Frequency analysis for EIS measurements in autoclaved aerated concrete constructions

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Abstract

In time of energy saving it is important to monitor humidity migration process through delimiting constructions. Autoclaved aerated concrete (AAC) is considered to be one of the most efficient load bearing construction materials in aspect of thermo insulation. For AAC it is important to determine the distribution of humidity throughout the cross section of the construction because it influences the thermic resistance of the construction. Therefore, it is important to monitor the drying process of AAC masonry constructions. This aim can be reached by non-destructive testing methods, which are easily applicable on inhabited buildings as well as on buildings, which are in construction phase. One of such methods is electrical impedance spectrometry (EIS), which can be used for determining of humidity distribution throughout the cross section of the construction. For this non-destructive testing method, it is important to choose correct measurement frequency in order to obtain credible results of humidity distribution. In this paper, methods of choosing suitable measurement frequencies are described as well as impact of different factors on choosing of the most suitable measurement frequencies of AAC masonry constructions.

Keywords: non-destructive testing; electrical impedance; spectrometry; frequency analysis; humidity distribution; aerated concrete

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1. Introduction

Autoclaved aerated concrete (AAC) is a load bearing construction material with high performance of heat insulation parameters. However, the heat insulation parameters of the ACC are influenced by moisture content and its distribution throughout the cross section of the AAC material. In order to reach the heat insulation parameters stated by the manufacturers it is necessary to control the drying process of the construction and to avoid situations when the drying is slowed down by application finishing layers on the surface of aerated concrete blocks which have not lost the excess of the humidity from the manufacturing process.

For the humidity distribution measurements electrical impedance spectrometry (EIS) can be used. EIS is a non-destructive testing method, which allows to monitor the drying process and moisture migration through the cross section of autoclaved aerated concrete masonry constructions simultaneously in several points of materials’ cross section. Such monitoring helps to prevent situations when early started finishing works prevent masonry construction from drying process and it prevents the autoclaved aerated concrete masonry construction to reach the stated heat insulation parameters.

Prior application of EIS measurements on AAC masonry construction in order to determine humidity distribution throughout the cross section of the construction it is important to perform several preparation works. One of the most important preparation activities is the determination of the most suitable measurement frequency for the corresponding type of AAC masonry elements. In order to determine this frequency range a frequency analysis must be performed. The subject of the research described in this paper is to determine the factors, which have significant impact on results of frequency analysis and their impact on further interpretation of EIS measurement data.

2. Methodology

2.1. EIS method

Method of electrical impedance spectrometry (EIS) enables detection of the distribution of impedance and other electrical variables (such as resistivity, conductivity etc.) inside a monitored object, and thus the observation of its inner structure and its changes. The possibility of application of this method on concrete based materials has been researched by McCarter and Garvin [1] and Rajabipour [2], Weiss et.al. [3, 4, 5]. As stated by Barsoukov and McDonald [6] EIS is the method of choice for characterizing the electrical behavior of systems in which the overall system behavior is determined by a number of strongly coupled processes, each proceeding at a different rate. This method is widely used in measuring properties of organic and inorganic substances. Researches of EIS performed by Skramlik, Novotny [7] and Parilkova et a. [8], state that EIS constitutes a very sensitive tool for monitoring processes that take place in objects (e.g. changes occurring in earth filled dams when loaded by water, in wet masonry sediments etc.), electrokinetic phenomena at boundaries (e.g. electrode/soil grain, between soil grains) or for describing basic ideas about the structure of an inter phase boundary (e.g. electrode/water or solid electrode to solid electrode boundary).

The EIS is based on the periodic driving signal – the alternating signal. Therefore, the main measurement value is the impedance of AC circuit, which consists of electrodes (measurement device), and the solid electrolyte (in particular case AAC masonry construction). The range of frequencies used for the driving signal enables the characterisation of systems comprising more interconnected processes with different kinetics. At an interface, physical properties - crystallographic, mechanical, compositional, and, particularly, electrical - change precipitously and heterogeneous charge distributions (polarizations) reduce the overall electrical conductivity of a system.
Electrical impedance is a basic property characterizing the AC electrical circuits. It is always greater than or equal to the real electrical resistance $R$ in the circuit. Imaginary resistance, i.e. inductance - reactance of inductor $XL$ and capacitance - reactance of capacitor $XC$, creates variable and therefore frequency - dependent part of the impedance. Electrical impedance is evidently made up of real and imaginary parts. Resistance $R$ creates real part and is frequency-independent. Imaginary part is created by reactance $X$, which is frequency-dependent. Electrical impedance can be expressed by Ohm’s equation for AC circuits, i.e. by the ratio of electric voltage phasor $U$ and electric current phasor $I$.

2.2. Z-meter III device

For the particular research, a Z-meter III device (Fig.1) will be used for EIS measurements. Z-meter III device has been developed within the solution of an international project E!4981 of programme EUREKA by leading scientist of the programme Parilkova [9, 10]. This instrument has been verified in laboratory experiments and measurements on objects in situ by group of EUREKA E!4981 engineers and scientists such as Fejfarova [11], Rubene and Vilnitis [12-16].

Z-meter III device consists of an electronic and detachable measurement probes. The output data of EIS measurements is divided into the main components of electrical impedance – the $R$ and the $X$ parts are separated in the output data, which brings more opportunities for data interpretation.

Fig.1. Z-meter III device and measurement probes with five active measurement channels (stainless steel elements on measurement probe).

3. Impact of measurement frequency on EIS measurement results

Prior beginning of moisture distribution measurements on autoclaved aerated concrete construction by EIS, a frequency analysis must be performed in order to determine, which frequency is most suitable for the respective material. Selection of a correct measurement frequency has a significant impact on the measurement results. Researches of Rubene et al.[12] prove that if unsuitable frequency is selected, the monitored changes of humidity distribution can be minimal or cannot be monitored at all. It is important to pay attention not only to a single frequency, which seems to be suitable for the measurements but also to a range of frequencies around the selected frequency. This is an important fact because the most suitable frequency tend to change its value along with changes of AAC structure during its drying process.

As stated by Barsoukov and McDonald [6] from the theory of physics capacitances and inductances are generally associated with space charge polarization regions and with specific adsorption and electrocrystallization processes at an electrode. Ordinary circuit elements, such as resistors and capacitors, are always considered as
lumped-constant quantities, which involve ideal properties. All real resistors are of finite size and thus are distributed in space; they therefore always involve some inductance, capacitance, and time delay of response as well as resistance. These residual properties are unimportant over wide frequency ranges and therefore usually allow a physical resistor to be well approximated in an equivalent circuit by an ideal resistance, one which exhibits only resistance over all frequencies and yields an immediate rather than a delayed response to an electrical stimulus.

The physical interpretation of the distributed elements in an equivalent circuit is somewhat more elusive. However, they are essential in understanding and interpreting most impedance spectra. There are two types of distributions with which needs to be concerned. Both are related, but in different ways, to the finite spatial extension of any real system. The first is associated directly with nonlocal processes, such as diffusion, which can occur even in a completely homogeneous material, one whose physical properties, such as charge mobilities, are the same everywhere. The other type, exemplified by the constant-phase element (CPE), arises because microscopic material properties are themselves often distributed. For example, the solid electrode–solid electrolyte interface on the microscopic level is not the often presumed smooth and uniform surface. It contains a large number of surface defects such as kinks, jags, and ledges, local charge inhomogeneities, two- and three-phase regions, adsorbed species, and variations in composition and stoichiometry. Such state can be referred to the situation of AAC sample and Z-meter measurement probe. Reaction resistance and capacitance contributions differ with electrode position and vary over a certain range around a mean, but only their average effects over each channel of measurement probe surface can be observed. The macroscopic impedance, which depends on the reaction rate distribution across such an interface, is measured as an average over each channel of the electrode. Such averaging is usual in one-dimensional treatments (with the dimension of interest perpendicular to the electrode property distributions occur throughout the frequency spectrum). The classical example for dielectric liquids at high frequencies is the bulk relaxation of dipoles present in a pseudoviscous liquid. Such behavior was represented by Cole and Cole [18] by a modification of the Debye expression for the complex dielectric constant.

Furthermore, Tamtsia et al. [19] have discovered that analysis of the spectra provides pore structure information. Their studies allowed to obtain real-time descriptions of microstructural change during creep and shrinkage of cement paste through the coupling of time-dependent deformation and impedance measurements. Impedance spectra recorded over a wide range of frequencies (from 15 MHz to 1 Hz) have provided information and insight on cement paste microstructure and hydration. Cement based materials generally contain a broad size distribution of conducting pores. The network of these conducting pores continuously changes during the drying process. This change can be detected in AC impedance spectra.

Therefore, taking into consideration the findings of Barsoukov and McDonald [6] and Tamtsia et al.[19] it is important to consider possible changes of the ACC material structure. The chemical properties of each AAC type have to be taken into consideration as well to proceed to EIS measurements on the material in order to determine humidity distribution throughout the cross section of the material.

4. Description of the experiment

The experiment was based on monitoring of the differences of preferable measurement frequencies in AAC materials due to the chemical and structural differences of the material.

For this experiment, five different AAC masonry block specimen were chosen. The samples came from different manufacturers and had slightly different densities. These facts allowed assuming that the porous structure as well as chemical structure of the samples slightly differed.

The material samples were prepared for the experiment by the same methodology as stated in Rubene et.al. [13, 16]. There were used sets of four samples from each type of AAC masonry blocks. Three samples were used for
EIS measurements and the fourth sample was used for the determination of initial moisture content throughout the cross section of the AAC block by the approach described by Akita [20].

Measurements of moisture distribution by EIS were performed by Z-meter device and one probe pair, which was inserted in previously prepared bores (Fig.1), each measurement probe, had five measurement channels, which mean that resistance data was obtained in five different depth levels of the samples.

During the measurement phase, several external impact factors on the results of frequency analysis were observed as well. The external factors were the position of the measurement probes – the tests were performed with horizontal and vertical alignment of the measurement probes in the sample; the settling time between EIS measurements; and the humidity state of the measured material.

For the detection of impact that is caused by the changes of samples’ density due to their drying process the frequency analysis was performed several times on each sample in different conditions. Firstly, the frequency analysis was performed on AAC masonry blocks in the state of uneven humidity throughout the cross section of the AAC masonry blocks (Fig.2). The humidity distribution throughout the cross section of the AAC samples was determined by the methodology introduced by Akita (1996) and V0 to V6 stand to number of pieces each part of AAC sample block was cut to.

![Fig. 2. Moisture content throughout the cross section of the samples at the beginning of the experiment.](image)

Therefore, uneven density of the sample due to uneven moisture distribution throughout the cross section of the samples was also evaluated in the results of frequency analysis. The second set of the measurements was performed after the samples had dried in such way determining the impact of humidity distribution in samples on the results of frequency analysis.

5. Results

5.1. Impact of humidity distribution throughout the cross section of the AAC sample on the results of frequency analysis

A separate frequency analysis was performed for each sample. For frequency analysis, the X component of resistance was used because the measurement frequency depends on the AAC material structure. The results of frequency analysis display that for all samples the most suitable frequencies are in range from 6,3kHz to 20 kHz and vary significantly (Fig.3 to Fig.7).

Therefore, it is possible to conclude that the AAC material structure has significant impact on determination the preferable measurement frequency for further monitoring of the material by EIS measurements.
After comparing the frequency analysis data with the information (Fig. 2) of humidity distribution throughout the cross section of the samples, it can be concluded that the density changes of the material, which are caused by moisture content of the porous material have significant impact on the results of frequency analysis. It can be observed that the regions of the samples with higher moisture content have wider frequency ranges, which are suitable for EIS measurements. However, it should be taken into consideration that the samples with large differences of the moisture content throughout the cross section of the sample also had large impedance measurement result differences. Therefore, the moisture distribution measurements should be performed on the samples prior frequency analysis.

![Frequency analysis for sample A.](image)

![Frequency analysis for sample B.](image)

For the first set of frequency analysis all measurements were performed with vertical measurement probe placement. However, during on-site measurements it is often necessary to perform EIS measurements on AAC masonry constructions with horizontal measurement probe placement (e.g. from one side of a wall). Therefore, the impact on the probe placement on the frequency analysis results was researched. The comparison of the results obtained from frequency analysis with vertical and horizontal probe placement (Fig. 8).

The results display that the probe placement has no significant impact on the results of frequency analysis. The maximum range of obtained results does not exceed 5% at the point of the most deviation. It means that the results, which are obtained for correlation equations between the EIS measurement values and the moisture content of the AAC material, such as in Rubene et.al. (2014) can be applied on the EIS measurements for detection of humidity distribution throughout the cross section of the AAC construction in horizontal as well as in vertical directions.
5.2. Impact of settling time between measurements on the results of frequency analysis

EIS is a measurement method, which is based on AC impedance measurements during continuous periods. Therefore, a question arise if the settling time between separate measurements have influence on distinct measurement results or it is possible to neglect the impact of this factor. The test results (Fig. 8.) have displayed that there is no significant impact on the results of frequency analysis and further measurement results. The maximal impact of the differences in settling time on measurement values are not higher than 10%. This factor ease the application of the EIS for the detection of humidity distribution throughout the cross section of AAC masonry constructions.

6. Conclusions

Frequency analysis is an important factor for correct interpretation of the EIS measurement results. It is a mandatory prerequisite to perform one prior starting the monitoring of moisture migration or drying process of the construction.

This research proves that the density changes have significant impact on the results of frequency analysis. Therefore, the frequency analysis should be performed on the material specimen with even distribution of density and humidity throughout the cross section of the sample. If these rules are not respected it may lead to misinterpretation of the EIS measurement data and further to misinterpretation of the moisture migration processes throughout the cross section of the AAC material. The results of frequency analysis prove that the impact of density on the results frequency analysis can reach up to 44% of the measurement value. Therefore, results of frequency analysis should not be neglected in overall process of the EIS measurement process.

Acknowledgments

This research was performed in Riga Technical university, Faculty of Civil engineering. The “Z-meter III” device was invented in framework of EUREKA E4981.

References

Sanita Rubene et al. / Procedia Engineering 108 (2015) 647 – 654


