



DOI: [10.71167/uaceg.2026.590118](https://doi.org/10.71167/uaceg.2026.590118)

Received: 15.10.2025

Accepted: 12.01.2026

## SHRINKAGE INVESTIGATION OF SUSTAINABLE CONCRETE WITH DIFFERENT BINDERS

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*Keywords: concrete shrinkage, sustainable binders, pozzolana*

### ABSTRACT

Concrete is the most used building material in the world, with production increasing year on year. It plays a significant role in modern construction due to its versatility in shaping, durability, and cost-effectiveness. However, the production of cement used for concrete is highly polluting due to the large amount of carbon dioxide emitted into the atmosphere. The aim is therefore to use sustainable concrete, for which the cement used is supplemented with a certain amount of additives or substitutes to reduce the clinker content. One such additive is the natural local pozzolana – opoka. Such concrete with opoka cement has the property of having higher shrinkage deformations at an early age, leading to cracks and fissures. To overcome this problem, chemical admixtures or other additives are used in concrete production to reduce the shrinkage of the concrete. The aim of the research is to investigate the effect of concretes with different binders (control CEM I 42,5 R, CEM II/A-P 52,5N, CEM II/B-M (P-LL) 42,5N, CEM II/A-LL 42,5 R) on the properties of fresh and hardened concrete. The focus is on the study of early and drying concrete shrinkage. After performing a shrinkage test, it was found that early shrinkage (till 1 day) concretes with CEM II/B-M(P-LL) 42,5N and CEM II/A-P 52,5N type binders reached more shrinkage than the control concrete, respectively. Also, within the first 24 hours after casting the samples, it was observed that both samples with pozzolans in their binder composition after approximately 6 hours have a shrinkage curve jump, i.e. a sudden increase in

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shrinkage. This happens probably due to natural puzzolana porosity which after some period released free water. After 14 days of hardening, it was found that concretes with CEM II/B-M (P-LL) 42,5N and CEM II/A-P 52,5N type binders reached 34 % and 5 % more shrinkage than the control concrete, respectively.

## 1. Introduction

Concrete is an artificial stone that is obtained by hardening a mixture of a binding material (cement), aggregates (fine and coarse sand and gravel) and water. Concrete is used more than any other artificial material in the world [1]. However, the problem is that the production of cement used for concrete contributes significantly to the increase in CO<sub>2</sub> emissions, which contributes to global warming. In order to combat climate change, the European Parliament adopted the European Climate Act, which sets the EU's goal of reducing emissions by 55 % by 2030 and achieving climate neutrality by 2050 [10]. The cement industry accounts for about 5 – 8 % of the world's CO<sub>2</sub> emissions [2, 3]. CO<sub>2</sub> is released when limestone is burned and CaCO<sub>3</sub> decomposes into CaO and CO<sub>2</sub>. Also, during combustion, extremely high temperatures are required, reaching 1450 °C, so fossil fuels are burned, which also emit CO<sub>2</sub> during combustion. The amount of CO<sub>2</sub> emitted by the cement industry can be compared with other CO<sub>2</sub> sources in the world (see [4]). Roughly speaking, 1 ton of clinker produced is equal to 1 ton of CO<sub>2</sub> emitted into the environment if fossil fuels are used for its production and no measures are applied to reduce emissions [2].

As the demand for concrete and reinforced concrete structures does not decrease, due to their diverse uses and low price, and as the whole world moves towards sustainability, various efforts are being made to reduce CO<sub>2</sub> emissions. Several strategies can be found in the literature to do this [5]:

- replacing part of the Portland cement clinker with other materials that leave a lower or no CO<sub>2</sub> footprint, otherwise known as supplementary cementing materials (PCM);
- replacing the coal used for clinker combustion with waste with a calorific value equivalent to that of fossil fuels;
- capturing and storing CO<sub>2</sub> during cement production (in the kiln);
- clinker-free alternative cements;
- capturing CO<sub>2</sub> without releasing it into the atmosphere;
- resource-efficient design;
- recycling or reusing concrete.

Over the past few decades, PCM has been widely used in concrete production. Most of the reasons for using PCM have been economic, for example to reduce the cost of concrete production and, at the same time, the cost of construction [6]. However, a more important aspect of using PCM is the sustainability of concrete, as the use of more PCM is expected to significantly reduce CO<sub>2</sub> emissions and thus mitigate the negative environmental impact of concrete production. PCM is currently one of the main tools to achieve this [7].

### Supplemented cementing materials

Additional cementing materials (SCM) – materials that can replace part of the portland cement clinker in cement, are usually divided into natural and artificial by origin.

Materials of natural origin – these are naturally formed materials, divided into volcanic and sedimentary by the way of formation. Volcanic materials can be unaltered or altered pyroclastic materials, glassy pumice and ash, tuffs. Examples of volcanic materials – volcanic ash, tuff, pumice, trass. Volcanic ash and pumice contain about 50 – 80 % active amorphous silica and alumina. All volcanic materials (except feldspars) are characterized by extremely high pozzolanic activity (pozzolans are materials of volcanic and sedimentary origin). Sedimentary rocks have a much greater chemical diversity. The main chemical component of all sedimentary rocks is opal –  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ , which is 50 – 80 %. Sedimentary rocks are divided into chemical, clastic, naturally burnt clays, diatomaceous earth, hydrothermal siliceous tuff and other rocks. Examples of sedimentary rocks are opal, shale, diatomite, gley.

Materials of artificial origin are waste from other industries, such as blast furnace slag, coal fly ash,  $\text{SiO}_2$  microdust, microsilica, calcined clay, metakaolin. Ash of organic materials is also classified as artificial PCM. Organic waste is burned, and ash is obtained from it, in which silica is concentrated. Most often, agricultural waste is burned. According to their activity, they are divided into low and high activity materials.

### Opoka

Opoka is a sedimentary siliceous rock of natural origin, consisting mainly of fine opal grains (up to 98 %), as well as other impurities – clastic quartz, feldspars, glauconite, clay particles, and skeletal remains of organisms. Opal mainly contains calcium carbonate (depending on the period of sedimentary rock formation, 25 – 48 %) and silicon dioxide (5 – 62 %) [8]. The structure is fine-grained, the texture is solid or layered. The color ranges from light gray to dark gray, almost black.

Opoka was formed from the remains of marine organisms, whose silicon skeletons accumulated on the bottom of the basin and changed diagenetically, or from solutions when silica was deposited. It is found in deposits of the Cretaceous and Paleogene systems.

Due to its chemical composition, opoka is used as a clinker substitute in the cement industry or as an additive to cement. There are cases where opoka is also used for facades due to its appearance and good thermal insulation properties [9]. Examples of opoka are presented in Figure 1.



**Figure 1**

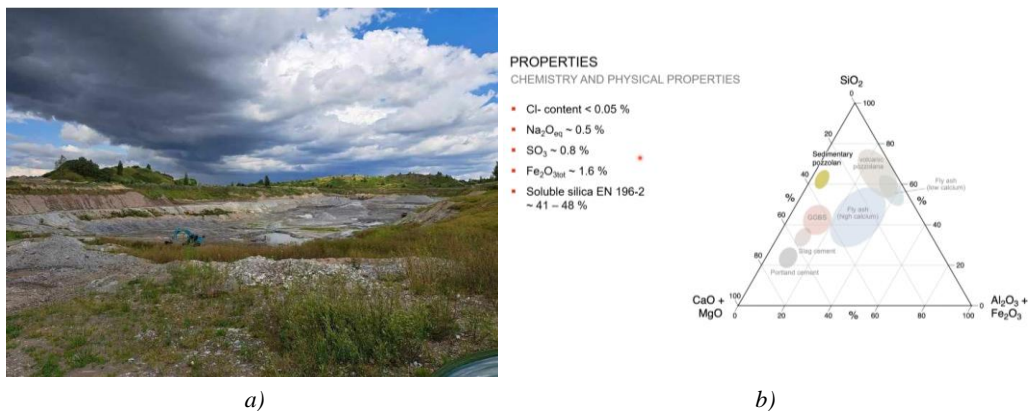
*a) Opoka in Lithuania [10] and b) shell impression in opoka [9]*

Opoka can be found in Lithuania, Poland, Russia, Germany, the Czech Republic, France and other countries [10 – 12]. The opoka deposits closest to the Earth's surface are in the Šilutė

district (Žemaitkiemis surroundings) and in the territories of Pagėgiai municipality (Stoniškės). The only deposit in Lithuania, Stoniškės-Žemaitkiemis, was discovered in 1957. Later, in 1989, it was put into operation, then its activities were suspended, but since 2022 it has been used by “Akmenės cementas” [13] (see Figure 2).

The total area of the deposit is 36,6 ha, the opoka layer is approximately 15 meters deep and its useful thickness is about 30 m. As far as is known, there are currently about 35 million tons of minerals, but there is a high probability that this deposit is even larger than previously thought. This deposit is characterized by instability of the opal composition – from pure opal to opal marl. This transition is not pronounced in the massif. The opal found in Lithuania is characterized by pozzolanic properties, as it contains a lot of opal [10]. The carbonate opal massif consists of the following petrographic varieties [14]:

- Variety I – dark gray, high-density opal with a characteristic shell fracture, contains the least carbonates (8 – 17 %) and the most silica (63 – 73 %);
- Variety II – light gray, siliceous (opalite) high-density marl,  $\text{CaCO}_3$  content – varies from 31 % to 66 %.  $\text{SiO}_2$  content varies from 25 % to 62 %;
- Type III – light gray, loose marl with carbonate opoka, the chemical composition is slightly different from type II.



**Figure 2**  
*a) Stoniškiai-Žemaitkiemis quarry and b) chemical and physical properties of opoka [14]*

The chemical composition of opoka mined in Lithuania compared to other components used for cement production is presented in Figure 2b. Opoka does not exceed the permissible content of substances in cement according to the LST EN 197-1 and LST EN 206-1 standards. The content of these materials in cement is limited due to their negative impact on concrete. Due to its good chemical composition, opoka is used in the cement industry to replace part of Portland cement clinker, thus making cement production more sustainable, and concrete also becoming more sustainable. There are two possible grades of cement with opoka: pozzolanic portland cement and composite portland cement.

### Concrete shrinkage – research

Ahmad [15] conducted a study where the influence of silica fume on the properties of concrete containing volcanic natural pozzolan was investigated. 3 concrete samples with

different cement compositions were tested: a control with 100 % Portland cement (OPC), a cement mixture containing 80 % Portland cement and 20 % natural pozzolan (OPC-NP), and a cement mixture containing 75 % Portland cement, 20 % natural pozzolan and 5 % silica fume (OPC-NP-SF). After measuring the drying shrinkage of these samples, it was found that 14 days after pouring the concrete, the shrinkage strain of all samples was almost the same (approximately 0,215 mm/m), and later an increase in the shrinkage strain of the OPC sample was visible together with the OPC-NP-SF, the deformation of which was slightly lower than that of the control concrete. The OPC-NP sample had the lowest shrinkage strain after 365 days. Ahmad [15] also measured the compressive strength of the samples. It was found that the sample with natural pozzolan and silica microdust had a higher strength, the strength of which exceeded the strength of the control sample. The lowest compressive strength was achieved by the sample with natural pozzolan, so a correlation is visible between the shrinkage and compressive strength of the samples when they contain natural pozzolanic materials. Silica microdust increases the compressive strength of concrete, while natural volcanic pozzolans reduce the shrinkage deformation of concrete.

Chaib [16] found that the shrinkage of concrete increases when 10 %, 20 % or 30 % volcanic pozzolanic cement is used in the mixture. The study also compared the effect of perlite (in the same amounts) on concrete shrinkage. It was found that in the long term, the shrinkage of pozzolanic concrete is higher than that of Portland cement concrete, and pozzolanic concrete also showed poorer properties in the first days of hydration compared to perlite concrete.

Leon-Miquel [17] conducted research and determined the influence of local resources of natural pozzolan of volcanic origin from Chile on the properties of concrete. A water-reducing admixture and polyvinyl alcohol (PVA) fibers were also used for all samples. The results of the research show that due to the low reactivity of natural pozzolan, high internal porosity and large particle size, a large amount (50 %) of this rock in cement can reduce the compressive (especially in early-age concrete) and tensile strength of concrete, as well as increase cracking and shrinkage.

Leon-Miquel [17] conducted studies with different amounts of pozzolans in cement and found that 30 % of natural pozzolan in cement was the most optimal. Concrete containing this amount had a smaller crack width after tensile testing, in contrast to samples with 10 % or 50 % pozzolans.

The use of another pozzolan, metakaolin, in concrete reduced its shrinkage over a period of 140 days and increased its compressive strength according to studies by Sinngu [18]. By adding 10 – 15 % metakaolin to cement, the drying shrinkage of concrete was significantly reduced – about 50 %. Drying shrinkage decreased accordingly with an increase in the amount of metakaolin in concrete mixtures. Concrete containing 10 – 15 % metakaolin showed an efficient hydration reaction and better sulfate resistance.

## **2. Research methodology and used materials**

The tests investigate the influence of different binders on fresh concrete mix and hardened concrete. The following sections describe the methodology of the tests performed. Four types of cement were used for the tests: CEM I 42,5 R, CEM II/A-P 52,5N, CEM II/B-M (P-LL) 42,5N, CEM II/A-LL 42,5 R. The characteristics of the cements as the mechanical, physical and chemical properties of the cements are presented in Table 1.

**Table 1. Mechanical, physical and chemical properties of each cement type**

	CEM I 42,5 R	CEM II/A-P 52,5N	CEM II/B-M (P-LL) 42,5N	CEM II/A-LL 42,5 R
Specific surface area, g/cm <sup>2</sup>	4440	5460	6720	5190
Specific density kg/m <sup>3</sup>	3150	2980	2960	3060
Compressive strength after 2 days, MPa	29 ± 2	30 ± 2	23 ± 3	29 ± 2
Compressive strength after 28 days, MPa	54 ± 3	57 ± 3	53 ± 3	54 ± 3
Setting time, min	185	150	185	160
Sulphate (SO <sub>3</sub> ) content, %	3,09	3,29	3,34	3,59
Chlorides (Cl <sup>-</sup> ), %	0,037	0,038	0,044	0,072
Alkali content, Na <sub>2</sub> O <sub>eq.</sub> , %	0,73	0,68	0,61	0,72

Dolomite with a fraction of 5/16 mm, from AB “Dolomitas”, was used as coarse aggregate. Sand with a fraction of 0/4 mm, from UAB “Rizgonys”, was used as a fine aggregate. The characteristics of the sand and the dolomite are presented in Table 2.

**Table 2. Characteristics of aggregates**

Aggregate	Fraction	Characteristics	Result
Sand	0/4	Particle density, kg/m <sup>3</sup>	2650
		Bulk density, kg/m <sup>3</sup>	1600
Dolomite	5/16	Particle density, kg/m <sup>3</sup>	2800
		Bulk density, kg/m <sup>3</sup>	1440

The superplasticizer “ViscoCrete D-790” manufactured by Sika was used for the research. Main chemical component – aqueous solution of modified polymers, density 1,08 ± 0,02 kg/l, pH-value 4,6 ± 1, Dosage 0,4 – 1,5 % of cement mass.

The used concrete mixes in the research are prescribed in the table below:

**Table 3. Concrete mix compositions, kg/m<sup>3</sup>**

Marking	CEM I	CEM II/A-LL	CEM II/A-P	CEM II/B-M
Cement	350	350	350	350
Fine agg	893	893	893	893
Coarse agg	1022	1022	1022	1022
Water	165	165	165	165
Superplasticizer	2,52	2,52	3,22	3,78

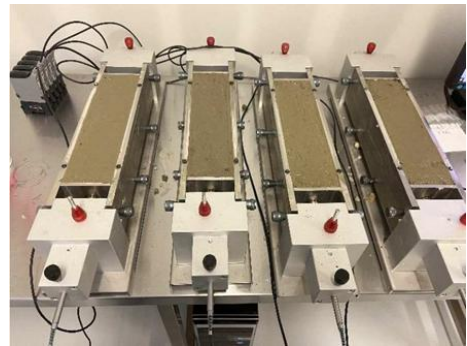
The concrete mixtures were prepared according to the methodology specified in the LST EN 12390-1:2021 standard. The formation of specimens was carried out according to the methodology specified in the LST EN 12390-2:2020 standard. A concrete mix slump test was performed according to the methodology presented in the LST EN 12350-2:2019 standard. A test to determine the density of the concrete mixture was performed according to the

methodology presented in the LST EN 12350-6:2009 standard. The influence of selected different binders on concrete shrinkage deformations was performed according to the methodology presented in the LST EN 12390-16:2019 standard. During the test, reference prisms were measured after 1, 3, 7 and 14 days, and the change in their length was calculated. Concrete compression testing was performed according to the methodology specified in the LST EN 12390-3:2019 standard.

The early and drying shrinkage test specimen for concrete used prisms of 75×75×285 mm. The sequence of mixing was done in accordance with the applicable provisions of practice ASTM C305 and always consistent (see Fig. 3). The concrete mix in the mold was placed in two approximately equal layers. Each layer was compacted with the tamper. The concrete mix was compacted carefully on the corners, around the gage studs, and along the surfaces of the mold with the tamper until a homogeneous specimen was obtained. After the top layer of the concrete mix had been compacted, the top of the mold was flushed, and the surface smoothed with a few strokes of a trowel, plexiglas plate. Immediately after completion of molding, the screw only at the front axe that holds the gage studs in position at the sensors end of the mold was loosened in order to prevent any restraint of the gage studs during initial shrinkage of the specimen. The sensors parameters: 5 mm displacement transducer measuring range, accuracy better than 0,1 μm, repeatability precision better than 0,05 μm, resolution better than 0,01 μm. The measuring process was started immediately after compaction and was measured with Swiss made software according to the scheme in Fig. 3d. During early shrinkage, measurement process test specimens were cured in the molds in a dry condition (RH 40 – 60 % and 20±2 °C).



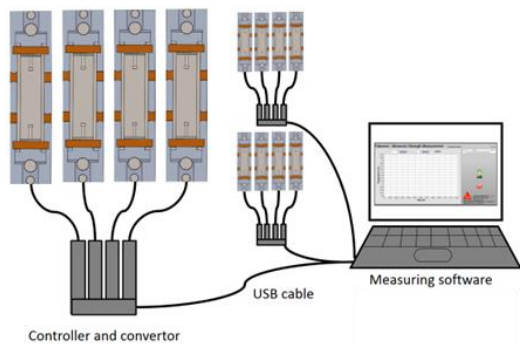
*a) sample preparation*



*b) filled samples*



*c) measurement process*



*d) software measuring scheme*

**Figure 3. Concrete early shrinkage measuring process**

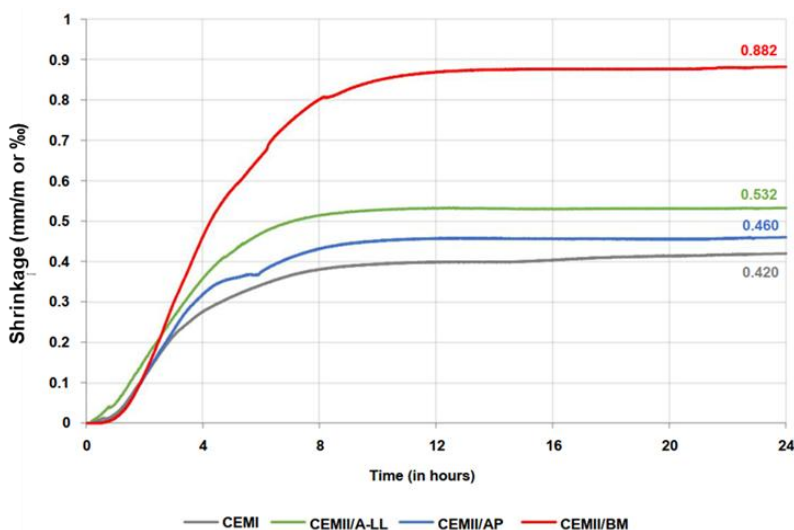
### 3. Test results

The slump and slump class, as well as the fresh concrete density, of each concrete mix were determined (see Table 4).

**Table 4. Fresh concrete properties**

Marking	CEM I	CEM II/A-LL	CEM II/A-P	CEM II/B-M
Superplasticizer dosage	0,7 % f.c.a.	0,7 % f.c.a.	0,88 % f.c.a. (+25 %)	1,05 % f.c.a. (+50 %)
Slump, mm	160	185	180	180
Slump class	S4	S4	S4	S4
Density, kg/m <sup>3</sup>	2485	2488	2453	2411

From Table 4 it can be seen that the superplasticizer dosage increases with the cement type blended composition. Compared to CEM I, CEM II/A-P requires +25 %, and CEM II/B-M +50 % more admixture to maintain the same workability (S4 class). This suggests lower efficiency of admixture with blended cements (especially CEM II/B-M). Despite different dosages, all mixtures reach S4 slump class, i.e., high workability (160 – 210 mm). It has been obtained that density decreases as more blended materials are used and it indicates higher porosity or lower clinker content in blended cements, which often results in lighter mixes.



**Figure 4. Early shrinkage during the first 24h**

Shrinkages are recorded with sensors, and a shrinkage graph is drawn up for each sample within the first 24 hours based on the data obtained. When analysing the results of early shrinkages (see Figure 4), it was observed that during the first day, the control sample shrank by 0,420 mm/m – this is the sample with the least shrinkage. Concrete with limestone filler (A-LL) shrank the fastest, i.e. its curve within the first 2 hours is the highest compared to other samples. After 24 hours, the sample had a shrinkage of 0,532 mm/m, which was 26,67 % higher than that of the control concrete. Concrete with pozzolans primary shrinkage is closest to shrinkage of the control concrete. After 24 hours, the sample had a shrinkage of 0,460 mm/m, which was 9,52 %

higher than the control concrete. Concrete with limestone and pozzolans showed the highest shrinkage. After 24 hours, the shrinkage of this concrete reached 0,882 mm/m, which exceeded the shrinkage of the control concrete by 2,1 times or 95,24 %. Also, it was obtained that fresh concrete mix with opoka Portland cement, due to opoka porosity, has a more pronounced and sharper shrinkage jump, which begins 6 hours after the start of concrete.

When analyzing the results of concrete shrinkage, it was observed that the average shrinkage of the control sample after 1 – 14 days is 0,303 mm/m (see Fig. 5). This group of samples shrank the least of all the tested groups. The group of samples with a binder containing pozzolana and limestone shrank the most, after 1 – 14 days the average shrinkage of these samples was 0,406 mm/m. The increase of Supplementary Cementitious Material in cement gives higher values of drying shrinkage.

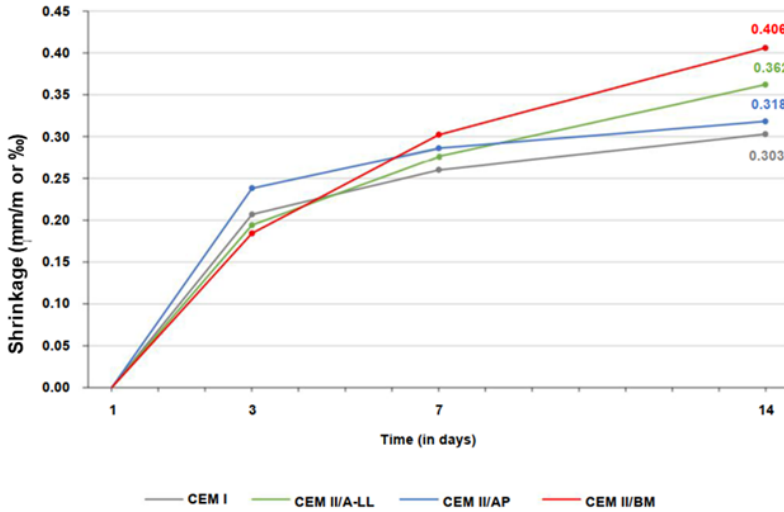


Figure 5. Drying shrinkage during 1 – 14 days

The highest compressive strength was achieved by the control sample, and the lowest strength after 7 and 28 days of hardening was achieved by the sample with the lowest Portland cement clinker content (see Fig. 6). This decrease in compressive strength is due to the additives in the binder – pozzolans and limestone. These additives, especially in the early period, reduce the strength of concrete compared to the control concrete.

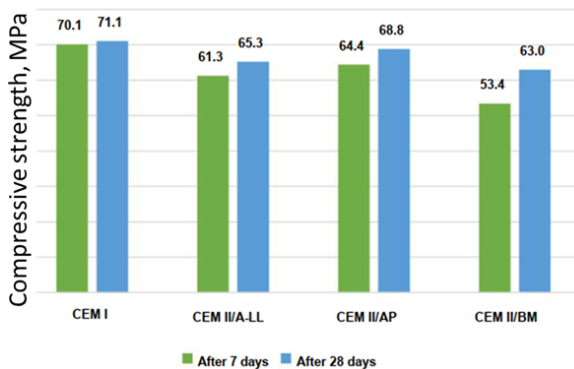


Figure 6. Compressive strength – after 7 and 28 days

## 4. Conclusion

The test results showed that all concrete compositions achieved the required S4 slip class due to the properly selected amount of superplasticizer. The amount of superplasticizer in concretes with CEM II/B-M (P-LL) 42,5N and CEM II/A-P 52,5N type binders was 1,5 and 1,25 times higher than in the control concrete, respectively.

After performing a shrinkage test within the first 24 hours after casting, it was observed that both samples containing pozzolans in their binder composition exhibited a sudden increase in shrinkage after approximately 6 hours, i.e., a noticeable jump in the shrinkage curve. This behavior is attributed to the high porosity of opoka.

Concrete with B type cement showed the highest shrinkage during the 1 – 14 days period (0,41 mm/m). The increase of Supplementary Cementitious Material in cement gives higher values of drying shrinkage.

It was found that concretes containing opoka in their binder do not reach high early compressive strength (after 7 days), but due to pozzolana reactivity after 28 days their compressive strength gain is greater than concrete with other cements.

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