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Kontrola parametrów mieszanek betonowych i betonów we wczesnym stadium twardnienia za pomocą pomiarów oporności elektrycznej

MONITORING PROPERTIES OF CONCRETE MIXTURES AND CONCRETES AT THE EARLY HARDENING STAGE BY MEASURING ELECTRICAL RESISTIVITY

Streszczenie

Firma Concretec opracowała metodę badania mieszanek betonowych i betonów w oparciu o porównanie wyników pomiarów oporności elektrycznej i standardowych testów. Ciągły pomiar oporności elektrycznej, który rozpoczyna się od momentu przygotowania mieszanki pozwala (w oparciu o wcześniej ustalone zależności) na kontrolę współczynnika w/c, szybkości opadania stożka i jego spowolnienie z upływem czasu, określenie początku i końca czasu wiązania, a także początku procesu krystalizacji w betonie.
System pomiarowy Contest-8 pozwala na kontrolę całego zakresu parametrów procesu w przypadku betonu i mieszanek betonowych zarówno w laboratorium, jak i na placu budowy. Kontrolę wszystkich parametrów przeprowadza się poprzez pomiar oporności elektrycznej pojedynczej próbki, gwarantując rzetelność uzyskanych wyników pomiarów i pozwalając na porównanie różnych właściwości betonu.

Abstract

The new principles of monitoring technology were developed by Concretec Ltd. company based on a comparison between the results of electrical resistivity measurements and the standard test results for concretes and concrete mixes.

Continuous measurement of electrical resistivity starting from a time of mixture preparation allows researchers (on the basis of pre-established correlations) to control the

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W/C-ratio, the cone slump value and its decrease over time, the beginning and the end of setting process, as well as the beginning of concrete crystallization process.

The proprietary measuring system Contest-8 allows monitoring the whole range of process parameters for concretes and concrete mixtures, both in a lab environment and at construction sites. For this purpose, monitoring of all properties is carried out by measuring electrical resistance on a single sample, which increases reliability of the results and allows comparison of various concrete properties.

1. Introduction

Concretec Ltd. company has developed common principles for monitoring the hardening and strengthening of cement-concrete compositions (further only concrete will be considered) and has designed the equipment for implementation of these principles. Physical basis of the method is a continuous evaluation of changes of physical moisture state in hardening concrete based on the results of direct measurements of its electrical resistivity ρ [1, 2, 3].

The concrete hardening is the result of combined development and interaction of chemical and structural transformations. The liquid phase (mixing water) actively participates both quantitatively and qualitatively in the whole complex of these processes.

Along with this, the physical moisture is the sole conductive component of concrete structure at all hardening stages, whereas the solid-phase components are effective insulators (for example, the electrical resistivity of usual dry sand varies between 1500–4200 ohm*m, and the resistivity of dry crushed stone is up to 22000 ohm*m, etc.).

In our approach we use the physical moisture as a key element for the study of complex and even contradictory hardening processes, which is based on the following main principles:

- physical moisture is always in the state of thermodynamical equilibrium with solid surfaces, on which (or by which) it is adsorbed,
- therefore, the moisture state immediately “responds” to all structural and configuration changes of the solid phase.

These provisions allowed us to formulate the basic principle:

The structural level of hardening concrete is characterized by condition of physical moisture – pore solution – that is distributed on the formed (and developing) solid surfaces.

In turn, the change in physical moisture state is evaluated by the rate of changes of electrical resistivity $\rho = f(\tau)$, which is continuously measured during the hardening process.

The above guidelines are used by us to monitor technological properties of concrete at early hardening stages, starting from a mixture preparation until the setting stage is completed and the crystallization processes are started.

2. Physical basis of the method

Electrical conductivity σ (electrical resistivity ρ) of porous medium can be determined from the Nernst-Einstein equation as follows:

$$\sigma = \frac{1}{\rho} = D\frac{(zF)^2 c_i}{RT} \tag{1}$$

where D_i – diffusion coefficient of ion i, z_i – ionic valency, F – Faraday constant, R – absolute gas constant, T – absolute temperature, c_i – concentration of ion i in pore solution.

Diffusion coefficient can be expressed from the Einstein-Smoluchowski ratio as follows:

$$D_i = \frac{RT}{6\pi r N_A \eta} \tag{2}$$

and electrical resistivity can be expressed through dynamic viscosity η and concentration c_i

$$\rho = \frac{1}{\sigma} = \eta \frac{6\pi r N_A}{(zF)^2 c_i} \tag{3}$$

where r – ion radius, N_A – Avogadro constant.

In equation (3), only viscosity and concentration are time τ varying parameters, i.e.

$$\rho(\tau) = \frac{1}{\sigma(\tau)} = \frac{\eta(\tau)}{c_i(\tau)} const \tag{4}$$

Thus, the amount and behavior of electrical resistivity of hardening concrete are determined by interaction of two following factors:

- Chemical – grow of dissociated ions concentration in pore solution $c_i(\tau)$ increases σ and reduces ρ value, and
- Structural – grow in pore solution viscosity η(τ) reduces σ and increases ρ value.

In the process of concrete hardening average radius of pores and average of structured water layer thickness decreases. Accordingly, increases energy of this water – pore solution – binding with solid surfaces. As a result, the viscosity of the conductive phase increases.

That is why dynamic viscosity η(τ) is a structural parameter, determined by concrete composition and hardening process conditions.

3. Measurement system

For practical implementation of the principles developed by Concretec Ltd. Company, the Contest-8 measurement system was designed (Fig. 1), which allows continuously measuring of concrete electrical resistivity and temperature [1,2,3, etc.]. Contest-8 system is suitable for use in the lab conditions, at construction sites and in plants for production of ready mix and precast elements.

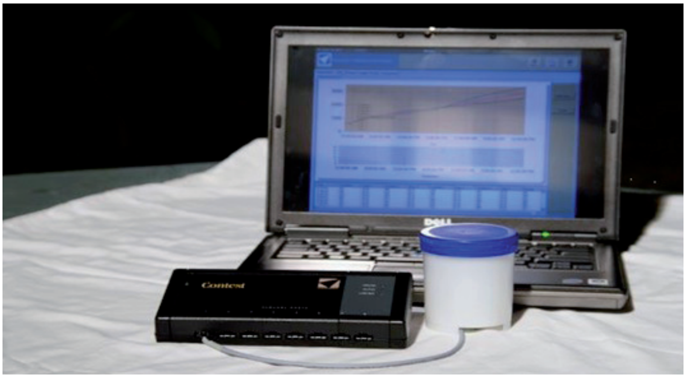

Fig. 1. Contest-8 measurement system

The main Contest-8 system component is an 8-channel Data Logger that is connected to PC and capable of simultaneously reading, processing and transferring information from 8 container-type sensors. The interval between the resistance ρ and temperature *t* measurements can vary from 3 to 30 minutes.

The values are measured using low frequency electric pulses (~70 *Hz*) at the current not exceeding 10 μA. Pulse duration is 10 μsec, periodicity ~100 *m* sec. These current parameters, on the one hand, do not affect the chemical processes of concrete hardening, and, on the other hand, eliminate the polarization effects (electrolysis) on electrodes.

Operating voltage of Data Logger is 5 υ, so, the power is supplied directly from PC; the built-in battery supports standalone operation of the system for about 40 hours.

The measuring system software (user interface) includes a set of control, processing and interpretive programs; all measured results are stored in a database. The results of ρ and *t* measurements are displayed graphically on PC screen in on-line mode.

Monitored material – concrete, cement-sand mortar or cement paste – is placed into a sensor of container type [1, 2]. The material from which the sensor is made, is electrically neutral.

The sensor consists of the following components:

- Cylinder that is filled with a monitored material; for stabilizing the electricity flow between two electrodes mounted on the bottom, the cylinder is divided by special partition. The ratio of partition height *h* to the total sensor height *H* is equal to $h/H=0.65$.
- The bottom cover is rigidly connected to the cylinder; a contact panel is mounted on the inner surface of the cover. On the contact panel are located: (1) the spring contacts that are pressed against the electrodes, and (2) the thermistor that measures the concrete temperature in the range $-40^{o}C \div +150^{o}C$, with accuracy $\pm 0.3^{o}C$.

Depending on a type of tested CCC, the sensors of different volumes are used:

- 36 cm^3 – for testing cement paste and cement-sand mortar,
- 250 cm^3 – for testing concretes with a maximum aggregate size of 12.5 mm,
- 780 cm^3 – for testing concretes with a maximum aggregate size of 37.5 mm.

4. Features of concrete hardening at early stages

Initial state of concrete hardening is always and without exception (!) the "compaction structure"* [4], physical and mechanical properties of which are determined primarily by compression effect of capillary pressures. Value of these pressures depends, in turn, on the initial porosity parameters of concrete immediately after placement. The compaction structure formation is determined by technological properties of the concrete mixture: W/C-ratio and Slump (or consistence).

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* *Without concept of "compaction structure" state there is no sense at all to talk about hardening and structure formation: the state of compaction structure and its properties are the beginning of hardening scale for any concrete.*

Table 1. Mixture's proportions of tested concretes

Mixture No.	**Amounts (kg/m^3)**						**W/C-ratio**
	Coarse aggregates	**Sand**	**Cement**	**Water**	**Admixture**	**FA**	
1	1260	860	290	190	1.7*)	120	0.66
2	996	797	430	176	4.91+2.46***)	---	0.41
3	1206	728	223	176	2.79**)	---	0.88
4	1207	675	279	176	3.49**)	---	0.70
5	1206	619	344	176	4.30**)	---	0.57
6	1224	494	497	179	6.21**)	---	0.40
7	1206	728	134	176	1.67**)	89	0.88
8	1207	675	167	176	2.09**)	112	0.70
9	1206	619	206	176	2.58**)	138	0.57
10	1224	494	298	179	3.73**)	199	0.40
11	1260	860	235	130	3.68*)	94	0.40
12	1260	860	235	173	3.68*)	94	0.53
13	1260	860	235	222	3.68*)	94	0.67
14	1260	860	235	249	3.68*)	94	0.73
15	1320	820	230	135	1.61*)	---	0.59
16	1260	860	270	195	1.61*)	40	0.72
17	1260	860	270	165	1.62*)	40	0.61

Admixtures: *) G100X, **) Supermix – 700, ***) C-3 + ЛСТ-Е

After completion of the compaction structure formation, begin concrete early hardening stage. This stage includes the clinker minerals dissolution step, the induction period and setting processes. Thus, concrete hardening at an early development stage is characterized by the interaction of chemical and structural transformations that are controversial processes by virtue of their physical nature.

Changes in electrical resistivity of various concretes during the hardening process are clearly reflecting the behavior of their structure formation and hardening. Fig. 2 demonstrates the results of measuring $\rho(\tau)$ during 28 days of hardening at constant temperature ($t \cong 20^{o}C$) for mixtures #1 **(**Fig. 2a) and # 2 **(**Fig. 2b) (Table 1).

We can see that the behavior of kinetic curves $\rho = f(\tau)$ is defined by steady increase in electrical resistivity of the concrete until it reaches of final 28-day strength.

However, in the context of our purpose we are interested in the initial phase of hardening. The results presented in Figures 3a and 3b show that the value $\rho(\tau)$ at the initial stage is changing in significantly non-monotonic manner.

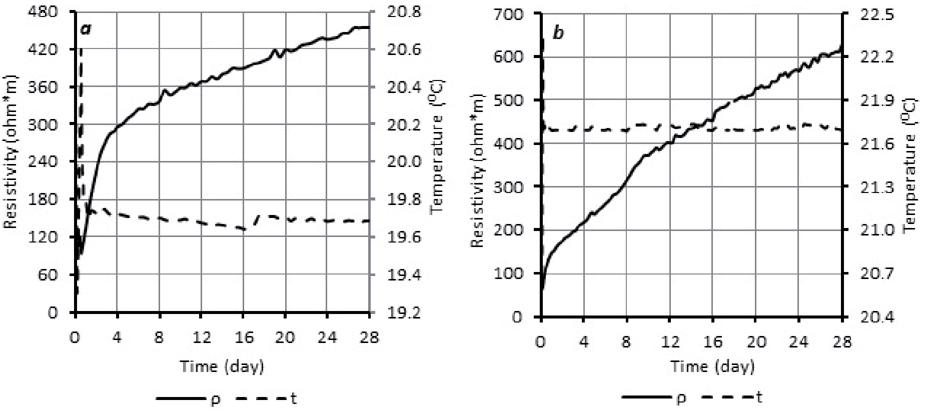

Fig. 2. Changes in the electrical resistivity and temperature during 28 days of concrete hardening: a – Mix 1, b – Mix 2

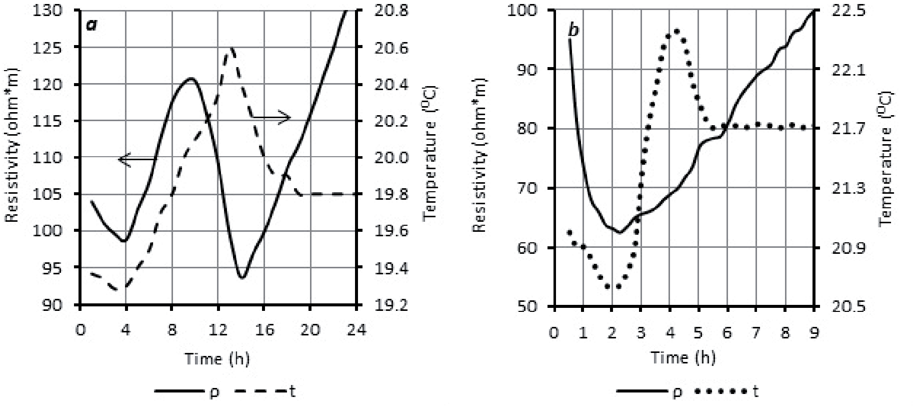

Fig. 3. Changes in the electrical resistivity and temperature at early stage of concrete hardening: a – Mix 1, b – Mix 2

Non-monotonicity of $\rho = f(\tau)$ curves is due by mutual development and interaction (or more accurately, counteraction) between the chemical process of clinker minerals dissolution and the colloidal-chemical processes of primary coagulation structure development during induction period, and of coagulation-crystallization* structure development during concrete setting processes.

In both cases, the initial reducing of electrical resistivity on the curves $\rho = f(\tau)$ at ($\tau = 0 \div 4.0$ hr. for Mix #1 and $\tau = 0 \div 2.5$ hr. for Mix #2) describe the process of clinker minerals dissolution. So, at this stage the measure and behavior of changing ρ value mainly affected by chemical factor: increasing of pore solution ionization, i.e. $\rho(c_i) > \rho(\eta)$.

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* *Crystallization at the early hardening stage mainly related to the formation of ettringite* $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot (30–32)\ H_2O$ *and portlandite* $Ca(OH)_2$ *minerals.*

As the chemical reactions develop, the concentration of reaction products – microdisperse particles (specific surface area 200–750 m^2/g and more [6]) – is increasing. Consequently, the capillary liquid (pore solution) is constantly reallocated on the newly formed solid surfaces. As the result, an average thickness of physical moisture layers is decrease, the impact of viscosity of conductive phase on the value and behavior of changes in ρ becomes dominant, i.e. $\rho(\eta) > \rho(c_i)$. As results electrical resistivity begins to increase.

After $\tau \approx 0 \div 4.0$ hr. (for Mix #1) and $\tau \approx 0 \div 2.5$ hr. (for Mix #2), the impact of chemical processes is balanced by primary structural effects: interactions between solid particles (reaction products) through the water films and interlayers, the thickness of which is constantly decreasing. Then the state of quasi-equilibrium occurs $\rho(c_i) = \rho(\eta)$, which is represented by first minimum on the curves $\rho = f(\tau)$ (Fig. 2a and 2b).

The inflexion points ρ_{1min} define the beginning of setting process, but the time $\tau(\rho_{1min}) < \tau(IS)$ (*IS* – initial setting time according to ASTM C-403).

Behavior of the subsequent changes in electrical resistivity for mixtures # 1 and # 2 is of the essence different:

- For mixture #1 the curve $\rho = f(\tau)$ is a broken line that is characterized by presence of well-defined one maximum and two minimum points (Fig. 3a) – in such case the consistent rise ρ begins after crossing the second minimum point (at time point $\tau_{2min} = 14.0$ hr.).
- However, for Mix #2 there is only one minimum on the curve $\rho = f(\tau)$ (at time point $\tau_{1min} = 2.3$ hr.). Then the resistivity dynamically increases until $\tau \approx 5.0$ hr., then a temporary stabilization begins, and after $\tau \geq 6.0$ hours it has continued by steady growth in the resistivity value (Fig. 3b).

The reasons for non-monotonic change of electrical resistivity value will be analyzed in Section 6.

5. Concrete ready mixes properties

5.1. Water/Concrete ratio evaluation

The following two possible ways for changing the W/C-ratio of the concrete mixture where considered (Table 1):

a – by increasing the amount of cement with a constant amount of water (mixes ##3 – 10) (Fig. 4a);

b – by increasing the amount of water with a constant amount of cement (mixes ##11 – 14) (Fig. 4b);

When the case "a" ($C \rightarrow max$, $W = const$) is used, the electrical resistivity increases, which corresponds to the result obtained by authors of [7].

It is clear that the increasing of ρ value in this case is caused by chemical factors. Particularly, the increasing of W/C-ratio in the mixtures ##3 – 10 (Table 1) is obtained by decreasing the amount of cement. In the volume of pore solution, this causes a corresponding decrease in concentration of dissociated ions c_i (carriers of electric charge) and $\rho \rightarrow max$.

Conversely, when the case "b" ($C = const$, $W \rightarrow max$) is used, the electrical resistivity decreases with increase of W/C-ratio (mixtures ##11 – 14 in Table 1). It can be assumed that, with a constant amount of cement, the concentration of dissociated ions will be $c_i = const$. In this case, the observed decrease in ρ is caused solely by a change in physical properties of pore solution, specifically by decrease of its viscosity η due to dilution of the concrete mix with water, as a result of W/C-ratio grow.

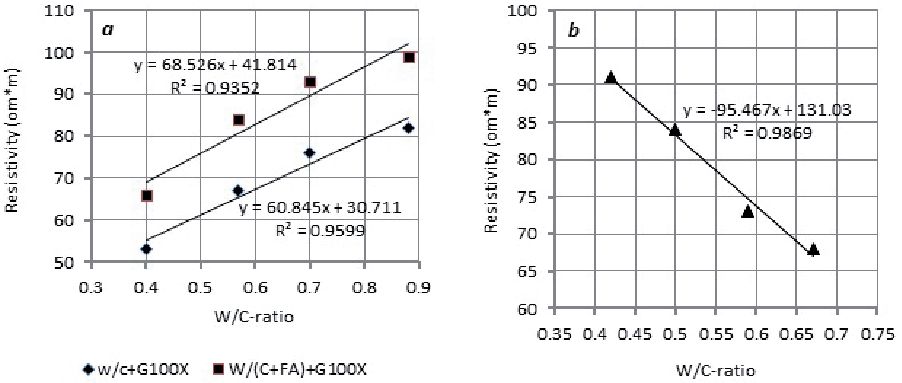

Fig. 4. Change in electrical resistance of concrete depending on W/C-ratio:
$a - C \rightarrow max$, $W = const$, $b - C = const$, $W \rightarrow max$

5.2. Cone slump and its changing by time

The main technological property of the concrete mixture during transportation and forming (i.e. when the "compaction structure" is created) is remoulding effort, which is determined from a slump value of the standard cone.

The value of slump is regulated by technical specification for concrete works. The most important is the ability of a concrete to maintain a predetermined slump value as long as possible, in order to provide maximum convenience while placing the concrete. Therefore, the prompt performing of non-destructive testing, in order to control changes in slump value, enables improvement of construction work efficiency.

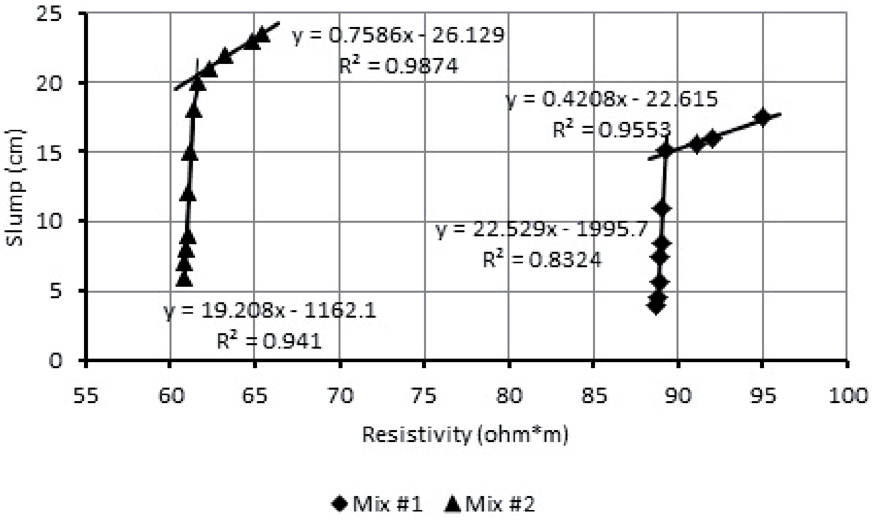

Fig. 5. Correlations between Slump and Electrical Resistivity for different concretes

Fig. 5 shows the correlations *"Slump – Re sistivity"* for the concrete brand B-30 (Mix #1, Table 1) (ready mix plant, Israel) and brand B45 (Mix #2, Table 1) (ready mix plant, Russia).

In both cases, the characteristic curves *Slump* – ρ are approximated by two linear sections:

- The first section is characterized by significant changes in the ρ value (due to intensive development of chemical reactions and increasing the concentration of dissociated ions in the pore solution) with little loss in slump value, i.e. at this section the remoulding effort of concrete mixture is maintained at a good level, allowing to carry out the required construction process steps.
- Conversely, the slump decreases sharply at the second section with minimal change in electrical resistance, which is caused by the development of structural effects, namely by formation of hardening coagulation structure (see Section 3).

The intersection points on the graph lines *"Slump – Re sistivity"* (corresponding to resistance value ρ = 89.3 ohm*m for Mix #1 and ρ = 61.8 ohm*m for Mix #2) are the concrete mixture status points, after which some operations (mixture feeding using a concrete pump, remoulding and placing and compaction in form) are significantly hampered.

Thus, the continuous measuring of electrical resistivity ρ at the early hardening stage allows us to control the slump value decreasing by time, which is very important for concreting operations in construction sites, as well as for manufacturing reinforced concrete elements.

6. Setting process monitoring

The finish phase of early concrete hardening stage is the setting process. The general setting pattern (discussed in Section 3) is determined by the coagulation and coagulation--crystallization processes of structure formation. The formation of ettringite and portlandite primary crystal structures also contributes to the development process.

In modern concrete studies, the results of electrical resistance measurements for cement-concrete compositions are used to study the setting processes [8, 9, 10].

Our methodology for monitoring of concrete setting is based on the analysis of curves non-monotonicity $\rho = f(\tau)$. However, the start of setting process (not to be confused with the *IS* according to ASTM C-403, which is observed much later!) is represented by a first minimum on the curve $\rho = f(\tau)$. Value ρ_{1min} determines the state of quasi-equilibrium $\rho(c_i) = \rho(\eta)$, wherein the action of ions concentration (chemical effects), which reduce the ρ value, is balanced by counteraction of increased viscosity of the conductive phase (structural effects), which increase the ρ value.

The subsequent enhancement of structural effects is derived from the formation of setting structure that precedes to the crystallization structure formation. As result, the impact of the structural factor (increasing of pore solution's viscosity) becomes predominant: $\rho(\eta) > \rho(c_i)$, and the electrical resistivity rises until it reaches the maximum value ρ_{max}.

Temporary decrease in resistivity of mixture #1 (Fig. 2a) and resistivity retardation for mixture #2 (Fig. 2b) on curves $\rho = f(\tau)$ can be related to $\rho(c_i) > \rho(\eta)$:

- on the one hand, the development of cement gel syneresis that is resulting from its contraction due to the intensive formation of calcium silicates and entering additional amount of water to the conducting phase,
- on the other hand, the reason for non-monotonicity ρ is reshaping of the setting structure during changing the structure formation mechanism from coagulation-crystallization

to condensation-crystallization process. As result, the total area of solid surfaces is reduced, the physical moisture is redistributed, and the thickness of moisture layers is correspondingly increased. The combination of these effects also causes a temporary drop of ρ value.

Based on the results of curves $\rho = f(\tau)$ analysis and their comparison with the results of determining the initial and finish time for setting process using the standard Proctor method (ASTM C-403), our method has been proposed [3], according to which the setting initial time (*IS*) is defined as a time on set of a fasted rise of resistivity, and the setting finish time is as a time of local maximum of resistivity, i.e. $FS = \tau(\rho_{max})$.

In most cases, time to maximum rate of change in resistivity curve can be defined with sufficient accuracy as follows:1

$$IS = \frac{\tau(\rho_{\min(1)}) + \tau(\rho_{\max})}{2}$$

In Figure 6, the curves $R = \psi(\tau)$ for determining concrete setting time using the standard Proctor method are correlated with the curves of electrical resistivity change $\rho = f(\tau)$ for the same concrete. These results were obtained during the concrete mixtures testing in production of large size units (~5 м^3 in volume) for hydro-engineering constructions.

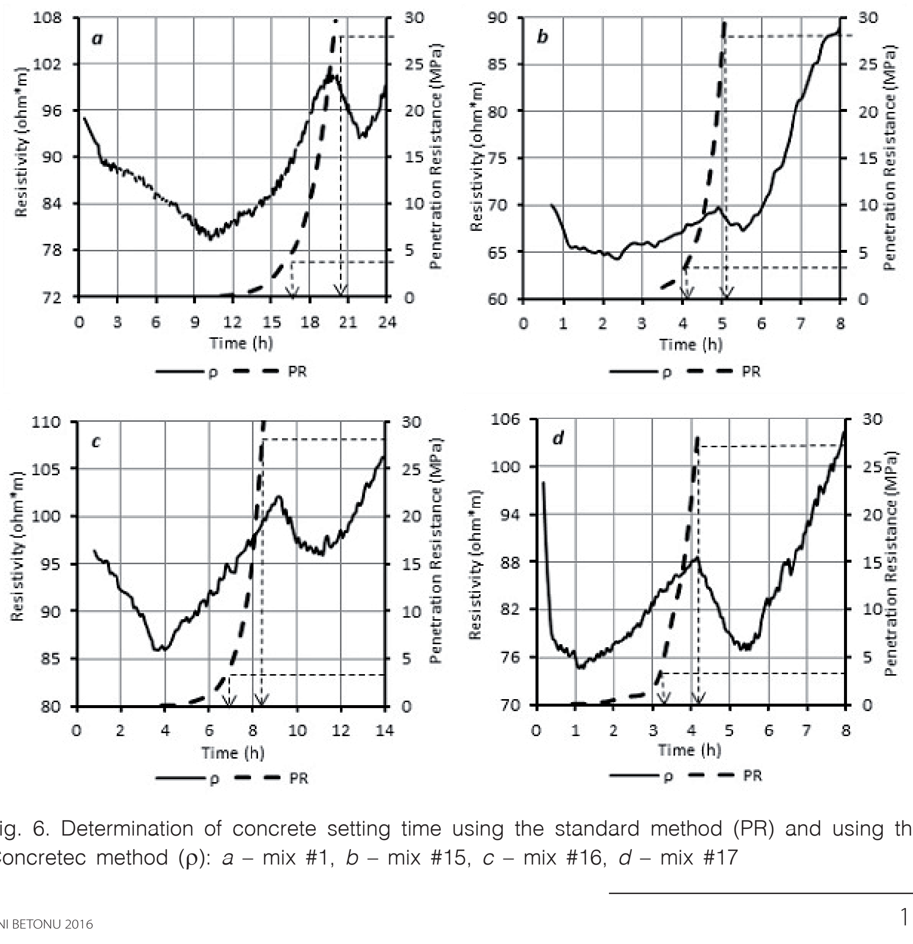

Fig. 6. Determination of concrete setting time using the standard method (PR) and using the Concretec method (ρ): *a* – mix #1, *b* – mix #15, *c* – mix #16, *d* – mix #17

The following interesting fact will be observed: in all cases (Fig. 6a, b, c, d), the start points on the curves $R = \psi(\tau)$ virtually coincide in time with the first minimum on the curves $\rho = f(\tau)$. This confirms the above assumption (see Section 3) that the value ρ_{1min} specifies an equilibrium state, which is completing the induction period and beginning the setting process.

Consequently, the ρ_{1min} parameter has a specific technological sense: "...provides a time in which concrete can be transported and placed before setting." (Scrivener and Nonat [5]).

Fig. 7 shows comparative data of the early concrete hardening monitoring for concrete brand B-30 with various concentrations of Supermix-700 admixture (setting retarder). The studies were performed in the laboratory of National Building Research Institute (Technion, Haifa) [11]. The graph clearly shows that, in accordance with the increase in admixture concentration, the inflection points on the kinetic curves of electrical resistivity are shifted to the right on the time axis.

Comparison of the inflection points on the curves $\rho = f(\tau)$ with the results of standard evaluation of *IS* and *FS* demonstrates good level of agreement between the results obtained by both methods.

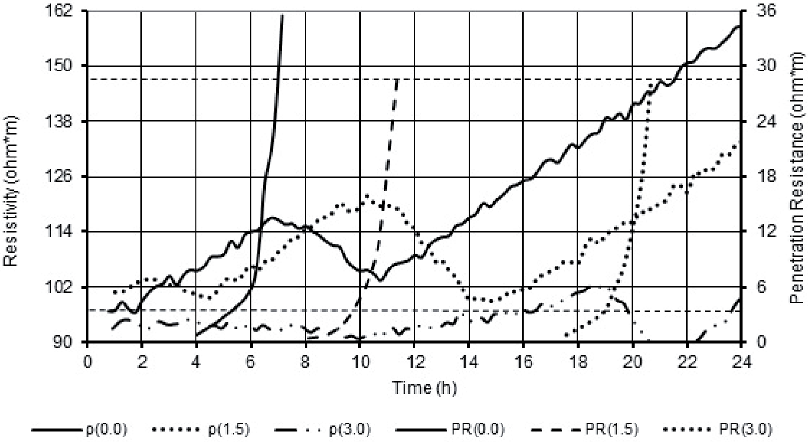

Fig. 7. Determination of the setting time for concrete (mix #4) with 0,0%, 1,5% and 3,0% of Supermix-700 additive using the standard method (PR) and using the Concretec method (ρ)

Table 2. Comparative results of the concrete setting times determination

Mixture (# from Table 1)	**IS** (h)		**FS** (h)	
	by Proctor	by Concretec	by Proctor	by Concretec
#1	15.78	14.95	19.8	19.8
#15	4.15	3.55	5.1	4.9
#16	6.75	5.95	8. 5	9.1
#17	3.3	2.8	4.25	4.4
#4(0.0%)	4.2	6.8	7.03	6.54
#4(1.5%)	9.76	7.2	11.05	10.15
#4(3.0%)	19.0	14.65	21.1	19.3

The summary of setting time results (Fig. 6 and Fig. 7) is shown in Table 2. In all cases, there is close fit between the results obtained by Proctor method (ASTM C-403) and by the method based on electrical resistance measurements [3].

Furthermore, the method of electrical resistivity measurement allows us to determine the setting time of real concrete mixtures, i.e. without separation of coarse aggregate.

7. Universal character of the curve $\rho = f(\tau)$

During the early stage of concrete hardening – from the moment of compaction structure' forming, and up to completion of the setting process – the structural and mechanical condition of concrete is constantly changing. In view of this, the standard testing of concrete mixture parameters is performed by essentially different methods using samples of different geometric shape and volume. This is why the results of standard tests virtually no correlate with each other.

This drawback can be eliminated by using the method bases on monitoring of early concrete hardening through the continuous measurement of its electrical resistivity.

In Fig. 8, the results of standard tests of process parameters for concrete mixture #1 [Slump (Fig. 5), Vebe$^{*)}$ and Setting times (Fig. 6)] are compared with the curve $\rho = f(\tau)$. Consistence of a concrete mixture by Vebe method was tested specially for this purpose: the obtained results define an intermediate structural state of the concrete after declining of slump properties and beginning of setting process.

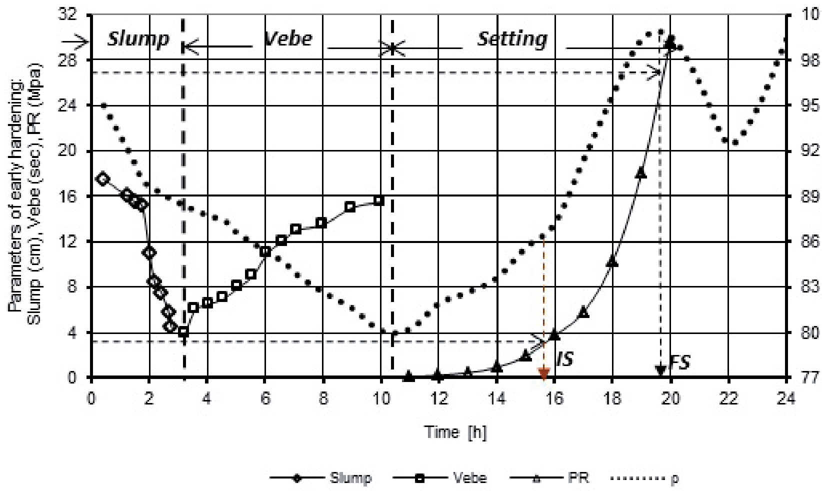

Fig. 8. Comparison of the standard test results for concrete mix #1 with the results of electrical resistivity measurements

It can be seen that the kinetics and the behavior of electrical resistivity clearly corresponds to the regular pattern of early concrete hardening process. The inflection points on the curve ρ = *f* (τ) determine the sequence of changes in the structural and mechanical state of the material:

- Viscous-flow state (Slump test),
- Viscoplastic state (Vebe test),
- Plastic hardening (Setting time test).

8. Conclusions

New principles for monitoring the concrete hardening at early stage, which are based on a continuous measurement of electrical resistivity, allowed to establish the following:

- The dependence "W/C-ratio – Resistivity" can be approximated by linear correlations, and
  - The electric resistance decreases in the case of adding amount of water at a constant amount of cement,
  - The electric resistance increases in the case of adding amount of cement at a constant amount of water,
- The dependence "Slump – Resistivity" can be approximated by a broken line consisting of two segments; the intersection point of these segments determines the state of concrete mixture, after which the process operations such as feeding mixture using a concrete pump, pouring and compaction in formwork are significantly hampered.
- Non-monotonic change in electrical resistivity during the early stage of concrete hardening is caused by the development of setting processes. There is close fit between the results obtained by the Proctor method (ASTM C-403) and by the inventive method used for determining *IS* and *FS* of different concretes.
- Continuous measuring of electrical resistivity at the early hardening stage is the universal method that can be used for monitoring the concrete structural and mechanical state and the behavior of basic technological parameters.

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