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## Monitoring of concrete hydration by electrical measurement methods

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### Abstract

Analysis of impedance spectra of inhomogeneous materials is a part of the impedance spectroscopy which is still waiting for its development. Materials having higher electric resistance values (over 500 k $\Omega$ ) can be considered – under certain simplifying assumptions – as dielectrics. A theory of dielectric polarization was formulated by Debye for homogeneous materials. Concrete setting and hardening determine the concrete quality. The impedance spectroscopy method, as one of the non-destructive testing method group, was used to characterize concrete specimens and track the changes in the concrete spectrum during the hydration process. Variances in the loss factor versus frequency  $\tan \delta(f)$  and impedance imaginary component  $\text{Im}(Z)$  versus impedance real component  $\text{Re}(Z)$  of the specimens under investigation have been observed. The specimen quality has been described by means of the loss type prevailing in the material. The results of this study are expected to provide information about the correlation between the n-factor (curve parameter obtained from Cole-Cole diagram) and the concrete setting time. At present, one is not able to determine unambiguously the individual material component contributions to the total electric conductivity and polarization at various frequencies of the exciting field.

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### 1. Introduction

The impedance spectroscopy is a non-destructive testing method, which uses the impedance characteristic frequency dependence to analyse the properties of the material [1]. The experiment set-up designed to study the

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system under investigation includes: a metal-material-metal network, which is relevant for identifying the application limits of the impedance spectroscopy method. The method cannot be applied to thick-layer low-conductivity materials. Reinforced concrete products may serve as an example. The principle of the mentioned method is based on evaluation of the dielectric losses versus frequency plots. The dielectric losses of composite materials and plastics can assume values which are many times higher than those of the most materials commonly used in the building industry.

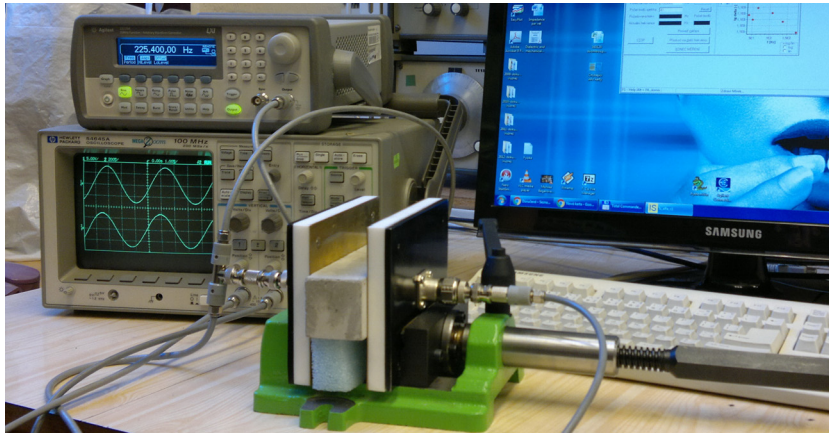


Fig. 1. Measuring set-up and one of specimens: AC power supply, specimen under test, double-channel oscilloscope [1] (illustrative image).

Analysis of impedance spectra of inhomogeneous materials is a part of the impedance spectroscopy, which is still waiting for its development. At present, one is not able to determine unambiguously the individual material component contributions to the total electric conductivity and polarization at various frequencies of the exciting field. Materials having higher electric resistance values (over 500 kΩ) can be considered – under certain simplifying assumptions – as dielectrics. A theory of dielectric polarization was formulated by Debye [2] for homogeneous materials. However, experiments carried out on real materials and the respective conclusions did not show to be in agreement with the fundamentals theories. K S Cole and R H Cole and, also, Fuoss and Kirkwood, started from the Debye's theory to derive models of a dielectric which appear to fit experiment results and conclusions [3] more closely. The behavior of a dielectric in an AC electric field is best described in terms of the complex relative permittivity. Debye has derived a formula for the complex relative permittivity,  $\varepsilon^*$ , of weakly polar liquid dielectrics, as follows:

$$\varepsilon^*(j\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau}. \quad (1)$$

Here  $\tau$  is the relaxation time, independent of the time, however dependent on the temperature,  $\varepsilon_s$  - static permittivity (frequency  $\rightarrow 0$  Hz),  $\varepsilon_\infty$  - optical permittivity (frequency  $\rightarrow \infty$  Hz), angular frequency  $\omega = 2\pi f$ ,  $f$  - frequency of the exciting electric field [4].

Following equation holds for the loss factor  $\tan \delta$ :

$$\tan \delta = \frac{\varepsilon''(\omega)}{\varepsilon'(\omega)} = -\frac{(\varepsilon_s - \varepsilon_\infty)\omega\tau}{\varepsilon_s + \varepsilon_\infty\omega^2\tau^2}. \quad (2)$$

There are several different relaxation times in a real dielectric. Their distribution is described by a distribution

function. Exact determination of a suitable distribution function being difficult, an approximation by a properly selected analytical function is usually carried out. According to Cole's, the complex relative permittivity can be expressed as follows [7]:

$$\varepsilon^*(j\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + (j\omega\tau_1)^{1-\alpha}} \quad (3)$$

Here,  $\tau_1$  is the most probable relaxation time, around which the particular relaxation times are distributed according to a distribution function  $f(\tau)$ , where  $\alpha$  is a distribution parameter ( $0 < \alpha < 1$ ).

J. R. Macdonald [8] made a reference to the formal equivalence between the complex relative permittivity as described by equations (1), (3), and the formulas for a complex impedance  $Z$ . Formulas for the real and imaginary components of the complex relative permittivity have been derived and, based on the above mentioned equivalence, equations for the components of the complex specific impedance have been obtained. Using an appropriate software package, parameters of the two model types have been searched for the material under investigation. The degree of correlation between the model and experiment results was expressed by means of Pearson's correlation coefficient  $r$ .

## 2. Material to be measured

For the impedance spectroscopy measurements, the concrete specimens of dimensions 100 mm × 100 mm × 400 mm (Fig. 2) have been split so as to reduce their thickness to 10 mm, i.e., 100 mm × 100 mm × 10 mm. The specimen composition is given in Table 1:

Table 1. Concrete component percentage.

C 30/37 XF4, S4, surface NH	(kg/m <sup>3</sup> )	(%)
Cement - CEM I 42.5R	320	14.45
Slag 420	100	4.52
Water	210	9.48
Plasticizer - Spolostan 7L	4	0.18
Aeration additive - Chrysoair	0.15	0.01
Sand - Halámky D5 0/4	800	36.13
Aggregate - Rejta 4/8	280	12.65
Aggregate - Rejta 8/16	500	22.58

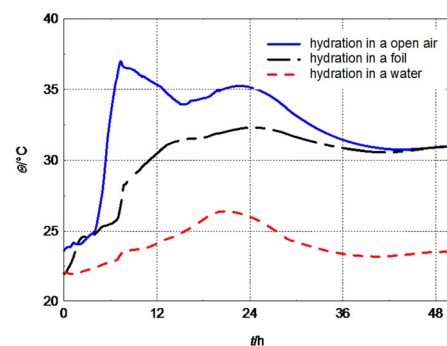


Fig. 2. (a) two concrete specimens intended for the temperature measurement, immediately after having been poured into the beams; (b) monitoring the temperature inside the concrete specimens during the concrete setting process.

### 3. Experimental set up

The experiment, described below, was performed using sinus signal generator Agilent 33220A and Agilent 54645A oscilloscope (Fig. 1). The experiment was designed so as to provide distinguishable impedance spectra of self-setting concrete during the setting process [4–6].

The specimens under investigation were inserted between two electrodes (which were pressed against the specimens using a screw fixture) and subsequently subjected to the impedance analysis.

The loss factor frequency dependence was obtained by using specific software for measurement instruments control. So the frequency dependence of imaginary part of specific impedance on real part of specific impedance was obtained. The specific impedance values were calculated from experimental values. Created and calculated models produced the coefficient  $n$  values, which are expressed in the Table 2.

### 4. Measurements results

When monitoring the concrete hydration, interesting dependence of imaginary component of the impedance  $\text{Im}(Z)$  on the real component of the impedance  $\text{Re}(Z)$  was observed. In this plot, we can determine the angle made by the abscissa (line connecting the arc starting point with the arc centre) with the impedance real axis (see Fig. 3 - the angle made by the straight line with the real axis). Putting the mentioned angle  $\theta$  into the formula, we can calculate the value of the  $n$ -factor, which can characterize the specimen porosity degree:

$$n = 1 - (2\theta / \pi) \quad (4)$$

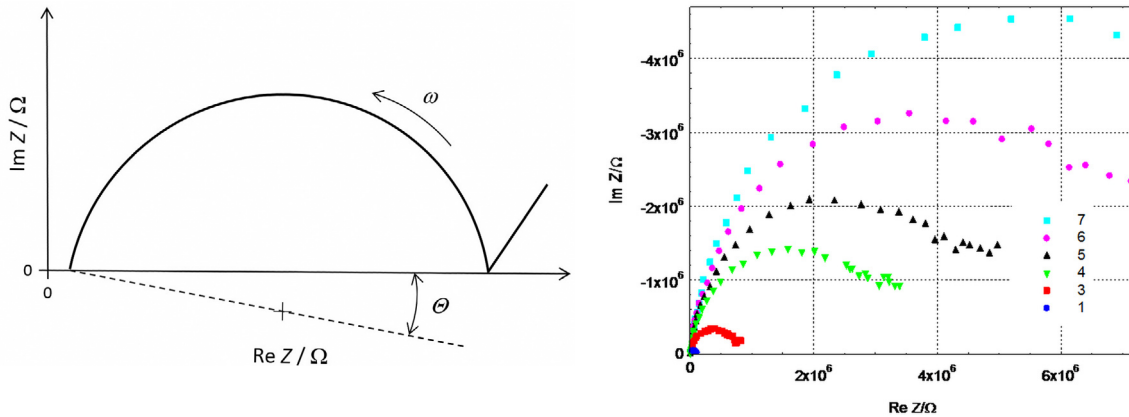


Fig. 3. (a) illustration of an impedance imaginary component  $\text{Im}(Z)$  versus the impedance real component  $\text{Re}(Z)$  plot [8]; (b) impedance imaginary component  $\text{Im}(Z)$  versus the impedance real component  $\text{Re}(Z)$  plot.

The Table 2 shows n-factors for different hydration stages calculated from the impedance imaginary and real parts.

Table 2. Calculated values of the n-factor.

Setting period (days)	<i>n</i>
1 day	0.618
3 days	0.899
4 days	0.908
5 days	0.930
6 days	0.932
7 days	0.933

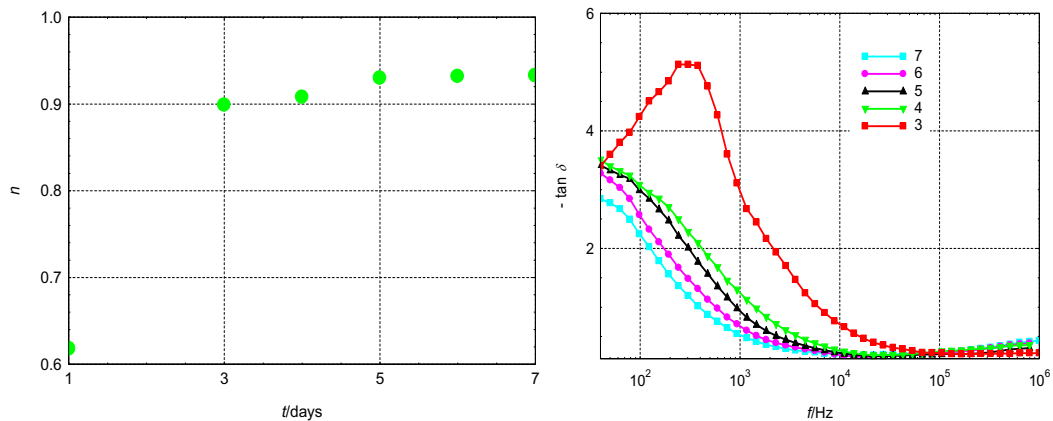


Fig. 4. (a) the n-factor versus time of hydration dependent; (b) loss factor versus frequency plot for a concrete specimen (the frequency being plotted in log scale).

It is clearly seen that the n-factor grows with the hydration time. It remains almost unchanged at the end of the 1-week cycle. The initial lines used in the n-factor calculations are shown in Fig. 3.

The drawback of this characteristic tracking method (Fig. 3b) consists in the need for equal-thickness specimens being cut from the original specimen. From this point of view, the loss factor ( $\tan \delta$ ) versus frequency ( $f$ ) plot is more convenient (in Fig. 4b, the frequency is plotted in a logarithmic scale for lucidity). The loss factor, as a parameter, is a pure material constant. It is related neither to the specimen size, nor to its dimensions [4].

In Fig. 3b, the curve gradation corresponds to a one-day time interval between the measurements. The values of the curve obtained after the first hydration day are by several orders of magnitude lower as compared with the other ones, so that this curve is difficult to identify in the diagram.

Fig. 4b diagram shows clearly the predominance of conductivity losses in the material (the loss factor  $\tan \delta$  decreasing with the frequency) throughout all concrete hydration stages. The longer the hydration time was, the lower the loss factor values were measured (almost throughout the whole frequency spectrum). For example, the sixth day minus the first day hydration  $\tan \delta$  value is about 2.5 for the frequency of 1 kHz. It means that the material electric conductivity decreased during the hydration process. One might assume that the lower the material conductivity (which in turn is due to newly formed capillary pores) the lower loss factors at given frequencies, however, the loss factor value depends on the material permittivity, too. It follows from the  $\text{Im } Z$  ( $\text{Re } Z$ ) phasor diagram, that when the impedance real part is going down, the impedance imaginary part is decreasing as well. This is in a good agreement with the growing loss factor. The predominance of the polarization losses (growing trend of  $\tan \delta$ ) was observed after the first hydration day only at frequencies below about 500 Hz. When the water content

grows, the material loss factor grows as well. In general, polarization mechanisms can be used to characterize the material water content, elasticity variations, defect occurrence etc.

## 5. Conclusion

The impedance spectroscopy method was used to characterize the concrete hydration process stages. The changes in the frequency characteristics respect the assumption that the physical property changes are reflected in the loss factor spectra. Dielectric losses were described.

To characterize the degree of hydration was selected the  $n$ -factor parameter, which was calculated from dependence of imaginary component of impedance  $\text{Im}(Z)$  on the real component of impedance  $\text{Re}(Z)$ . The characteristics were reproducible in short time intervals.

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