

ON THE CHANGE OF ELECTRIC PARAMETERS OF CINCRETE MEASURED
WITH A CONTACT COMPOUND

Ivo Kusák¹, Bohumil Koktavý¹, Miroslav Luňák¹

¹Department of Physics, Faculty of Civil Engineering, Brno University of Technology,
Veveří 95, 602 00 Brno, Czech Republic

Abstract

NDT method of impedance spectroscopy was used to characterize concrete slab specimens without and with contact graphite compound coating. Differences in $\tan\delta(f)$, $\text{Im}Z(f)$ and $\text{Re}Z(f)$ spectra for dry specimens were observed. Furthermore, based on the predominant loss type, the material quality was characterized. Our measurements confirmed the reproducibility of this method. Furthermore, the influence of the concrete specimen composition on their electric parameters was assessed.

Key words: *impedance spectroscopy, dielectric losses, loss factor, conductivity losses, polarization losses, contact paste*

1. INTRODUCTION

The measurement method presented here is based on the measurement of electric impedance characteristics of the system whose configuration is metal - building material - metal. The building material behaves as a dielectric in this system. We are therefore studying the properties of this dielectric. The key indicators, representing the specimen system to be analyzed are the measured system impedance real and imaginary part and, mainly, the loss factor versus frequency plots. As supplementary indicators serve the resistance and capacitance versus frequency plots of the system into which the building material in question was built in.

The method is intended to provide both qualitative and quantitative evaluation of the above-mentioned parameters. The curve shape makes it possible to detect the effect of dampening and drying on the material, changes in its structure, change in porosity, its service life reduction, technology related defects, etc. By comparing the results with an impedance spectrum of defined specimens, we may exactly identify the deviations in the properties being tracked. However, it is beyond the capacity of this method to quantify the building material parameters. On the other hand, this method can be very well used as a comparative method and we expect that, after some analysis and refinement of the idea are accomplished, it can be used as an absolute method.

The frequency dependence of the quantities in question is being evaluated by means of impedance spectroscopy from the voltage ratios and phase shifts between the A, B, inputs of the oscilloscope.

The values of the capacitance C_B and resistance R_B forming the parallel input network depend on the channel B input capacitance and resistance (including those of the lead-in cables with BNC connectors). There is, therefore, no need for inserting any accurate $C - R$ network into the B input. Let the resulting impedance of the $C_B - R_B$ network be denoted Z_B and its phase, φ_B (ω = angular frequency).

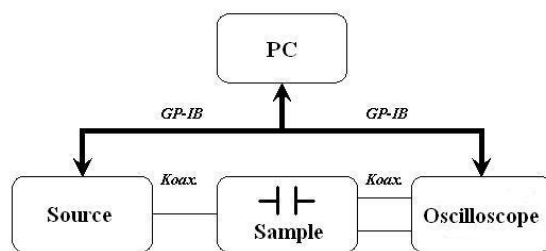


Fig 1 Block diagram of the measuring apparatus.

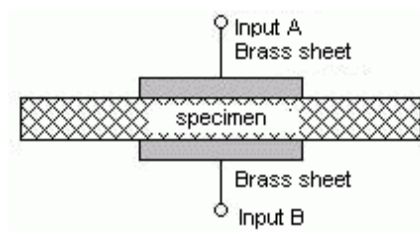
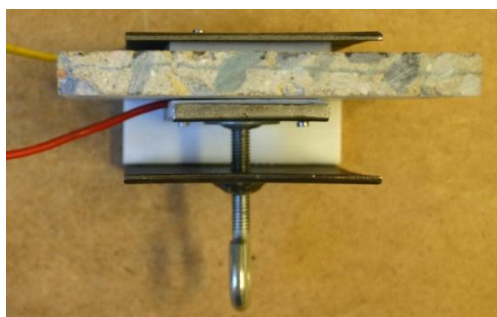


Fig 2 Metal – building material – metal system. (Brass electrodes are pressed against the measured specimen surface).

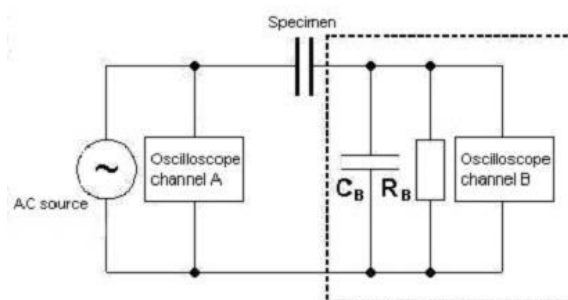
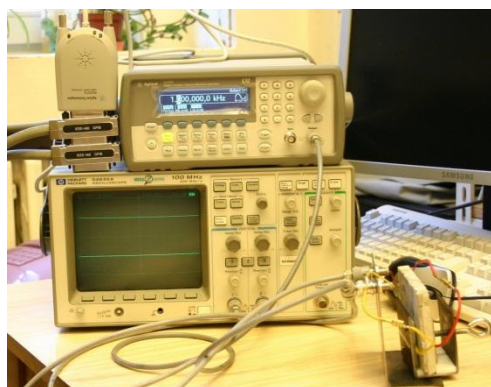


Fig. 3 Illustration and block diagram of the apparatus employing Agilent and HP measuring instruments.

The phase of the measured impedance Z will be

$$\varphi_I = \varphi_B + \arctan \frac{\sin \Delta\varphi}{\frac{U_B}{U_A} - \cos \Delta\varphi}. \quad (1)$$

The absolute value of the measured impedance will therefore be

$$Z = \frac{Z_B \cdot \sin \Delta \varphi}{\frac{U_B}{U_A} - \sin(\varphi_B - \varphi_I)} . \quad (2)$$

The loss factor of the system under test:

$$\tan \delta = \frac{I}{\tan \varphi_I} . \quad (3)$$

The resistance of the parallel RC network representing the measured system (the specimen) is

$$R = \operatorname{Re}(Z) \cdot (1 + \tan^2 \varphi_I) \quad (4)$$

and its capacity

$$C = -\frac{\tan \varphi_I}{\omega R} . \quad (5)$$

2. CONCRETE SLABS: DESCRIPTION AND FORMULA

Steel moulds were used to manufacture the test specimens. The mix was poured into the moulds in two consecutive layers. Having been filled, the moulds were deposited in the lab to be kept at a temperature of 20 ± 1 °C and relative humidity RH of 45 ± 5 %. The moulds were covered with a PE foil after the surface had withered. After 24 hours had elapsed, the specimens were subjected to final moulding. Subsequently, they were immersed into a water bath for 28 days.

The dimensions of thus formed blocks were 100 x 100 x 400 mm. These blocks were cut by a diamond saw to produce slabs of a thickness of 10 mm. Therefore, the dimensions of these slabs were 100 x 100 x 10 mm. Out of these dimensions, the thickness is the most important parameter because of the signal attenuation.

B20/25 concrete with 4 m maximum grain size was selected as the reference formula.

Table 1. Formulas for the different concrete mixes.

R3E	quantity	unit	R4B	quantity	unit
	1 m ³			1 m ³	
CEM II 32.5 R cement	325	kg	CEM II 32,5 R cement	325	kg
0 to 4 mm sand (Bratčice)	1885	kg	0 to 4 mm sand (Bratčice)	1125	kg
-	-	-	4 to 8 mm grit (Tovačov)	760	kg
real water	283.7	l	real water	223.8	l
water to cement ratio w/c	0.87	-	water to cement ratio w/c	0.69	-

- machine mixed, using a manual mixer

- dried sand

- The ingredients having been blended according to the formula, the mix appeared to be dry (loose). A total of 113.7 L of water have been added per 1 m³ of fresh concrete

- consistence test: cone slump = 0 cm

- 13 + 20 s compaction on a vibrating table

- machine mixed, using a manual mixer

- dried sand, 4-8 mm aggregates, "slightly" wet

- The ingredients having been blended according to the formula, the mix appeared to be dry (loose). A total of 53.8 L of water have been added per 1 m³ of fresh concrete

- consistence test: cone slump = 0 cm

- 2 x 15 s compaction on a vibrating table

R5E	quantity	unit	R6B	quantity	unit
	1 m ³			1 m ³	
CEM II 32.5 R cement	325	kg	CEM II 32.5 R cement	325	kg
0 to 4 mm sand (Bratčice)	1125	kg	0 to 4 mm sand (Bratčice)	1125	kg
4 to 8 mm grit (Tovačov)	380	kg	4 to 8 mm grit (Tovačov)	253	kg
8 to 16 mm grit (Olbramovice)	380	kg	8 to 16 mm grit (Olbramovice)	253	kg
-	-	-	16 to 22 mm grit (Želešice)	253	kg
real water	207,9	l	real water	219,2	l
water to cement ratio w/c	0,64	-	water to cement ratio w/c	0,68	-

- machine mixed, using a manual mixer

- dried sand, 4 to 8 mm aggregates: "slightly" wet, 8 to 16 mm aggregates: dried up

- The ingredients having been blended according to the formula, the mix appeared to be dry (loose). A total of 37.9 L of water have been added per 1 m³ of fresh concrete

- consistence test: cone slump = 0 cm

- 15 + 20 s compaction on a vibrating table

- machine mixed, using a manual mixer

- dried sand, 4 to 8 mm aggregates: "slightly" wet, 8 to 16 mm and 16 to 22 mm aggregates: dried up

- The ingredients having been blended according to the formula, the mix appeared to be dry (loose). A total of 49.2 L of water have been added per 1 m³ of fresh concrete

- consistence test: cone slump = 0 cm

- 2 x 15 s compaction on a vibrating table

3. EXPERIMENT

Concrete slab specimens were inserted – one behind the other – in a fixture between 2 brass electrodes of dimensions 40 x 25 mm (which were pressed to them by means of a jackscrew) to be subjected to an impedance analysis. As this was a comparison method, the specimen surfaces had to feature the same roughness and, as far as possible, an overall adherence to the electrodes. The input voltage equalled 5 V and the applied frequency ranged from 40 Hz to 1 MHz.

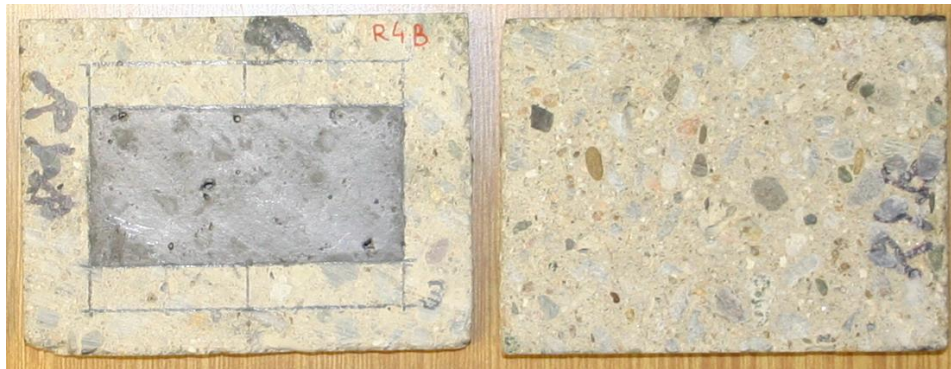


Fig. 4. Concrete specimens with and without graphite coating (carbon paste).

4. MEASUREMENTS RESULTS

In the diagram, there are four curves characterizing the mixes. They differ from each other. The highest loss factor values (Fig. 5) occur at lower frequencies of the exciting signal. The loss factor is observed to decrease at higher frequencies. The first peak to observe is identified for R3 mix at the left-hand side of the frequency scale. The second peak is situated on the right of the first one and pertains to R4 mix. For R5 mix, the loss factor curve is situated slightly higher than that of R4 mix at frequencies below 10 kHz. Except the lowest frequency range, the curves almost mingle with one another. For R6 mix, the peak is observed on the right of other mixes' peaks, namely at about 4 kHz. The mixes under test differ in the kind of the aggregates used. From the shape of the loss factor frequency spectra, we infer a connection between the aggregate grading and the loss factor peak frequency and its shift to higher frequencies. However, the loss factor values near each other for these peak frequencies. Therefore, for the time being we do not infer any connection between $\tan\delta$ and the grading parameters.

Prior to coating the specimen faces with the graphite compound, local peaks were observed in the loss factor spectrum (Fig. 5), whose position in the spectrum shifted to lower frequencies with the decreasing grading size.

The occurrence of the peaks in the frequency spectra indicates the polarization losses to predominate, whereas their decreasing protrusions indicate conductivity-related losses. (Fig. 5).

After the specimen faces were coated with graphite compound, the loss factor doubled approximately (Fig. 6). No local peaks are observed in the diagrams and the curve shape corresponds to the conductivity- related losses. This in turn corresponds to the real and imaginary component drops observed. The impedance real component values did not drop as much as the imaginary ones.

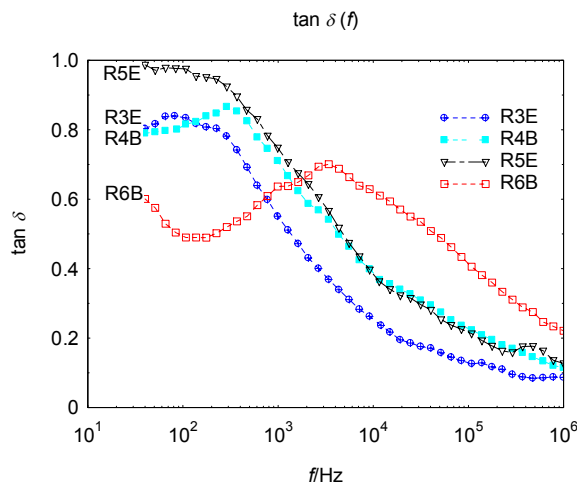


Fig. 5 Loss factor versus frequency diagram (without graphite compound).

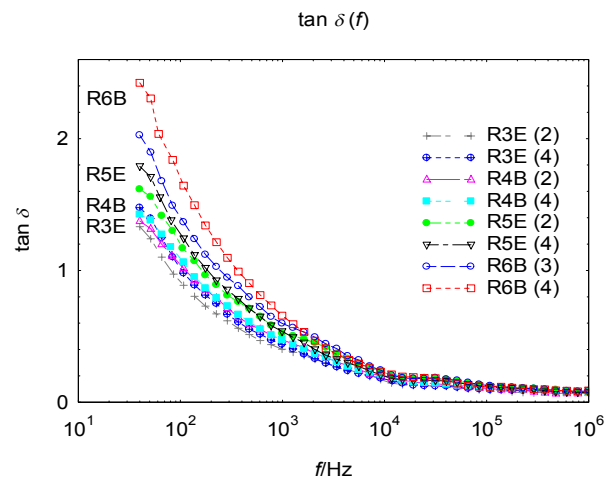


Fig. 6 Loss factor versus frequency diagram (with graphite compound).

The numerals in parentheses in Figs. 6, 8 and 10 stand for the specimen number of the compound used, so that the result reproducibility can be tracked for different specimens of the same mix.

In view of the fact that the electrode modification (spreading the graphite compound over the surface) is expected to result in reducing the impedance real and imaginary component values, we are not able to estimate the change in the loss factor. It is just this quantity which would provide us with information about which of the concrete slab impedance components is more sensitive to the specimen-to-electrode contact face quality improvement.

Assessment of the impedance real component spectra (Fig. 9 and 10) confirmed the expected drop in the real component resulting from coating the specimen faces with the graphite compound, for all specimens and for all aggregate gradings. The drop amounted approximately to 30 % at the lowest frequencies. The non-graphite curve shape is convex. Coating the faces with the graphite compound makes the curve turn into a concave-shaped one in the lowest frequency region.

For different frequencies of the exciting signal, the electrical resistance (Figs 9 and 10) trend shows to be opposite to the electrical capacitance (as calculated from the impedance imaginary component, i.e., the capacitance). Larger-sized gradings result in lower resistances, being nearly identical for specimen No 4 and 5. The curve differences can only be observed in the frequency range from 40 Hz to 1 kHz. The differences are hardly measurable in the remaining part of the frequency range.

When analyzing the impedance imaginary component versus frequency diagrams, we observe a drop in values of $\text{Im}(Z)$ of about 60% for each of the specimens. The curve shape changes from convex to concave in the lowest frequency region, too. A local peak (Fig. 8) occurred for specimens R5, R6 (higher aggregate grading).

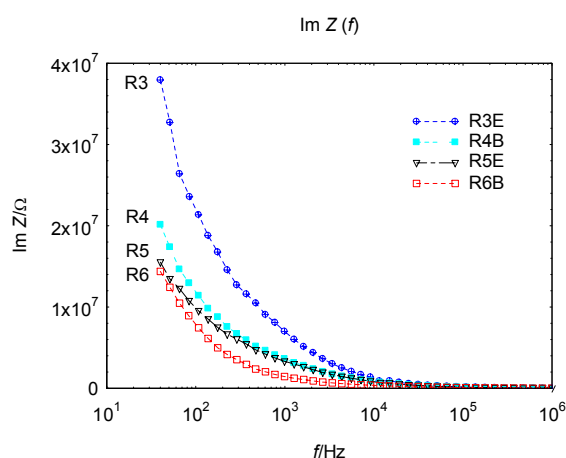


Fig. 7 Impedance imaginary component versus frequency diagram (without graphite compound).

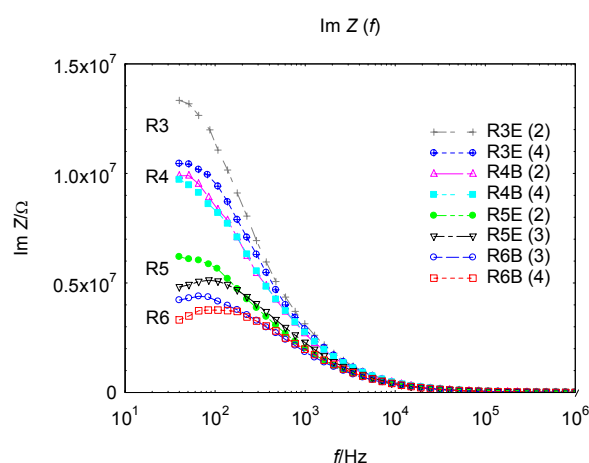


Fig. 8 Impedance imaginary component versus frequency diagram (with graphite compound).

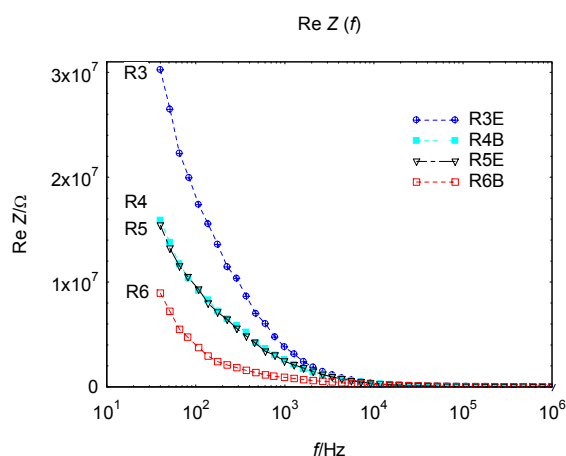


Fig. 9 Impedance real component versus frequency diagram (without graphite compound).

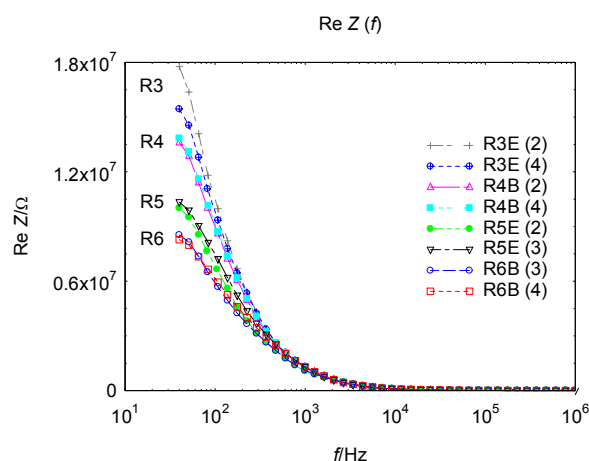


Fig. 10 Impedance real component versus frequency diagram (with graphite compound).

The highest capacitance values to measure were observed for the mixes containing the largest-size aggregate gradings (Table 2). Reduced gradings size resulted in lower specimen capacitance values. Judging from the electric capacitance value, the static relative permittivity ϵ_0 is higher for larger-sized gradings. How these values changes after having coated the specimen surface with graphite compound is shown in Table 3.

Table 2. Electrical parameters for the selected (reference) frequency of 100 Hz prior to and after coating the surface with graphite compound.. C - capacitance, R - resistance, $|Z|$ - impedance absolute value, $\tan \delta$ - loss factor, $-\varphi$ - phasor angle.

	Prior to coating	After coating	Prior to coating	After coating	Prior to coating	After coating	Prior to coating	After coating	Prior to coating	After coating
	$C/\mu\text{F}$	$C/\mu\text{F}$	$R/\text{M}\Omega$	$R/\text{M}\Omega$	$ Z /\text{M}\Omega$	$ Z /\text{M}\Omega$	$\tan \delta/-$	$\tan \delta/-$	$-\varphi/^\circ$	$-\varphi/^\circ$
R3E	69.4	143.4	17.40	9.95	27.50	14.91	0.82	0.89	315	318
R4B	130.0	190.6	9.08	8.53	14.57	11.94	0.79	1.05	316	323
R5E	156.0	280.2	9.30	6.71	13.31	8.78	0.98	1.18	322	255
R6B	199.0	379.8	3.75	5.66	8.34	7.04	0.50	1.64	307	23.3

The capacitance C corresponding to the reference frequency of 100 Hz was calculated from the impedance imaginary component $X_C = \text{Im}(Z)$ using following formula:

$$X_C = \frac{1}{\omega C} = \frac{1}{2\pi f C} \Rightarrow C = \frac{1}{2\pi f X_C}. \quad (6)$$

Table 2 summarizes the specimen electric quantities for the selected frequency of 100 Hz. (prior to and after coating the surface with the graphite compound. The capacitance and resistance trends are in agreement with those observed in the frequency spectra. Like the electric resistance values, the impedance absolute values are decreasing with the growing size of the aggregate grading. This confirms the predominance of the real component and is in agreement with the specimen phase angle detected at the selected frequency.

The measurement results are providing us with a true picture of the concrete slab bulk properties. The precondition that the specimen growing capacitance together with decreasing resistance should result in reducing the impedance absolute value therefore appears to be met here.

Table 3. Orienting comparison of the quantities considered.

Non-coated surface		Coated surface	
$\text{Re}(Z)$	>	$\text{Re}(Z)$	Impedance real component (resistance)
$\text{Im}(Z)$	>	$\text{Im}(Z)$	Impedance imaginary component
C	<	C	Capacitance
S	<	S	Electrode superficial area

However, we are not able to infer any connection between the phase angle and the loss factor values at 1 kHz on the one hand and the aggregate grading on the other. The frequencies of 1 kHz and higher do not appear to be suitable for fast evaluation of electric parameters of cement-based and similar materials.

The specimens under test, while dry, were also subjected to reproducibility tests several times at various time periods. Thus obtained curves proved to almost duplicate each other with minimum variances, thus giving evidence of a good reproducibility of the method.

It is to be noted that the different polarization types do not contribute equally at different frequencies to the permittivity of various dielectrics featuring various structure and in the entire frequency range [12]. In a technical dielectric, where basic polarization types occur, the permittivity (or other quantity under consideration) versus frequency curve at a constant temperature results from the combination of the curves corresponding to the different mechanisms. It is from this point of view that the different components' predominance at given frequencies were assessed.

Beside the effect of the frequency, the influence of the temperature should also be taken into account. When increasing the temperature, the dispersion regions are shifting to higher frequencies, because the dipole molecules are becoming more mobile. This is why our measurements were carried out at the laboratory temperature.

5. CONCLUSIONS

The impedance spectroscopy method was applied to characterize cement-based concrete specimens, which differed in various gradings aggregates. We observed a change in the electrical quantity frequency spectra prior to and after having coated the specimen surface with graphite compound. Thin slabs against whose walls electrodes were pressed were studied. A relationship between the aggregate grading parameters and the impedance real and imaginary components was found out. Furthermore, a relationship between the position of a local peak in the loss factor spectrum and the grading employed. No connection between the water-to-cement ratio and the electric parameters has been observed.

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