

## Investigation of Shale Ashes Influences on the Self-Compacting Concrete Properties

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This work presents study of the influence of shale ashes on the cement paste hydration processes, cement stone and self-compacting concrete properties, when cement was replaced by shale ashes (from 5 % to 100 %). It was estimated, that replacement of 30 % of cement mass by shale ashes does not change water demand to obtain the normal consistence of cement paste and the replacement of 15 % of the cement mass even reduced the water demand. The results show that the compressive strength of the cement stone increases up to 4 % and the bending strength increases up to 26 % when 30 % of cement is replaced by shale ashes. During pozzolanic reaction they bind with free  $\text{Ca}(\text{OH})_2$  form and compose new calcium hydro silicates.

When admixture of shale ashes is used in self-compacting concrete production, segregation and bleeding decreases. The experiment showed that in the initial period (up to 3 days) shale ashes slightly retard the hardening of concrete. However, after 28 days the strength of concrete stone was higher compared to concrete without ashes and this strength increased with time. As shale ashes are 1.6 times finer than Portland cement, it improves concrete structure, increases the compressive strength, density and modulus of elasticity, and decreases shrinking strains. The comparison of the same design class concrete showed that compressive strength and static modulus of elasticity of concrete increases by about 15 % when 14.5 % cement was replaced with shale ashes.

*Keywords:* self-compacting concrete, fly ashes, shale ashes, spread, segregation, shrinkage.

### 1. INTRODUCTION

Self-compacting concrete (SCC) is a new category of high-performance concrete characterized by its ability to spread into place under its own weight without the need of vibration, and self-compact without any segregation and blocking [1]. As the use of SCC in the world markets increases, investigation of durability and production of this concrete becomes of great interest. Recently quality requirements for concrete products produced under conditions of production supervision have become much more stringent. Not only rapid improvement of strength and durability properties is required, but excellent surface finish quality as well [2]. Mineral admixtures (e. g. fly ashes, silica fume) and chemical additives (superplasticizer) are required for the production of concrete with excellent operating properties [3]. If hydration process of Portland cement concrete typically ends after 28 days, the use of pozzolanic admixtures extends hydration reaction. Extended reaction produces good structure of micropores, significantly increases the density and, consequently, results in improved durability [4, 5]. The use of both fly ashes and  $\text{SiO}_2$  silica fume enhance and optimize concrete properties. Both types of admixtures “bind” portlandit, which is the weak element of concrete structure, especially, than having in mind the chemical impact. Portlandit is “bound” and turns into less soluble calcium hydrosilicate (C-S-H). When fly ashes with active  $\text{SiO}_2$  content > 40 % are used, the quantity of cement in concrete may be reduced even more.

Experiments of different researchers confirm their use fullness [6 – 8] because proper use of micro aggregates enables to enhance the density of newly-formed mixtures, concrete or mortar strength, reduce water absorption or permeability and decrease shrinkage strains of concrete, improve resistance to freezing and optimize operating characteristics of materials [9, 10]. All these features guarantee both technological and economic advantages.

Shale ashes in Estonia, as well as phosphoric gypsum in Lithuania, form a big amount of environment polluting waste. Ashes have different chemical and phase composition depending on the type of fuel, combustion conditions and other factors. This waste contains unburned fuel, which deteriorates characteristics of ashes used as active admixture [11, 12]. Shale ashes usually contain more CaO than coal ashes. The content of calcium oxides ranges from 10 % to 40 %. When ashes contain such a high amount of CaO, the latter is usually over-burned and thus may cause irregular change in the volume of binding material [13, 14]. Shale ashes and some other types of ashes may contain calcium sulphate (up to 10 % – 15 %  $\text{SO}_3$ ). Calcium sulphate may form ettringite, which causes damaging deformations and may disturb the structure of concrete containing those binding materials. Estonian shale ashes are known for hydraulic characteristics as it contains 8 % – 15 % of low-base calcium silicates and aluminates and about 25 % – 35 % of glass phase. One of the negative characteristics of these ashes is higher alkali content, which reaches 4 % when translated into proportion of  $\text{Na}_2\text{O} + 0.658 \text{K}_2\text{O}$ . Results of cement stone containing shale ashes research conducted by different authors showed that specimens produced only using shale ashes

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crumbled after a few days. Breaking was caused by deformations resulting from hydration of over-burned CaO and anhydrite and formation and growth of Ca(OH)<sub>2</sub> and etringite crystals in the voids of hardened binding stone [15, 16].

The main objective of this study was to determine possibilities of using shale ashes in SCC production and make experimental evaluation of the characteristics of fresh and hardened concrete.

## 2. MATERIALS AND EXPERIMENTAL METHODS

Materials used for tests: Portland cement CEM I 42.5R, produced by the JSC "Akmenēs cementas", gravel fraction 2/8 and crushed gravel fraction 8/11, 11/16 produced by the JSC "Žvyro karjeras", sand of 0/4 fraction from the JSC "Rizgonių karjeras". Concrete admixtures produced in Germany's company "MC Bauchemie": superplasticizer "Muraplast FK 63.30" made on the basis of polycarboxylic resin and the stabilizer of the mix "Centrament 525". Shale ashes were collected from electrical power station "OÜ Omlox Export" (Estonia) and coal ashes collected in "Balux" firm's (Poland, Bialystok) thermoelectric power station.

X-ray diffraction measurements of cement stone with shale ashes specimens were made by device diffractometer "DRON-2", Cu anticathode, Ni filter, Voltage between the ends of X-ray tubes – 30 kV, current of anode ~8 mA, width of cranny diaphragm from 1.0 mm to 0.01 mm.

The specifics surface of the shale and coal ashes was measured applying Blain's method and was found to be 570 m<sup>2</sup>/kg and 450 m<sup>2</sup>/kg respectively.

The specific surface of the Portland cement – 360 m<sup>2</sup>/kg. The bulk density of the shale ashes – 900 kg/m<sup>3</sup> and coal ashes – 700 kg/m<sup>3</sup>, particle density of those materials are 2800 kg/m<sup>3</sup> and 2400 kg/m<sup>3</sup> respectively. The bulk density of the Portland cement was established 1060 kg/m<sup>3</sup>, particle density – 3100 kg/m<sup>3</sup>. Chemical constituents of the shale ashes was determined: SiO<sub>2</sub> – 31.5 %; Al<sub>2</sub>O<sub>3</sub> – 8.5 %; CaO – 33.4 %; Fe<sub>2</sub>O<sub>3</sub> – 4.3 %; MgO – 4.8 %; Na<sub>2</sub>O – 0.3 %; K<sub>2</sub>O – 4.8 %; SO<sub>3</sub> – 8.3 %; Na<sub>2</sub>O<sub>(eq)</sub> – 3.5 %.

The experimental studies were carried out in the following succession: the part of the cement was replaced with fly ashes (from 5 % to 100 %) and with forced mixing mixer "Automix 65-L0006/A" prepared cement pastes of normal consistence were made. Research was accomplished by LST EN 196-1:2005 standard. Samples formed were (4 × 4 × 16) cm size prisms.

Sample prisms (4 × 4 × 16 cm) were made and compacted using vibration (vibration rate – 50 Hz, amplitude – 0.4 mm). The prisms were cured in a chamber (temperature 20 °C ± 2 °C, relative humidity 95 %) for 28 days or in a steam chamber for 8 hours at 60 °C after demoulding. Then compressive strength and flexure, also water absorption kinetics of the cement stone with and without fly ashes admixtures was determined.

Self-compacting concrete mixes were prepared in forced mixing laboratory mixer, replacing 14.5 % – 78.6 % of cement with shale ashes. SCC mixture spread was determined using the standard Abrams cone, flow spread

and flow time (up to 500 mm). The stability of the mix sedimentation was measured applying the "Three cylinder" method. In order to secure a good flow down the wall, immediately after the mixing the concrete mix was poured into a three part cylinder that was oblique at an angle of 45°. The concrete mix from the upper and lower parts of the cylinder was washed through the 8 mm sieve in 30 minutes, because the diameter of the largest particles of the used aggregates was 16 mm. The part of the mass of the aggregate that was left on the sieve cannot differ more than 15 %, then a concrete mix does not exceed the limit if permissible segregation.

Further on, standard cubes were prepared – (10 × 10 × 10) cm and the prisms of which were (7 × 7 × 21) cm, (10 × 10 × 30) cm. For 28 days the specimens were hardened in the regime chamber at the temperature of 20 °C ± 2 °C and at the relative humidity of 95 % or in steam chamber for 8 hours at 60 °C. Using an automated "Toni Technik" testing machine the following characteristics of the samples were determined: compressive strength, modulus of elasticity, dependence of concrete strain deformations and stress (up destruction). Relative shrinkage strains were measured by optical ruler IZM-10M (with 0.001 mm scale resolution).

## 3. RESULTS AND DISCUSSION

### 3.1. The influence of shale ashes on the cement paste and cement stone properties

To determine the influence of ashes on the characteristics of cement paste and cement stone the following experiments were carried out where a certain part of cement was replaced by fly ash (from 5 % to 100 %): water demand to obtain cement paste of required consistency, cement stone strength, density and water absorption kinetics. Specimens were tested after 2 and 28 days setting in normal curing mode chamber. Some specimens were thermally treated in order to accelerate cement hydration and evaluate the impact of active SiO<sub>2</sub> present in the ash on concrete characteristics at higher than room temperature. Before steam-curing specimens were kept for 24 hours, then temperature was raised up to 60 °C during 2 hours and the specimens were thermally treated for 8 hours. Data of the experiment are presented in Figs. 1 – 3. Cement stone porosity parameters were determined according to water absorption kinetics and are presented in Table 1.

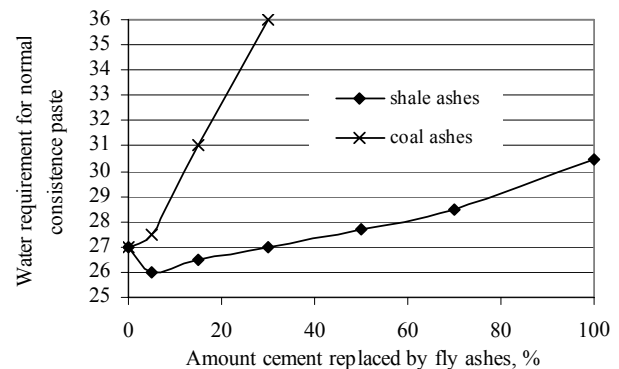
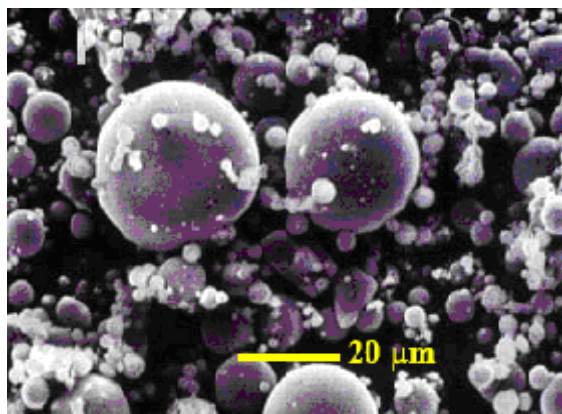
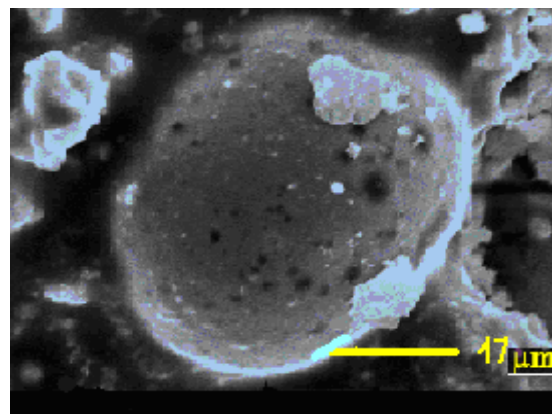


Fig. 1. Dependence on water requirement from amount of cement replaced by fly ashes



a



b

**Fig. 2.** Particle shapes and sizes of shale (a) and coal ashes (b) [17]

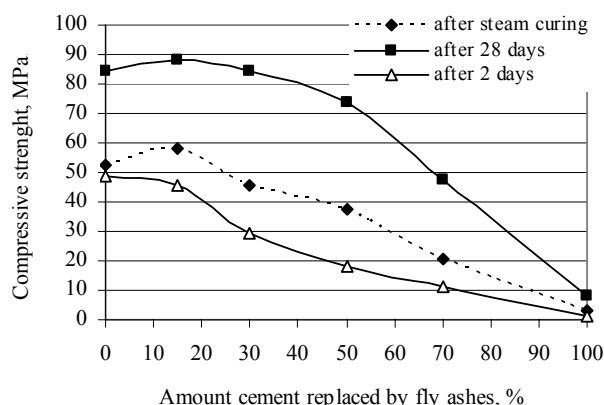
Experimental data of comparing the cement paste made with and without fly ash have revealed that due to larger specific surface area fly ash has an impact on water demand when cement paste of normal consistency is produced. As coal ashes particle size are similar to that of cement, only small quantity of coal ashes are recommended (up to 10 % of cement mass). When bigger quantities are used, the coal ashes act more like cement dilutor instead of improving the overall granulometric composition of cement-ashes mix. This observation was proved by significantly higher water demand for cement paste of normal consistency (Fig. 1). Much better results were obtained by using shale ashes. When up to 30 % of cement mass is replaced by the shale ashes, there is practically no change in water demand for the cement paste of normal consistency, and when 15 % of cement mass is replaced, water demand even goes down (Fig. 1). This can be explained by improved granulometric composition of the cement when 30 % shale ashes are added and less voids in the paste although the density of cement stone is lower. This can be explained by different densities of added ash and cement. Water demand remains constant also because minor particles of the shale ashes are less porous and have a glassier round or spherical form. Such particles distribute more evenly in the paste and in some cases water demand is lower even without using the superplasticizer (Fig. 2). This is one of the reasons why the compressive strength of cement stone increases [17].

Later water demand gradually increases with bigger ashes content. Explanation to this is overfilling of voids around cement particles with bigger amount of ashes. Therefore the void overfilling rate increases along with the specific surface area of the cement and dispersive admixture as well as porosity of the paste and all that result in higher water demand for normal consistency paste.

Shale ashes admixture has no strong influence on cement stone pores properties, but improves closed porosity and reduces pores (Table 1), which has positive influence on cement stone durability.

Binding and hardening of shale ashes are determined by the same physical and chemical processes as those present in the hardening of latent hydraulic materials. When shale ashes are used, hydration reaction of calcium silicates, aluminates and ferrites also takes place during setting. As hydraulic activity of these ashes is very low, the

binding and stiffening of these binding materials is very slow in the initial period (up to 3 days), however later the compressive strength increases much more compared with cement stone without ashes admixtures (Fig. 3). The setting intensity increases significantly when concrete products undergo hydrothermal treatment, e.g. steam curing. Results of compressive and bending strength after 2, 28 days setting and after steam curing are presented in Figs. 3 – 4.



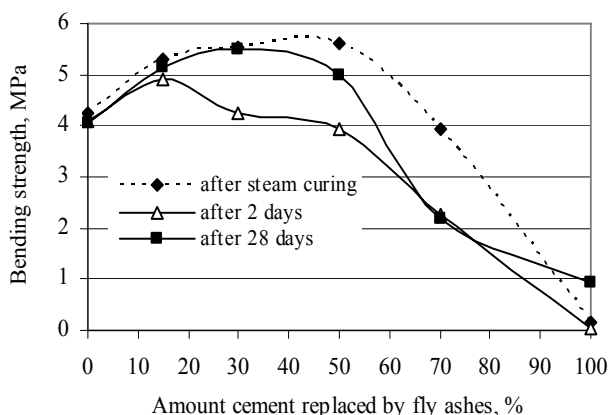
**Fig. 3.** Dependence on cement stone compressive strength from amount of added shale ashes

Experimental data show that the cement stone compressive strength increases by 4 % and bending strength increases up to 26 % when 30 % of cement is replaced by shale ashes. Further increase of ashes content is not feasible because granulometric composition of the mix deteriorates, strength characteristics of the specimens decline and the concrete may suffer from alkali corrosion in the future due to higher alkalinity of the ashes. Increased strength may be also explained by sufficiently big specific surface area of ashes (570 m<sup>2</sup>/kg) and the fact that about 30 % of active SiO<sub>2</sub> may take part in cement stone hardened process. During pozzolanic reaction they bind with free Ca(OH)<sub>2</sub> and form calcium hydrosilicates, which fill in the volume of capillary pores. Lower capillary porosity and higher relative density of newly-formed elements make the cement stone stronger and more durable. In ashes containing up to 15 % – 40 % of free CaO (34 % in our case) the latter is over-burned. This over-burned CaO may cause uneven change in the volume

**Table 1.** The variation of water demand and porosity of hardened cement stone, modified with shale ashes

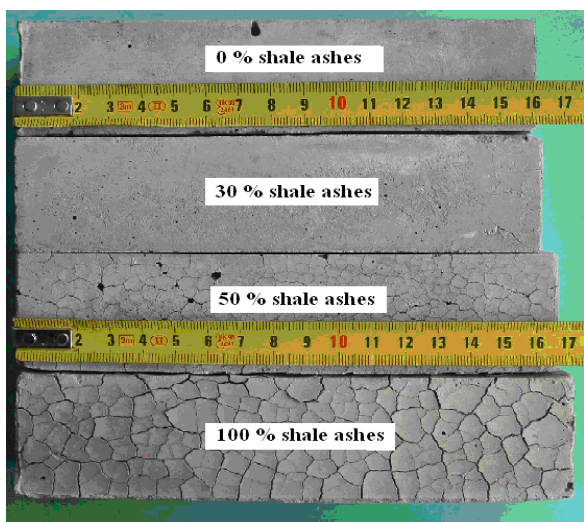
Properties of cement stone	Amount of cement replaced by shale ashes, %					
	0	15	30	50	70	100
Open capillary pores average rate, $\lambda$	2.41	2.31	1.96	2.29	2.50	2.75
Full porosity, %	30.88	31.87	35.74	38.10	40.53	50.86
Closed porosity, %	4.16	4.29	4.56	4.50	5.40	4.18
Open porosity, %	26.72	27.58	31.19	33.60	35.13	46.68
Water absorption, %	14.4/–	15.8/15.0*	18.04/17.3*	20.3/18.8*	22.0/20.0*	35.3/–

Note: \* – specimen were cured in steam chamber for 8 hours at 60 °C.



**Fig. 4.** Dependence on cement stone bending strength from amount of added shale ashes

of binding material, however, according to RILEM TC191 ARP recommendations [18], it must be experimentally verified. The presence of calcium sulphate (from 10 % to 15 % of  $\text{SO}_3$ ) in shale ashes and other ashes may lead to the formation of ettringite, which causes damaging expansion deformation and may destroy the binding material stone structure. Therefore the usability of these ashes must be evaluated. When more than 50 % of cement mass was replaced by shale ashes, specimens demonstrated the extension in length up to 5 % and significant surface cracking during setting (Fig. 5).

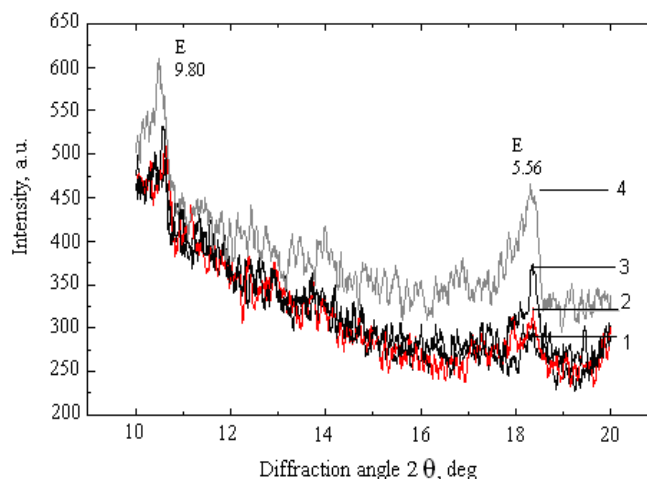


**Fig. 5.** Shale ashes influence on the change of specimens' volume after 28 days of hardening in regime chamber

In spite of the absence of high-base calcium aluminates, ettringite may still form from the solution saturated with calcium and  $\text{SO}_3$  ions. Specimen cracking can be explained by increased content of free water when cement of the specimens is replaced by bigger ratio of shale ashes. During the setting of specimens water from the surface layers evaporates quicker compared to diffusion of additional water from lower levels to the surface. Different drying of layers make causes specimen cracking.

### 3.1.1. The influence of shale ashes on cement stone hardened process

Shale ashes influence on cement stone hardened processes was analyzed by X-ray diffraction method. Four specimens of test materials were chosen (cement stone in which Portland cement was replaced by 15, 30 and 50 % of fly ashes and without ashes). Before the test materials were dried in 105 °C temperatures and sieved with sieve No. 008. X-ray diffraction patterns with ashes admixture and without ashes admixture are shown in Fig. 6.



**Fig. 6.** X-ray diffraction patterns of cement stone steamed at 60 °C. E – the peaks of ettringite. 1 – Specimen without shale ashes; 2 – specimen with 15 % of shale ashes; 3 – specimen with 30 % of shale ashes; 4 – specimen with 50 % of shale ashes

According to the obtained results we may state that qualitative composition of examined cement stone did not change with the change of shale ashes content and setting conditions. Similar new compounds are formed in all

**Table 2.** Relative amount of basic minerals and hardening conditions of cement stone

Amount of cement replaced by shale ashes, %	The amount of minerals in relative units					Hardening conditions
	Etringite, (~5.56)	Portlandit, (~4.91)	Amorphous compounds	C <sub>3</sub> S (~1.76)	SiO <sub>2</sub> (~3.35)	
0	9	95	11	35	0	Steam curing at 60 °C
15*	10	87	13	20	15	Steam curing at 60 °C
30	15	66	14	20	30	Steam curing at 60 °C
50	24	59	13	11	30	Steam curing at 60 °C
0	13	74	16	20	0	28-days of natural hardening
15*	18	93	17	17	19	28-days of natural hardening
30	22	88	17	10	30	28-days of natural hardening
50	28	52	16	7	34	28-days of natural hardening

Note: \* – the highest compressive strength of cement stone achieved under respective setting conditions.

cases. Data of quantitative assessment of the research results and the measurement of relative height of the nodes of respective compounds are presented in Fig. 6 and Table 2. According to these data we may state that with the increase of shale ashes content (from 0 % to 50 %) the amount of etringite and quartz minerals in cement stone increases and the amount of portlandite and tricalcium silicate reduces. The change in the amount of amorphous compounds is insignificant. This tendency of the formation of new compounds remains irrespectively of concrete setting conditions (28-days natural setting or steam curing at 60 °C). In both cases the highest compressive strength of cement stone was achieved when Portland cement was replaced by 15 % of shale ashes.

### 3.2. Shale ashes influence on the SCC properties

To prepare SCC mixtures for three different concrete classes (C30/37; C40/50 and C50/60) with respect to technological and economic requirements four different compositions of concrete mixtures with shale ashes content ranging from 0 % to 78.6 % were chosen (Table 3).

The spread of concrete mixes with shale ashes was very similar, namely 680 mm – 700 mm. The spread of control specimen without shale ashes was 640 mm. The experiment demonstrated that shale ashes had almost no

effect on water demand to obtain concrete mix of the same consistency. This can be explained that shale ashes particles contain between the particles of sand and coarse aggregates and therefore the concrete composition is improved and density of concrete is increased, however the impact on air content in concrete mix was very small. Taking into consideration self compacting characteristic of concrete we may see that it retains a very small amount of air, only about 2.5 % – 4.0 %. This factor may negatively affect concrete durability. To avoid this negative effect we recommend to air entraining agents. Concrete mixtures with ashes didn't demonstrate bleeding, while segregation rate met the requirement of below 15 %, although cement consumption was respectively lower (Table 4). The analysis of SCC mixtures research data has shown that shale ashes reduce segregation of concrete mix and improve workability. We determined that polycarboxylate superplasticizer "Muraplast FK63.30" added at 1.5 % – 2.6 % of the cement mass would reduce water/cement ratio and enable to achieve the required spread for natural thickening of concrete mix. Stabilizing agent "Centrament 525" added at 0.4 % – 0.6 % of the cement mass would reduce segregation and bleeding of SCC mix, while higher content of this agent significantly increases the amount of entrained air and reduces concrete strength. As shale ashes are 1.6 times finer than cement, it improves the composition

**Table 3.** Designed concrete mix composition

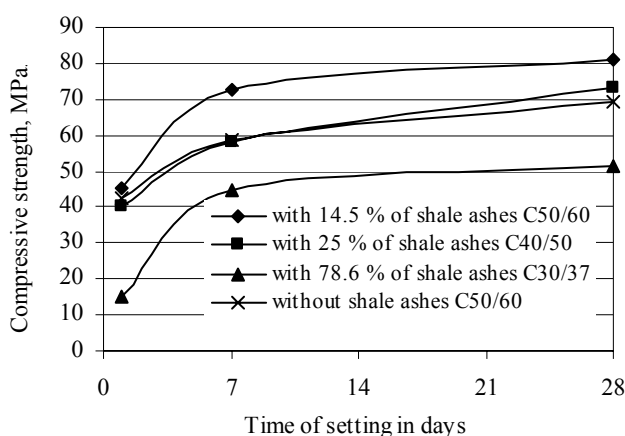
Mix No.	Relative amounts of materials			Amount of admixtures in % of cement mass		
	Amount of cement in % of dry materials mass	Relative of small and large aggregate	W/(C + S · 0.4)	Shale ashes	Superplasticizer	Stabilizer
1	20.7	1.03	0.32	14.5	1.59	0.39
2	17.1	1.62	0.35	25.0	1.64	0.58
3	13.6	1.03	0.39	78.6	2.63	0.66
4*	21.2	1.03	0.32	–	1.50	0.50

Note: 1 – designed concrete class C50/60; 2 – designed concrete class C40/50; 3 – designed concrete class C30/37; 4\* – designed concrete class C50/60 without shale ashes. S – Shale ashes.

**Table 4.** The properties of self compacting concrete mix with shale ashes

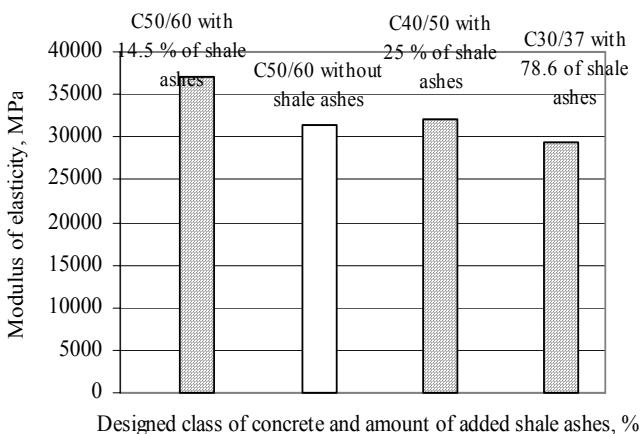
Mix No.	Properties of concrete mix						Slump flow, spread of the mix, mm
	Density, kg/m <sup>3</sup>	Time of spread, s		Air content, %	Index of segregation, %	Temperature, °C	
		to 500 mm	to final				
1	2410	5.57	36.60	2.50	7.20	17	700
2	2440	4.15	32.56	3.20	9.75	23	680
3	2449	6.15	36.99	2.50	10.90	23	700
4*	2400	6.85	32.50	4.00	9.15	23	640

of concrete mixture, increases compressive strength, density and elasticity module and also reduces contraction deformations (Figs. 7 – 9). The comparison of the same design class concretes showed that compressive strength and static elasticity module increases by 15 % when 14.5 % of cement is replaced by shale ashes.



**Fig. 7.** Dependence on concrete with shale ashes compressive strength from setting time

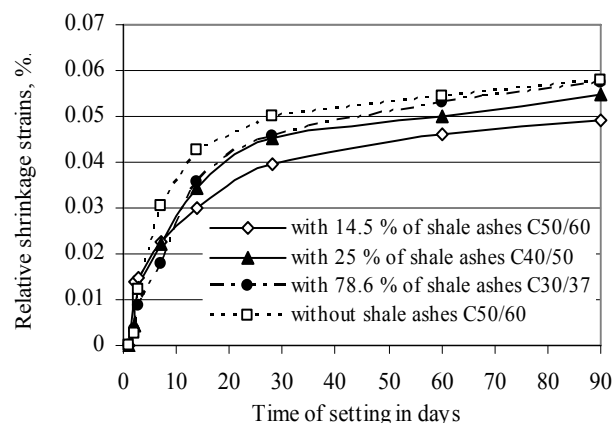
When bigger amount of cement is replaced by ashes in the design of low grade concrete, the compressive strength and modulus of elasticity remain at notably high levels and are 52 MPa and 30000 MPa respectively for C30/37 grade.



**Fig. 8.** Dependence on concrete modulus of elasticity from amount of added fly ashes and from designed class of concrete

Relative shrinkage strains were determined in hardened concrete by comparing different grades of

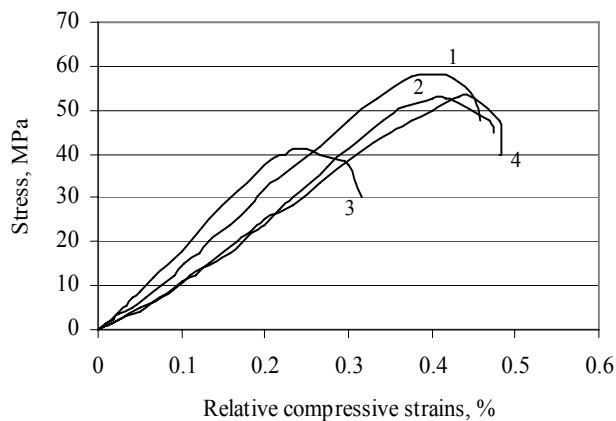
concrete and the amount of ashes in the mixture. Shrinkage strains were measured after 1, 2, 3, 7, 14, 28, 60 and 90 days hardening (Fig. 9). As concrete elements contract evenly in all directions, these deformations can be called volumetric shrinkage strains [19]. They notably reduce down to 16 % when cement is replaced by 14.5 % of shale ashes in concrete of the same composition (Fig. 9).



**Fig. 9.** Effect on concrete shrinkage from amount of added shale ashes

One of the reasons is positively modified overall granulometric composition of concrete mixture; another reason is the ability of ashes particles to encapsulate with jelly structure of newly formed compounds during the hardening of cement; they absorb the energy of compression force during the setting of the system and thus resist to the contraction of the whole system [19]. When bigger portion of cement up to 78.6 % was replaced by ashes, contraction deformations reduced insignificantly, however they were lower compared to the control mixture No. 4, where no ashes were used.

The dependence of deformations on tensile stress caused by short-term accidental load was determined by testing reference concrete prisms with dimensions of (10 × 10 × 30) cm. Deformation at the moment of force growing is in direct proportion with the force increment and is called resilience; when the force does not grow but is applied for a certain period of time, deformation depends on the duration of force application and tensile stress amount and is called plastic deformation [18]. Determination of these strains revealed that resilience of hardened concrete (design class C50/60), where cement was replaced by 14.5 % of shale ashes, increased (Fig. 10), i. e. under higher stress concrete retains elasticity characteristics and returns back to the initial state after the load is



**Fig. 10.** Effect of concrete relative strain deformations on amount of cement replaced by shale ashes. 1 – C50/60 concrete class with 14.5 % of shale ashes from cement mass; 2 – C40/50 concrete class with 25 % of shale ashes from cement mass; 3 – C30/37 concrete class with 78.6 % of shale ashes from cement mass; 4 – C50/60 concrete class without shale ashes

removed. We can also note that when cement is replaced by shale ashes higher plastic deformations are obtained. This was characteristic of all design classes of concrete. That means that concrete with this admixture will remain unchanged under growing stress for a longer period of time (Fig. 10).

#### 4. CONCLUSIONS

1. Shale ashes reduced segregation of SCC mix and improved its workability.
2. The test results prove that replacement of cement by up to 30 % of shale ashes partially improves granulometric composition of cement, reduces porosity of the mixture, and increases compressive strength of cement stone up to 4 % and bending strength up to 26 %, although the density of cement stone slightly diminishes.
3. When more than 50 % cement mass is replaced by shale ashes, during cement stone hardening specimens showed the extension in length up to 5 % and significant surface cracking. However these negative phenomena were not observed in hardening concrete even with higher ashes content.
4. When 15 % of cement was replaced by shale ashes, the concrete with compressive strength above 80 MPa was obtained with the characteristics complying with high compressive strength of SCC class C 60/75.

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