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THE EFFECT OF SYNTHETIC ZEOLITE ADMIXTURE ON THE DURABILITY OF CONCRETE PAVING BLOCKS

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Abstract. Concrete paving blocks that were started to be used in the last century have become very popular. They became an alternative to the natural stone. Concrete paving blocks are used for the paving of pedestrian and vehicle zones. Durability is one of the main characteristics in the production of high-quality concrete paving blocks in the Baltic region climate zone. The article describes tests with concrete paving blocks, the top layer of which contains 5% of synthetic zeolite admixture obtained by means of low temperature synthesis in laboratory conditions. This zeolite admixture is obtained from aluminium fluoride production waste. The durability of concrete paving blocks was tested according to abrasion resistance, tensile splitting strength, absorption and frost resistance. The test results revealed that 5% of zeolite admixture added to the top layer of concrete paving blocks reduce the absorption, increase the tensile splitting strength by more than 10%, and decrease abrasion by 6.5%. The zeolite admixture used in concrete paving blocks reduces the scaling about 4 times after 28 freeze-thaw cycles when 3% NaCl is used as the freezing solution. The tests revealed that synthetic zeolite admixture can be used in concrete elements production by means of vibropressing (pavement elements) to increase their durability.

Keywords: abrasion, concrete, durability, freeze-thaw resistance, paving block, synthetic zeolite, tensile splitting strength.

1. Introduction

Concrete paving blocks are widely used in Europe. In Northern European climate conditions these blocks are often exposed to high humidity and freeze-thaw cycles, the number of which has a great effect on the durability of the paving blocks. During winter time de-icing salts are used to melt snow and ice on roads and walkways; however de-icing salts speed up the deterioration of concrete elements (Šelih 2010). Durability is one of the most important concrete products quality indications in Lithuania.

The mechanism of concrete deterioration due to freeze-thaw and de-icing salts has been studied since 1938. American researcher (Powers 1949) was among the pioneers of these studies along with Scandinavian and German scientists. They discovered that concrete deterioration was caused by the following conditions:

- obtained hydraulic pressure of freezing water and subsequent increase in volume;
- build-up and expansion of ice crystals in capillary pores;
- osmotic pressure obtained as a result of the difference of alkali and salt concentration in the liquid phase;
- ice formation in the top layer of vibropressed concrete products (Yener 2014).

Surface scaling is the main type of concrete deterioration caused by de-icing salts. The most widely used de-icing salts are: NaCl (sodium chloride), $MgCl_2$ (magnesium chloride), and $CaCl_2$ (calcium chloride). When concrete is subjected to de-icing agent solutions, the deterioration process is accelerated 4–5 times by osmotic pressure built-up during cyclic freezing and thawing. The adverse effect of de-icing salts is observed in road concrete, pavement elements and other concrete structures (Šelih 2010).

Recently, extensive studies of modified concretes have been carried out. Researchers test the effect of various plasticizing admixtures or active mineral additives, such as silica fume (Brykov *et. al* 2010; Xu *et. al* 2000), fly ash (Brykov *et al.* 2010; Canpolat *et al.* 2004), catalysts from catalytic oil cracking reactors (Paya *et al.* 1999; Stonys *et al.* 2008; Tseng *et al.* 2005) etc.

Admixtures from industrial waste have many positive effects: they not only improve the properties of concrete but are also recycled to reduce the industrial waste. Public limited liability company (PLLC) *Lifosa*, a Lithuanian producer of aluminium fluoride, generates about 5927 t of waste per year. Lithuanian researchers have studied the potential utilization of this waste. One of the results is the *Patent LT 5756* (Skripkiūnas *et al.* 2011).

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The tests have led to the conclusion to use laboratory synthesized zeolite in cement concrete as strength and durability enhancing admixture. Zeolite synthesized from aluminium fluoride waste significantly improved the durability of concrete products. There are many tests (Sabatino *et al.* 2011; Poon *et al.* 1999; Yılmaza *et al.* 2007) showing the increased strength of cement concrete when part of the binding agent is replaced with zeolite admixture. Synthetic zeolite used in concrete pavement elements enables to adjust porosity parameters and freeze-thaw resistance.

In many tests silica fume, slag and fly-ash were used as cement substitutes. Zeolites contain a high amount of active SiO_2 and Al_2O_3. Silica fume and fly ash, like other pozzolanic substances, can improve concrete strength through $Ca(OH)_2$ reaction with pozzolans. Zeolite, like other pozzolanic substances, has a great effect on the strength of hardened cement paste. On the other hand, zeolites also influence the formation of adverse products in hardened cement paste, such as alkali and other complex compounds (Sabatino *et al.* 2011). Lithuanian researchers investigated the effect of synthetic zeolite (hydrosodalite) on the compressive strength of hardened cement paste. Cement paste specimens containing 2%, 5%, 10% and 15% of zeolite admixture were formed and tested. The results showed that the highest compressive strength was achieved with 10% of zeolite admixture (Vaičiukynienė *et al.* 2011).

Skirpkiūnas *et. al* (2009) and co-authors tested the compressive strength of hardened cement paste with different content (5%, 10%, 15%, 20%) of zeolite admixture (hydrosodalite) after 3, 7 and 28 days of curing. The test with 5% of zeolite admixture showed reduced compressive strength of hardened cement paste. The increase in compressive strength, especially after 3 and 7 days of curing, was noticed with 5–20% of zeolite admixture (Skripkiūnas *et al.* 2009).

Vaičiukynienė *et al.* (2014) with co-authors claim that zeolite admixtures in hydration process of cement systems influence the re-crystallization of portlandite mineral $Ca(OH)_2$ into calcium hydrosilicate products and the morphology of newly formed calcium hydrosilicate products. The test results showed that the increasing content of zeolite admixture from 0%, 3%, 5% to7% reduces the content of $Ca(OH)_2$ in hardened cement paste: after 28 days of hydration $Ca(OH)_2$ caused weight loss ranged from 4.40% to 3.5% and 3.24% when admixture content was 5% and 7%. It was found that synthetic amorphous zeolite can be used in cement systems to replace part of Portland cement with a cheaper material. The best results were obtained when 5% Portland cement was replaced with amorphous zeolite. In this case the compressive strength was increased by about 10% (from 40 MPa to 44 MPa) (Vaičiukynienė *et al.* 2014).

According to Vaičiukynienė *et al.* (2012), the pozzolanic activity of hydrosodalite and the formation of hydroaluminate phases increase the compressive strength of hardened cement paste, especially at the beginning of hydration process. According to the test results, the highest compressive strength was recorded in specimens containing 15% of modified hydrosodalite. Higher compressive strength of the specimens may be related with active SiO_2 and Al_2O_3 present in modified hydrosodalite (Vaičiukynienė *et al.* 2012).

According to Nagrockienė *et al.* (2014) after 28 freeze-thaw cycles the scaling of concrete specimens significantly decreases when 10% of zeolite is added to Portland cement concrete. They also found that synthetic zeolite admixture changes the morphology of hardened cement paste: $Ca(OH)_2$ and ettringite contents reduce, whereas C-S-H phase increases and thus influences the increase in density of hardened cement paste, its strength and freeze-thaw and de-icing salts resistance (Nagrockienė *et al.* 2014).

Concrete paving blocks are produced by means of vibropressing. The air is removed to the highest extent and the product is highly compacted. This process enables to significantly reduce concrete pores and subsequently water absorption, which effect product strength deterioration caused by cyclic freezing and thawing in the cold season. Concrete porosity and air entrainment during the production of concrete mix must be strictly controlled in the production of road paving elements, besides these elements have higher frost resistance requirements depending on the conditions of their use (Kumara, Bhattacharjee 2003).

Concrete pores may be closed by adding admixtures to concrete mix. Less capillary pores and more closed pores increase the durability of concrete products. The fine-grain structure of the mix enables to produce concrete paving blocks by precisely repeating moulding process. Paving blocks are usually made in two stages. In the first stage the main layer of concrete is poured into the form and compacted; afterwards a top layer is poured onto the compacted main layer. The main layer contains coarser aggregates up to 11 mm in size; the top layer contains only fine aggregates up to 4 mm in size.

No scientific articles were found about using zeolite admixture in concrete products. A number of research articles analyse the characteristics of paving blocks where crushed concrete waste (Poon *et al.* 2002; Poon *et al.* 2009), crushed ceramic brick waste (Jankovic *et al.* 2012; Poon *et al.* 2006) and marble waste (Gencel *et al.* 2012) is used as aggregates.

Concrete paving blocks from concrete of exposure classes XF3 and XF4 are mainly used. It is recommended to ensure the durability of paving blocks related to environmental actions in the country where the products will be placed on the market. There are several freeze-thaw resistance testing methods to be used according to the standards applicable in Europe. The main paving block durability testing method is one-sided freezing method according to *EN 1338:2003/AC:2006 "Concrete Paving Blocks – Requirements and Test Methods"* standard. Freeze-thaw and de-icing salt resistance is determined by subjecting only one surface of the product to freeze-thaw cycles. A similar method CDF with 56 freeze-thaw cycles is used for other types of concrete according to *CEN/TS 12390-9:2006*

"Testing Hardened Concrete – Part 9: Freeze-Thaw Resistance – Scaling".

The goal of the testing described in this article is to determine the changes in density, water absorption progress, abrasion resistance, splitting tensile strength and freeze-thaw resistance (using 2 methods) of concrete paving blocks, the top layer of which contains up to 5% of synthetic zeolite admixture.

2. Materials

Compositions of vibropressed concrete used for the main and top layers of the paving blocks tested for durability are presented in Table 1.

Cement CEM I 42.5R produced by PLLC *Akmenės cementas* was used as the binding material: 350 kg for the main layer and 500 kg for the top layer. 5% of cement was replaced by zeolite admixture in the top layer. The following aggregate materials were used in the main layer: crushed gravel 2/11, crushed granite 2/8 and 0/4 fraction sand. The following aggregate materials were used in the top layer: granite screenings 0/4, sand 0/2. Plasticizer Plastolith was added at different weights in the main and top layers, 1.05 kg and 2.0 kg respectively. Part of the cement in the top layer was replaced by zeolite admixture obtained by means of low-temperature synthesis at 95 °C for 120 minutes under laboratory conditions. AlF_3 production waste from PLLC *Lifosa* was used to obtain the zeolite admixture. Synthesis products were modified with $CaCl_2$ (Skripkiūnas *et al.* 2011). Chemical composition of zeolite is presented in Table 2.

3. Specimens formation and test methods

Paving blocks of dimensions 198×98×80 mm were produced in the factory by means of vibropressing. Concrete pastes were prepared in forced action mixers. One mixer was used to prepare the paste for the main layer and the other for the top layer. Materials in the mixers were mixed for 210±5 seconds. The mixed pastes were placed into the hoppers of the block moulding machine. The block moulding machine is computer controlled by the operator from the control unit. The blocks were moulded in two stages. In the first stage the paste for the main layer was placed into the moulds and vibrated; afterwards the top layer was placed onto the main layer. The top layer in the tested blocks contained zeolite admixture added at 5% by mass of cement. The moulded blocks were cured for 48 hours in the curing chamber at +16±2 °C. After two days of curing in the chamber the paving blocks were manually selected and placed on the pallets and stored outside in the average temperature of 4±2 °C. Physical and mechanical properties of the products were determined after 7 and 28 days of curing. The durability properties of paving blocks were measured according to the standards listed in Table 3.

Freeze-thaw resistance of the paving blocks was tested according to two different methods. At first the paving blocks were tested according to *LST EN 1338/AC Annex D*. All surfaces of specimens, except the top surface, were covered with de-icing salt resistant 3 mm-thick rubber sheet. The edge of the rubber sheet was glued 20 mm above the concrete surface. All surfaces of specimens, except the top surface, were thermally insulated prior to starting the cyclic one-sided freeze-thaw test. To this end the specimens were placed into a box made of 20 mm-thick polystyrene foam. A 5–2 mm layer of 3% NaCl solution was placed on top of the tested surface 30–15 minutes prior to the placing of the specimens into the climate chamber. The box was covered with a horizontal polyethylene sheet to protect the solution from vaporization. The freeze-thaw cycle was controlled in the automated freezing chamber RUMED 3001 bis 3601 with adjustable temperature and duration. The surface temperature of at least one specimen was recorded by the computer during the freeze-thaw test in order to have the freeze-thaw cycle within the test limits. The changes of freeze-thaw cycle duration and temperatures are presented in Fig. 1.

Table 1. Composition of concrete in pavement blocks (amounts of materials for 1 m^3)

Aggregates	Main layer	Top layer without zeolite admixture	Top layer with 5 wt% of zeolite admixture
Cement CEM I 42.5 R	350	500	475
Crushed gravel 2/11	400	–	–
Crushed granite 2/8	400	–	–
Sand 0/4	1100	–	–
Sand 0/2	–	814	814
Granite screenings 0/4	–	750	750
Zeolite	–	–	17.74
Water	120	128	128
Plasticizer (Plastolith)	1.05	2	2

Table 2. Chemical composition of zeolite

SiO_2	Al_2O_3	Na_2O	CaO	H_2O
39.3	26.6	7.2	7.5	19.4

Table 3. Test methods of paving blocks

Properties	Testing methods
Density after 28 days of curing	*EN 12390-7*
Total absorption after 7 and 28 days of curing	*EN 1338/AC Annex E*
Tensile splitting strength after 7 and 28 days of curing	*EN 1338/AC Annex F*
Abrasive resistance after 7 and 28 days of curing	*EN 1338/AC Annex G*
Freeze-thaw and de-icing resistance after 28 days of curing	*EN 1338/AC Annex D*
Freeze-thaw and de-icing resistance after 28 days of curing (CDF test)	*CEN/TS 12390-9*

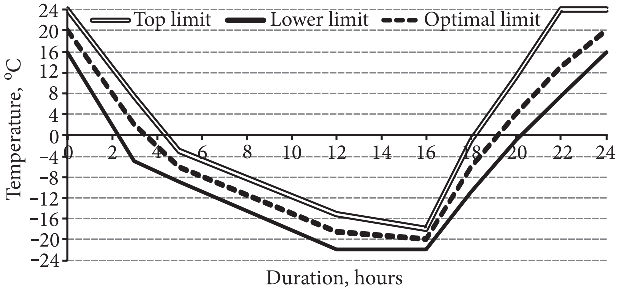

Fig. 1. The function of freeze-thaw cycles and temperature changes according to *EN 1338/AC Annex D*

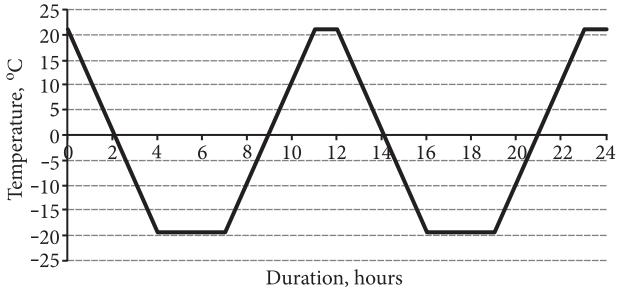

Fig. 2. The function of freeze-thaw cycles and temperature changes according to *CEN/TS 12390-9* CDF method

Table 4. Density of paving blocks

Characteristics	Paving blocks without zeolite admixture, kg/m^3		Paving blocks with zeolite admixture, kg/m^3	
	from	to	from	to
Low density	2284	2314	2253	2304
Medium density	2315	2353	2305	2345
High density	2354	2378	2346	2378

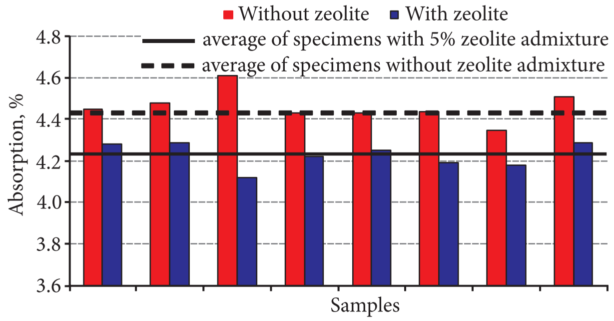

Fig. 3. Water absorption of paving blocks after 7 days

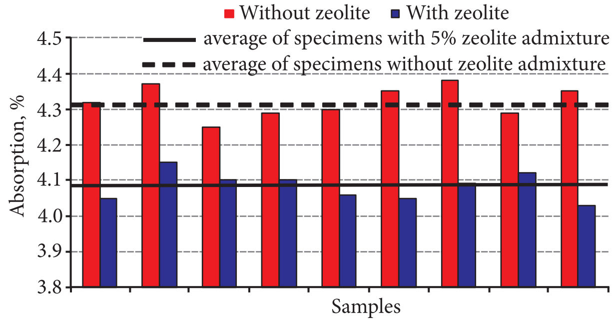

Fig. 4. Absorption of paving blocks after 28 days

The specimens were taken out after 7, 14, 21, 28, 42, 56 freeze-thaw cycles and the test surface was washed/cleaned. The collected spall was dried at 80 °C.

The second test was done according to CDF method of the standard *CEN/TS 12390-9:2006 "Testing Hardened Concrete. Freeze-Thaw Resistance. Scaling"*. 3% NaCl solution was used as de-icing agent. The sides of the specimen were sealed with solvent-free epoxy resin durable at −20 °C temperature. During freeze-thaw cycles the specimens were kept in stainless steel containers. A 7 mm height spacer was placed on the bottom of the container to guarantee a defined thickness of the liquid layer between the test surface and the container. The freeze-thaw cycle lasted for 12 hours. The temperature curve is presented in Fig. 2.

The mechanical cleaning of the test surface was done after 7, 28 and 56 cycles at the temperature higher than 15 °C. The scaled material was filtered, dried and weighted.

4. Test results

Due to high differences in the properties of manufactured products, the paving blocks of each composition were divided into three density levels: low density, medium density and high density (Table 4). The medium density of paving blocks is the total average density of all tested blocks in kg/m^3. Low density is the medium density minus 20 kg/m^3 and less; high density is the medium density plus 20 kg/m^3 and more. Three density levels were used for the purpose of precision in further tests, i.e. abrasion resistance, tensile splitting strength, water absorption and freeze-thaw resistance. The results of all these tests, except for tensile splitting test, will be presented for the blocks of the medium density.

No changes in the blocks with zeolite admixture and without the admixture were found after the densities of paving blocks were determined. The absence of difference was expected because zeolite admixture was used only in the top (finishing) layer of about 8–10 mm. It should be noted, however, that density intervals of the tested blocks differ. The difference in densities of blocks with zeolite admixture is 125 kg/m^3; the difference in densities of blocks without zeolite admixture is 94 kg/m^3. It should be also noted that medium densities of paving blocks differ: 2315–2353 kg/m^3 in blocks without zeolite admixture; 2305–2345 kg/m^3 in blocks with zeolite admixture. These results lead to the conclusion that zeolite admixture slightly reduces the density of concrete paving blocks.

The results of water absorption testing after 7 and 28 days are presented in Figs 3–4.

The properties average values of specimens without zeolite admixture are marked by dotted lines in Figs 3–8, and the solid lines mark the average values of specimens with 5% zeolite admixture in the top layer of concrete paving blocks. 9 blocks of each composition were tested. Fig. 3 illustrates that after 7 days the average water absorption values of the blocks with zeolite admixture are 5.3% lower than the values of the blocks without zeolite admixture.

Water absorption test results after 28 days show that water absorption in paving blocks with 5% of zeolite admixture is 5.5% lower compared to the paving blocks without zeolite admixture in the top layer. The comparison of water absorption after 7 and 28 days shows insignificant reduction of water absorption after 28 days due to cement hydration processes. Although the volume of the top layer is small, it has a great effect on the average water absorption.

Abrasion resistance is one of the main properties in the evaluation of the durability of concrete paving blocks. This test was done after 7 and 28 days. The test results are presented in Figs 5–6.

Four paving blocks were tested both after 7 and after 28 days to determine the abrasion resistance. Fig. 5 illustrates that zeolite admixture in the top layer has a slight positive effect on abrasion resistance of concrete paving blocks after 7 days. A 1.22% narrower groove was received compared to specimens without zeolite admixture.

However, the additional curing of 21 day gave a significant positive effect of zeolite admixture on the abrasion resistance of the paving blocks (Fig. 6). A 6.5% narrower groove is seen in specimens with zeolite admixture in the top layer. The comparison of grooves after 7 and 28 days showed that after 28 days the groove in the blocks without zeolite admixture reduced 1.24 mm compared to the groove after 7 days, whereas the groove in specimens with zeolite admixture reduced 2.22 mm after 28 days. The abrasion of paving blocks reduces with time due to cement hydration and hardening of the cement paste. After this test it can be state that zeolite admixture used in the top layer of paving blocks reduces the abrasion, i.e. the surface of the paving block becomes harder and more durable.

The tensile splitting strength of concrete paving blocks after 7 and 28 days of curing was tested in accordance with *LST EN 1338/AC Annex F*. Three blocks of low density, eight blocks of medium density and three blocks of high density were tested to determine the tensile splitting strength.

Fig. 7 illustrates tensile splitting strength test results after 7 days of curing. The solid line illustrates the average values of tensile splitting strength of paving blocks with zeolite admixture; the dotted line illustrates the average tensile splitting strength values of paving blocks without zeolite admixture. The results illustrate that low density paving blocks with zeolite admixture have 4% higher tensile splitting strength compared to the strength of blocks without zeolite admixture. Accordingly, the tensile splitting strength is 5.4% higher in blocks of medium density and 6.7% higher in blocks of high density. Therefore, the addition of 5% zeolite admixture by mass of cement into the top layer of vibropressed concrete paving blocks produces 5.4% higher tensile splitting strength after 7 days of curing.

Fig. 8 presents tensile splitting strength testing results after 28 days. The solid line illustrates the average values of tensile splitting strength of paving blocks with zeolite admixture; the dotted line illustrates the average tensile splitting strength values of paving blocks without zeolite admixture.

The same positive effect of zeolite admixture on tensile splitting strength of the paving blocks is seen after 28 days of curing as after 7 days of curing. The tensile splitting strength increases 5.4% in low density blocks with zeolite admixture, 6.1% in medium density blocks, and 7.6% in high density blocks compared with the blocks without zeolite admixture. After 28 days of curing the tensile

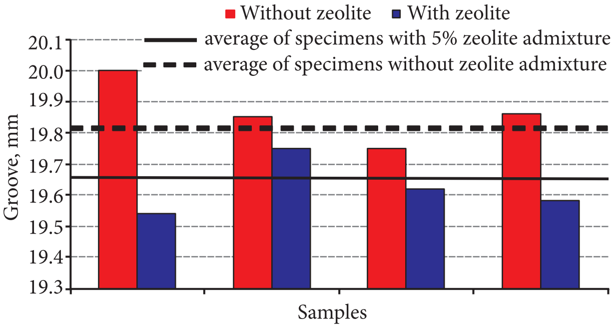

Fig. 5. Abrasion resistance of paving blocks after 7 days

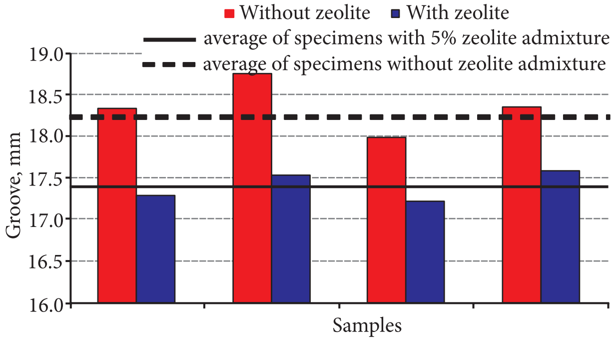

Fig. 6. Abrasion resistance of paving blocks after 28 days

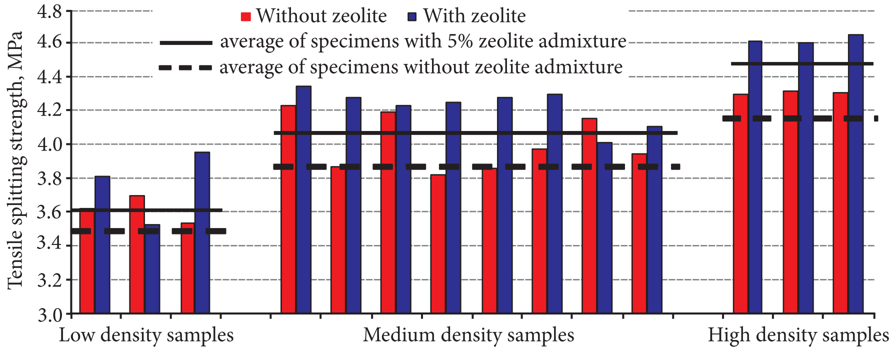

Fig. 7. Tensile splitting strength of paving blocks after 7 days of curing

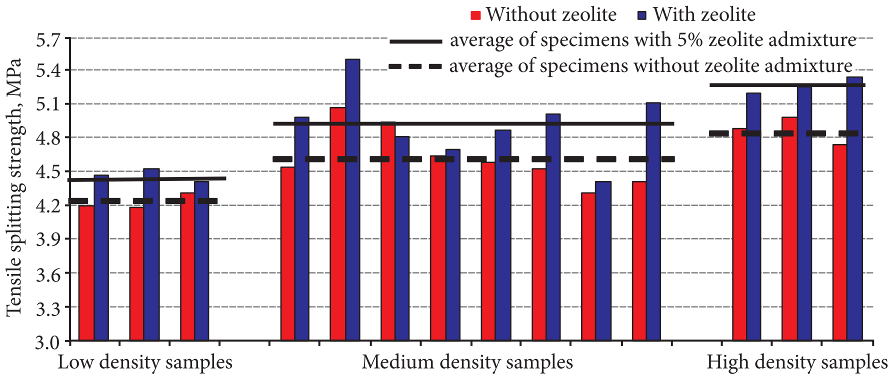

Fig. 8. Tensile splitting strength of paving blocks after 28 days of curing

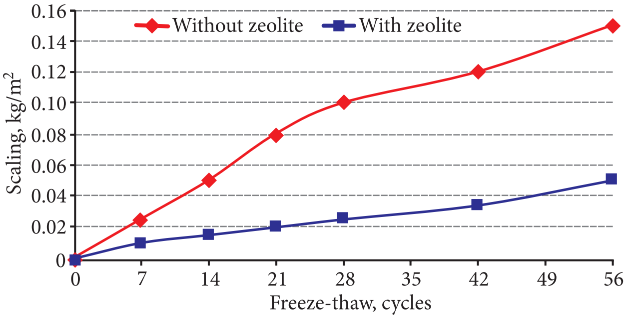

Fig. 9. Scaling of paving blocks after 56 freeze-thaw cycles according to LST EN 1338/AC Annex D

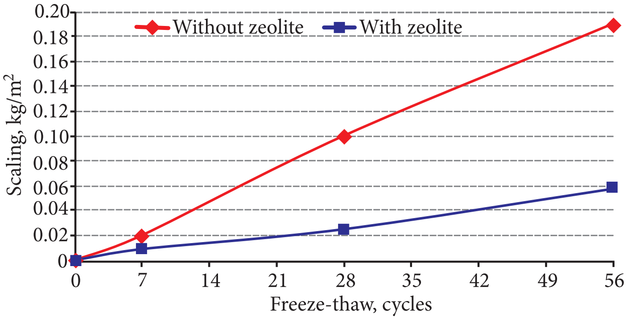

Fig. 10. Scaling of paving blocks after 56 freeze-thaw cycles according to *CEN/TS 12390-9* CDF method

splitting strength in blocks with zeolite admixture increases 6.3% compared with blocks without zeolite admixture.

Freeze-thaw and de-icing resistance test was done according to *EN 1338/AC Annex D* after 28 days of curing. The results are presented in Fig. 9.

The most widely used 3% NaCl solution was used as de-icing agent for the freeze-thaw resistance test according to *EN 1338/AC Annex D* method. The most effective de-icing occurs at not lower than – 10 °C temperature. At lower temperatures $CaCl_2$ is used. Fig. 9 illustrates that after 7 freeze-thaw cycles the scaling of the top layer of paving blocks increases more intensively until 28 freeze-thaw cycles. After 14 cycles the scaling of paving blocks without zeolite admixture is 0.05 kg/m^2 and 0.015 kg/m^2 in blocks with zeolite admixture. After 21 and 28 freeze-thaw cycles the difference in weight loss increases up to 4 times. After 56 cycles weight loss is only 0.05 kg/m^2 in concrete paving blocks with zeolite admixture in the top layer, whereas the blocks without zeolite admixture demonstrate the weight loss of 0.15 kg/m^2. The test results have proved that zeolite admixture significant increases the freeze-thaw and de-icing resistance of concrete paving blocks.

Freeze-thaw resistance of the paving blocks was also determined according to *CEN/TS 12390-9* Standard CDF method. Like in *EN 1338/AC Annex D* the freezing agent was used 3% NaCl solution. In this test the scaling was measured after 7, 28 and 56 freeze-thaw cycles.

As seen from Fig. 10, the scaling trends are similar as in the test done according to *EN 1338/AC*. The scaling of paving blocks without zeolite admixture steadily increases after 7, 28 and 56 cycles. The scaling of blocks with zeolite admixture is much lower. A significant difference (four times) between the scaling in blocks with and without zeolite admixture is observed after 28 freeze-thaw cycles, i.e. the scaling of blocks without zeolite admixture is 0.1 kg/m^2 and 0.025 kg/m^2 of blocks with zeolite admixture. After 56 freeze-thaw cycles the scaling differed 3.3 times.

The recorded scaling values in the tests done according to *EN 1338/AC* and CDF method of *CEN/TS 12390-9* standard are similar: a slight difference in scaling value after 7 cycles and a steadily increasing difference that reaches 4 times after 28 freeze-thaw cycles. After 56 freeze-thaw cycles the scaling measured according to *EN 1338/AC* Standard is 0.15 kg/m^2 in blocks without zeolite admixture and 0.05 kg/m^2 in blocks with zeolite admixture The scaling measured according to CDF method of *CEN/TS 12390-9* Standard is 0.19 kg/m^2 in blocks without zeolite admixture and 0.06 kg/m^2 in blocks with zeolite admixture.

Detailed tests with concrete paving blocks containing synthetic zeolite admixture in the top layer showed that zeolite admixture reduces water absorption, increases tensile splitting strength, abrasion resistance and reduces the surface scaling 4 times after 28 freeze-thaw cycles with 3% NaCl solution used as de-icing agent. It can be stated that synthetic zeolite admixture obtained from aluminium fluoride production waste and used in the top layer of concrete paving blocks increases the durability and lifetime of the blocks.

5. Conclusions

1. The density test with concrete paving blocks showed that zeolite admixture slightly decreases the density of the blocks.

2. After 28 days of curing the water absorption in paving blocks containing 5% of zeolite in the top layer was 5.5% lower compared with the blocks without zeolite admixture.

3. The comparison of the grooves formed in abrasion resistance test after 7 and 28 days showed that after 28 days the groove in the blocks without zeolite admixture narrowed 1.24 mm compared to the groove after 7 days, whereas the groove in the blocks containing zeolite admixture narrowed 2.22 mm after 28 days.

4. The tensile splitting strength of paving blocks with zeolite admixture increased 5.4% after 7 days of curing and 6.3% after 28 days of curing compared to the blocks without zeolite admixture.

5. Freeze-thaw resistance tests revealed that after 28 freeze-thaw cycles the surface scaling of blocks with zeolite admixture was 4 times lower than in blocks without zeolite admixture.

6. The tests results lead to the conclusion that synthetic zeolite obtained from aluminium fluoride production waste can be used as an admixture to increase the durability of concrete pavement elements (paving blocks, tiles, curbs).

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