Impact of graphite admixture on electrical properties of alkali-activated slag mortars

Ivo Kusak[1,*], *Miroslav* Lunak[1] and *Pavel* Rovnanik[2]

[1]Institute of Physics, Faculty of Civil Engineering, Brno University of Technology, Veveří 95, 602 00 Brno, Czech Republic
[2]Institute of Chemistry, Faculty of Civil Engineering, Brno University of Technology, Veveří 95, 602 00 Brno, Czech Republic

Abstract One of the objectives of the applied research's striving consists in providing the users with new slag-mortar-based materials. Basic research, in its turn, aims at examining the newly created materials from the viewpoint of all possible testing methods. Slag mortar specimens were subjected to electrical analysis carried out by means of an ZNC vector analyser and an SPEAG-made DAK-12 coaxial probe within the frequency range from 100 MHz to 3GHz and, furthermore, a dedicated automatically measuring device within the frequency range from 40 Hz to 1 MHz. The frequency spectra of interest were measured on various copolymer specimens differing from each other by the content of the carbon powder. Higher graphite powder content increases the electrical conductivity of cement/slag-based building materials, which thus become easier to measure by means of electromagnetic measuring methods. Carbon admixture may also improve the material's antistatic properties.

1 Introduction

The development of alkali-activated slag (AAS) cements has been object of intensive research recently [1]. The AAS cements are manufactured by mixing fine-ground glass slag with strong alkaline solutions, such as water glass NaOH and Na_2CO_3. As far as their mechanical properties are concerned, the AAS cements are comparable with the PC (Portland cement), particularly in the case where water glass is used as activator. They also feature better endurance, better resistance against aggressive environment, better frost resistance and lower amount of hydration heat released. Among their most important drawbacks, there are the high autogenous as well as dry-up-related shrinkage, which is particularly characteristic for activators in the form of water glass. If NaOH and Na_2CO_3 are used, they show similar shrinkage as the PC. Among other drawbacks there are their propensity to form efflorescences and, in some cases, to too fast setting [2, 3].

To enhance the applicability of electrical measuring methods, graphite powder appears to be a convenient admixture, increasing the electrical conductivity of the bodies under test. Thus obtained increased electrical conductivity will enhance the measurability and provide for better utilization of the impedance spectroscopy method. The amount of the graphite

* Corresponding author: kusak.i@fce.vutbr.cz

powder admixture is of the order of several per cent of the specimen total weight. Attainment of the so-called percolation threshold could be detected by the measurement [4, 5].

2 Experiment setup

Following brands of input raw materials were used for the specimen preparation: SMŠ 380 granulated blast-furnace slag, SUSIL MP 2.0 dried water glass, PG1-3 sand, COND 8 96 powdered graphite, Triton X-100 nonionic detergent, Lukosan S.defoamer.

First, the suspension of COND powdered graphite with Triton X100 detergent and about 100 ml of water were added to the water glass to be stirred in a mixer for 1 minute. Subsequently, SMŠ 380 slag and 3 gradings of sand (PG1-3) were added. Finally, Lukosan S defoamer was added. The moulding being completed, the specimens were immersed into water. After 28 days, they were taken out to be kept in the open air for 7 days in order to stabilize their water content. Subsequently, the specimens were dried up at a temperature of 105 °C until a constant mass was reached.

Three test bodies of dimensions 40 × 40 × 160 mm from each of the fifteen mixtures were manufactured. The results obtained from each test body are compared with those of the reference specimen.

Table 1. Chemical composition of granulated blast-furnace slag (%).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	MnO	SO_3
39.75	6.61	0.46	39.03	10.45	0.63	0.38	0.37	0.71

Table 2. Mixture composition.

Mixture	**Slag** [g]	**Water glass** [g]	**Sand** [g]	**Graphite** [g]	**Triton X-100** [ml]	**Lukosan S** [ml]	**Water** [ml]
G0				0	0	0	185
G1				4.5	30	5	150
G2				9	30	5	155
G3				13.5	30	5	160
G4				18	30	5	165
G5				22.5	60	10	135
G6				27	60	10	140
G7	450	90	1350	31.5	90	15	110
G8				36	90	15	115
G9				40.5	120	20	85
G10				45	120	20	90
G15				67.5	180	30	75
G20				90	240	40	70
G25				112.5	300	40	70
G30				135	390	40	70

The bodies to be tested (Table 2) were characterized by means of the impedance spectroscopy method [6]. The specimens were measured in the frequency range from 40 Hz to 1 MHz. An R&S ZNC vector analyser with DAK12 coaxial probe (made by Speag) was used to measure at higher frequencies (from 100 MHz to 3 GHz). The electrical conductivity σ was measured within this frequency range.

Micro-images of alkali-activated slag mortars were taken at TESCAN MIRA3 XMU by means of a scanning electron microscope in the SEM mode. The experiments were carried out on dry specimens, the surface of which was coated with dust gold using an accelerating voltage of 20 kV.

3 Results

Electrical properties have been measured in two frequency bands of the high-frequency electric field.

Fig. 1 shows the specimen electrical resistance vs. frequency plot within the frequency range from 40 Hz to 1 MHz with graphite powder content as a parameter. The electric resistivity decreases when the graphite amount and the electric field frequency increase. However, there is quite a distinct peak, which is located on the reference mixture curve (G0) at a frequency of about 1 kHz. A tiny peak can be observed at this frequency for the mixture, which was enriched by adding 1% of graphite. Only a monotonous decrease of the electric resistivity without any pronounced anomalies is observed when higher graphite amounts are added. The resistivity of the composites, which contain higher amounts of the powdered graphite, reaches its minimum value at relatively low frequencies below 10 kHz. The electric resistivity of all tested specimens decreased to a similar level above 100 kHz. To analyse the influence of the added graphite amount, a reference frequency of 1 kHz was chosen (at this frequency, highest differences in the resistivity are observed).

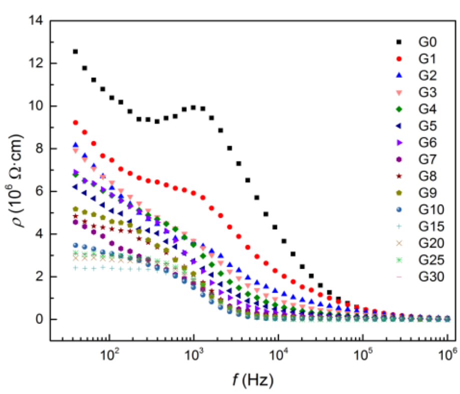

Fig. 1. Electric resistivity of AAS composites with 0 to 30% of powdered graphite in the frequency range from 40 Hz to 1 MHz (the frequency is plotted in log scale).

Fig. 2a shows that even a small amount of added graphite reduces the electric resistivity substantially. The electric resistivity decreases when the amount of the filling substance (graphite) is growing. Its most effective value in terms of the graphite amount to the electric resistance ratio is reached at 10% of graphite. Such an amount of graphite may be supposed to be sufficiently high for ensuring contact between adjacent particles and, therefore, appears to be the best suited from the electric resistance (conductivity) viewpoint. It may therefore be stated that a higher graphite content than 10% influences the electric resistance of AAS composites to a lesser extent in the low and medium frequency range.

Fig. 2 illustrates the electrical conductivity vs. graphite content plot for six different frequencies of the electric field, namely, 0.5 GHz, 1 GHz, 1.5 GHz, 2 GHz, 2.5 GHz and 3 GHz. The mixture containing 10% of graphite features a substantial growth of the electric conductivity for all selected frequencies of the electric field. An electric conductivity limit is evident to occur at lower frequencies (0.5 GHz, 1 GHz and 1.5 GHz) for at least 25% of graphite added. At other frequencies, a steady growth of the electrical conductivity is observed irrespective of the graphite content (corresponding, for example, to a G30 mixture with 30% of graphite). Generally, the steepest growth of the electrical conductivity is observed below and up to 10% of added graphite. The growth is slower for higher graphite percentage. Electric conductivity versus frequency relationship is clearly evident here. The most noticeable differences can be observed at a frequency of 3 GHz and the powdered graphite content of 30%. The higher the electric field frequency the higher value of the electric conductivity is reached for 30% graphite specimens.

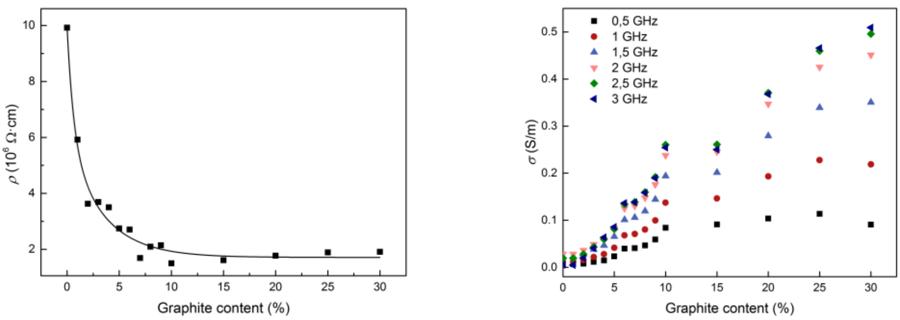

Fig. 2. a) Change in electric resistivity of AAS composites versus the graphite content at 1 kHz; b) electrical conductivity of AAS composites with 0–30% of graphite powder at different frequencies.

Fig. 3a, 3b and 4a show the morphology of the fracture surface of AAS composites with conducting filling substance. Graphite particles in the form of relatively thin slabs with lamellar structure are clearly visible (Fig. 4b). In view of the fact that in this case these particles are sparsely dispersed (the AAS matrix contains only 5% of graphite), only few graphite particles can be seen here. Amorphous structure, which is prevailingly seen in the microscopic image, corresponds to the alkali-activated slag that is predominating here (Fig. 3a). Gradual increase of the graphite amount results in the particle denser packing, which in turn enhances the electric conductivity. After 3% of the powdered graphite is added, the graphite will occupy about 50% of the matrix volume and the particles will be in close contact with each other (Fig. 4a). Such an amount enables the contact conductivity of the AAS matrix, the graphite internal resistance becoming the limiting factor. We would like to thank Dr Patrik Bayer for microscopic measurements (SEM – Scanning Electron Microscope).

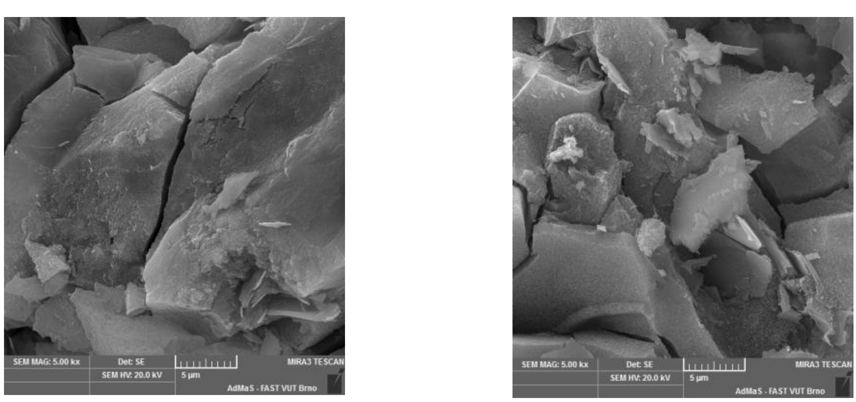

Fig. 3. a) Microstructure of AAS composites with 5 % of graphite; b) 10 % of graphite.

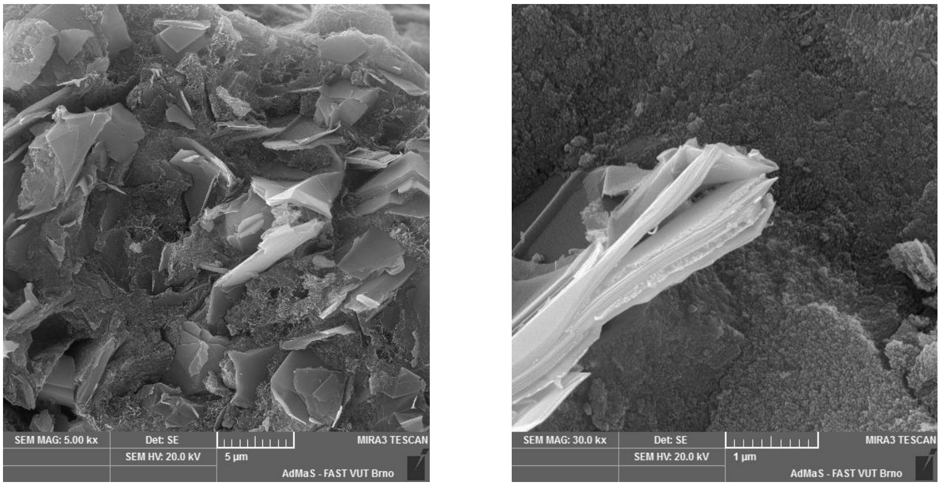

Fig. 4. a) Microstructure of AAS composites with 30 % of graphite; b) lamellar structure of graphite particles.

4 Conclusion

The present paper deals with the change of electric parameters of alkali-activated slag mortars to which powdered graphite has been added. This graphite admixture improves the electric conductivity of the materials in question, making them easier to measure by electromagnetic-principle-based methods. An ideal graphite admixture is described from the viewpoint of both its amount and the most effective increase of the electrical conductivity. Another quantity to study was the electric resistivity and its frequency dependence. The micro-structure of the AAS composites with conducting filling substance has also been analysed by means of a scanning electron microscope.

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References

1. R. Zhao, J. G. Sanjayan, Mag. Concrete Res. **63**, 163 (2011)
2. F. Puertas et al., J. Eur Ceram. Soc. **31**, 2043 (2011)
3. A. Fernandez-Jimenez: Cement Concrete Res. **29**, 1313 (1999)
4. S. Matsutani, Y. Shimosako, Y. Wang, Physica A. **391**, 5802 (2012)
5. K. Y. Kim, T. S. Zun, K. P. Park, Cement Concrete Res. **50**, 34 (2013)
6. I. Kusak; M. Lunak, P. Schauer, Appl. Mech. Mater. **248**, 370 (2013)