

Effect of Coffee Grounds Addition on Efflorescence in Fly Ash-based Geopolymer

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Abstract. The article aim is to analyse the influence of addition of the cheap and environmental friendly material that can reduce – coffee grounds on the efflorescence effect. The additives should reduce this effect trough decreasing pH. The geopolymer matrix was prepared from fly ash from Skawina CHP plant and sand in ratio 1:1. The samples were prepared using sodium promoter - 12M sodium hydroxide solution combined with the sodium silicate solution and coffee grounds (1, 3 and 5% by mass of the composite). The samples were prepared according to the methodology described in the standard EN 12390-1. The empirical part of the research is based on: visual analyse for efflorescence, absorptivity test, pH test and compressive strength test. The results show the influence of coffee grounds on the pH reduction from about 11 to 9.21, the elimination of efflorescence and the reduction of the compressive strength.

1. Introduction

Nowadays, Portland cement is commonly used material in construction industry. This technology has a lot of disadvantages among others: high energy consumption, high level of CO₂ emissions and questionable durability. The most promising alternative is the technology of alkali-activation. The material is created by reaction between alkaline solutions and solid aluminosilicates. Unfortunately, the activation process required high pH [1,2]. The one of the limitation of wide using this materials is common problem with efflorescence caused by excess sodium oxide remaining unreacted [1] The unreacted sodium oxide creates white efflorescence on the surface – it is sodium carbonate heptahydrate Na₂CO₃ · 7H₂O. It can be the significant problem, because it decreases not only aesthetic value for the final products, but also mechanical properties of the material [3,4]. The research on additives shows that certain admixtures can eliminated or limited efflorescence effect, exemplary alumina-rich additives such as natural pozzolan or zeolites [4,5].

Coffee grounds are an organic waste from the food and beverage industry. There are usually destined for landfill [6,7]. They have barely acidic pH (reported usually about 5.0) [6,8].

2. Experimental procedure

Specimens based on geopolymer matrix and additive of coffee grounds in amount: 1 (samples marked as P1), 3 (P3) and 5% (P5) by mass were investigated. The variable includes different amount of reinforcement and reference samples based on matrix material without addiives (samples marked as P0).



2.1. Materials

2.1.1. Geopolymer matrix. The geopolymer matrix was made from fly ash from the CHP plant in Skawina (Poland) and sand in ratio 1:1. This kind of fly ash is rich in oxides such as SiO₂ and Al₂O₃. The oxides content is presented in table 1. The fly ash particles morphology was typical for such by-products (from coal combustion) and suitable for the process of alkali-activation.

Table 1. Chemical composition of the fly ash.

	SiO ₂	Al ₂ O ₃	Na ₂ O	CaO	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	TiO ₂	P ₂ O ₅	BaO
[% mass]	55.89	23.49	0.59	2.72	5.92	2.61	3.55	0.16	1.09	0.82	0.2

The process of alkali-activation has been made by 12M sodium hydroxide solution combined with the sodium silicate solution. The ratio of liquid parts to dry parts was 0.4 by mass.

2.1.2. Coffee grounds. The coffee grounds came from coffee brewed traditionally. There were mixture of different species of coffee, collected and dried. Next, there were added to the composites in amount: 1, 3 and 5% by mass.

2.2. Specimens

The alkaline solution was prepared by means of pouring the aqueous solution of sodium silicate over the solid sodium hydroxide. The tap water replaced the distilled one. The solution was mixed and left until its temperature became stable and the concentrations equalized. The fly ash, sand, coffee grounds and alkaline solution were mixed about 15 minutes to receive the homogeneous paste by using low speed mixing machine. Then, the mixture was poured into moulds on cylindrical shape with dimensions: Ø = 50 mm and h = 100 mm. The samples were hand-formed and then subjected to vibratory removal of air bubbles. Tightly closed moulds were heated in the laboratory dryer for 24h at 75°C. Then, the samples were unmounted.

3. Analytical procedure

3.1. Microstructure research

Scanning electron microscope (SEM) type JEOL JSM 820 with EDS has been used for microstructure research. The research has been made for the samples previously broken while compressive strength test. The samples were covered with a thin layer of gold with JEOL JEE-4X vacuum sputter. The investigations were made at various magnifications.

3.2. Efflorescence - visual examination

The cured samples - after 7 days, there were put into distilled water. The water level was on the half of each sample. The specimens were left there 7 days. Then, the efflorescence process has been initiated. It became more intense during the next few week. The visual examination were made after 28 days.

3.3. Compressive strength test

The compressive strength test was carried out on cylindrical shape with dimensions: Ø = 50 mm and h = 100 mm. Test was performed on a testing machine MATEST. The specimens were cured in humidity 50% and temperature 21°C and investigated after 28 days and 3 months. The compressive strength test was carried out according to the methodology described in the standard PN-EN 13791. The characteristic compressive strength is the lowest value from (equation 1):

$$f_{ck,is} = \min \left\{ f_{cm(n),is} - k, f_{is,lowest} + 4 \right\} \quad (1)$$

where:

$f_{ck, is}$ – characteristic compressive strength of concrete ‘in-situ’

$f_{cm(n), is}$ – average compressive strength of concrete ‘in-situ’, for ‘n’ samples

$f_{is, lowest}$ – the lowest result for compressive strength of concrete ‘in-situ’,

k – coefficient, it is depended on the number of results ‘n’ and is equal: for $n = 10 \div 14 \rightarrow k = 4$; for $n = 7 \div 9 \rightarrow k = 5$; for $n = 3 \div 6 \rightarrow k = 6$,

After taking under consideration the correction coefficient $\gamma = 0,85$ the equation 2 is applied:

$$f_{ck, is} = \min \left\{ f_{cm(n), is} - k \right\} \cdot \frac{1}{0,85} \quad (2)$$

3.4. Water absorption tests

Firstly, the cured samples - after 7 days, were weighted and next immersed completely in distilled water. Then, after next 7 days they were weighted again. The water absorption was calculated according the equation (3).

$$n_w = \frac{G_2 - G_1}{G_1} \cdot 100\% \quad (3)$$

where:

G_1 – average mass of dry samples,

G_2 – average mass of samples after 7 days in distilled water.

In the second step, the samples were again dried during the 7 days and after this time they were weighted.

3.5. pH test

The cured samples - after 14 days, were tested according to pH. The first 7 days after de-moulded, specimens were cured in humidity 50% and temperature 21°C. Next 7 days there were immersed in distilled water. The pH of the leachate was measured using a pH meter (CP-105 type).

4. Experimental results and discussion

4.1. Microstructure research

The SEM observations show differences between the samples reinforced by coffee grounds and samples without reinforcement (figure 1.).

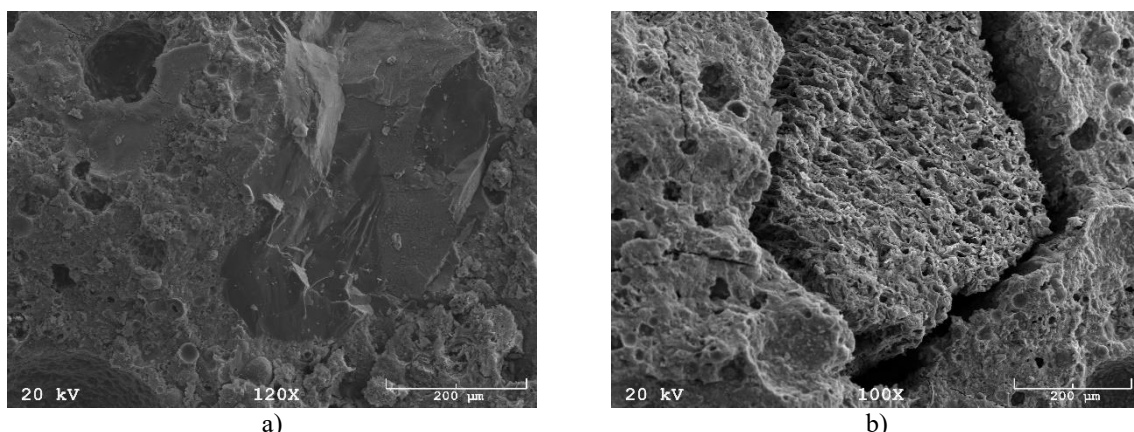


Figure 1. SEM image: a) morphology of geopolymer matrix b) morphology of geopolymer matrix reinforced by coffee grounds

The EDS studies show the coffee grounds were impregnated by the alkaline solution used in the geopolymerization process. The weight ratio of Al/Si in sample without reinforcement was 0.5 and the weight ratio of Na/(Al+Si) was 1.0. The samples with coffee grounds have following ratios:

- for geopolymer matrix: weigh ratio of Al/Si – 0.4, and weigh ratio of Na/(Al+Si) -0.1.
- for reinforcement: weigh ratio of Al/Si – 0.2, and weigh ratio of Na/(Al+Si) - 0.4.

4.2. Efflorescence - visual examination

The results of the visual examination of efflorescence are shown in the figure 2. The efflorescence occurred on all samples, however it had different character depending on amount of additive. On the samples with 5% additive, the efflorescence was rather directly up the line of water and it was reduced on the upper surface. It is probably caused by “closing” capillary pore system by coffee grounds.

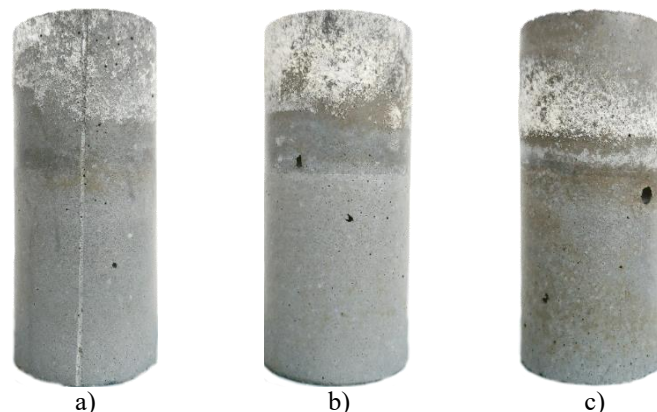


Figure 2. Efflorescence – the samples with coffee grounds: a) 1% b) 3% c) 5%

Carbonate efflorescence on concrete surfaces is formed as a result of the carbonation of calcium hydroxide - $\text{Ca}(\text{OH})_2$. Calcium hydroxide, also called portlandite, is one of the cement hydration products (phases of silicate Portland clinker). Carbonate efflorescence occurs when the calcium hydroxide transported through the capillary pore system gets to the surface of the concrete element.

The rate and intensity of carbonation is influenced by CO_2 concentration and ambient humidity. In concrete saturated with water, the carbonation process is practically non-existent, and in the case of dried concrete, carbonation occurs very slowly. Generally, the carbonation occurs when there is a water film on the surface of the capillaries, in which the rapidly diffusing CO_2 dissolves and reacts with the calcium ions. The fastest carbonation process is in the range of 50% to 70% relative humidity [9].

A similar mechanism occurs in the case of geopolymers and geopolymer concretes. The difference in the chemical composition of the geopolymers comparison with traditional concretes results in the possibility of the appearance of sodium carbonate heptahydrate on the surface, which is revealed quite quickly in the right hydrothermal conditions [3].

The intensity of carbonation of concrete exposed to the external environment increased with the porosity of the concrete, its susceptibility to cracks and scratches and the w/c ratio in the mixture [10,11]. It also depends on density of the concrete mixture (the lower density favours the carbonization), the concrete care and the concentration of CO_2 in the ambient air (the higher amount of CO_2 favours the carbonization) [12, 13].

For conventional concretes, efflorescence is generally harmless except for discoloration [14]. The standards for concrete products (eg PN-EN 1338: 2005) allow the occurrence of the efflorescence as a natural element of concrete maturation. In the case of geopolymers the efflorescence could influence on the mechanical properties.

4.3. Compressive strength test

The results of the compressive strength test are shown in the figure 3.

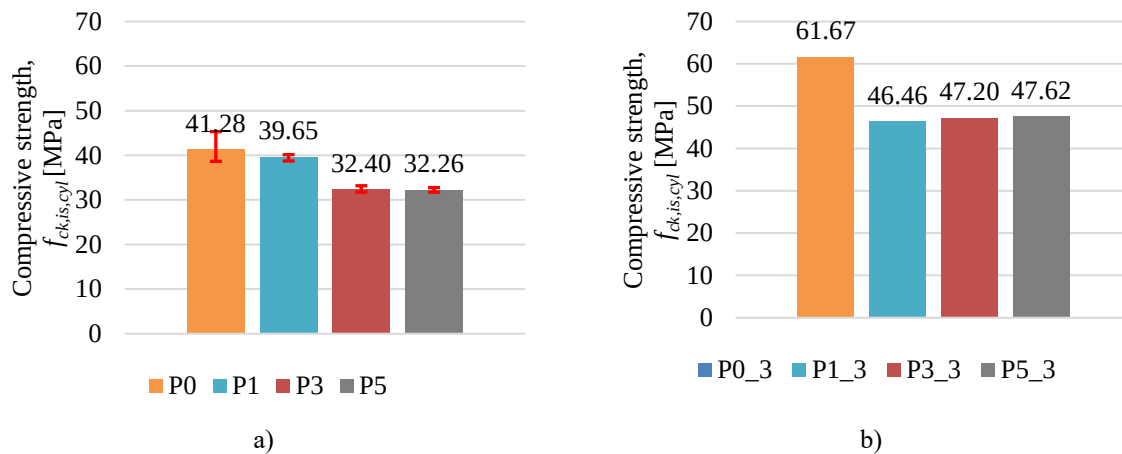


Figure 3. The results of the compressive strength test: a) after 28 days b) after 3 months.

The results of the research show the alignment of strength values for samples with the coffee grounds in relation to plane samples after 28 days.

4.4. Water absorption tests

The results of the water absorption test are presented on the figure 4. The blue columns are for first stage and the red one for second stage of the process.

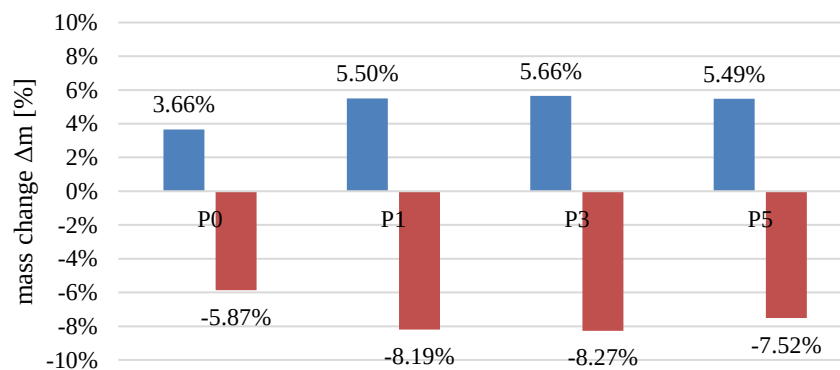


Figure 4. The results of the water absorption test (upper: after 7 days of soaking; red: after re-drying)

The results of the research show that the samples containing the additive of coffee grounds have better absorbent properties compared to the plane samples (without reinforcement). Regardless of the amount of the coffee grounds, the difference in absorbability is relatively small. The additive of coffee grounds largely allows the moisture to evaporate as a result of increased porosity.

4.5. pH test

The results of the pH test are presented in the table 2.

Table 2. The pH values of the leachate.

	P0	P1	P3	P5
pH	10.99	9.61	9.33	9.21

The results of the research confirm the decrease of pH of the leachate for the samples containing coffee grounds. The change in the weight content of coffee grounds in geopolymer samples did not

significantly change the pH. Coffee grounds significantly reduce the pH of the leachate in comparison with plane samples (without reinforcement).

5. Conclusions

Geopolymer composites reinforced with coffee grounds in amount 1, 3 and 5% by weight as well as plain (without reinforcement) specimens have been produced and characterized. The results of the research and literature review show that the additive of the coffee grounds to geopolymers could prevent the efflorescence. In the same time, the composites have reasonable properties for many application, exemplary in construction industry.

The results of the research show the influence of coffee grounds on the composite properties. The additive of coffee grounds reduced the pH from about 11 to 9.21. At the same time, the amount of efflorescence on the surface decreased, but also the mechanical properties came down. The results of the compressive strength test show 20% reduction of the compressive strength for samples containing 5% of coffee grounds compared to the reference samples (plane samples). Also, the results of the water absorption for the samples with reinforcement are worse. However, the mechanical properties for the samples with reinforcement are lower, these samples have more repeatable results. The additive regulates the process of curing and seems to protect the material against changeable conditions.

6. References

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Acknowledgments

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