrasonic velocity; f0, f10: Strength in bending before and after wet–frost–dry cycles; E0, E10: Modulus of elasticity in bending before and after wet–frost–dry cycles; EU0, EU10: Modulus of elasticity in bending before and after wet–frost–dry cycles determined by ultrasonic impulse method; Δf, ΔE, ΔEU: Changes of strength and modulus of elasticity in bending after 10 wet–frost–dry cycles; PB: Particleboard; RH: Relative humidity; BUT: Brno University of Technology.

Acknowledgements

All tests and analyses were carried out at Brno University of Technology, Faculty of Civil Engineering, Institute of Technology of Building Materials and Components, and at AdMaS Centre.

Authors' contributions

Conceptualization, TM and MV; methodology, TM and JBr; validation, JBy, TM and JBr; formal analysis, JBy; investigation, TM and JBr; resources, TM; data curation, JBy and MV; writing–original draft preparation, TM; writing—review and editing, TM and JBy; visualization, TM; supervision, TM and JBy; project administration, TM and MV; funding acquisition, TM, JBy and MV. All authors read and approved the final manuscript.

Funding

Research presented in the article was funded by the Czech Science Foundation (GA ČR), project 19-00291S "Analysis of Processes during Forming the Structure in Silicate Composites with Organic Fillers and their Behaviour at Specific Conditions of Stress". This paper was also realized with the financial support from the national budget via the Ministry of Industry and Trade under the project FV30072 "Effective optimization of cement-bonded particleboards production waste utilization for manufacturing new competitive building materials".

Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Completing interests

The authors declare that they have no competing interests.

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Received: 21 October 2021 Accepted: 1 December 2021
Published online: 04 January 2022

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