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Electrical and Thermal Properties as an Indicator for Setting Time of Concrete

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Abstract - The process, rate, and quality of cement hydration affect the properties development of concrete, such as its compressive strength, penetrability, and setting. Among all, setting time is one of the important properties for the concrete industry. The accurate determination of the setting time in concrete projects has always been a question. Setting time is traditionally measured in laboratory setups using the Vicat needle (as standardized in ASTM C191). However, the field condition of a concrete pour is different from a laboratory setup, so it is important to replace the lab-based setting time test with an alternative method applicable to the field. Proposing a test method, which not only estimates the setting time but also considers the actual job site conditions, is essential. In this paper, the development of setting time using the Vicat method of three cement paste mixtures is studied for the first 24 hours from mixing. The electrical resistivity and temperature evolution of the cement pastes are monitored during the same period. From the results, it is observed that the changes in the electrical resistivity in conjunction with changes in the temperatures of the fresh cement pastes are related to the setting times as measured by ASTM C191. Correlation between the changes in the electrical resistivity and temperature of the cement pastes with the setting times development can result in a practical method for estimating the in-place field setting properties.

Keywords**:** Cement Paste, Vicat Test, Setting Time, Fresh Properties, Electrical Resistivity.

1. Introduction

Concrete is a complex composite material, whose micro and macro structures change over time. The process, rate, and quality of cement hydration (as a complex physical-chemical process) affect the matrix of concrete and therefore its properties. These properties are important for concrete construction, as it influences its performance and long-term durability. Among all properties of concrete, the setting time is an important one. The concrete setting time determines the consolidation, finishing, and curing time of a concrete pour. Inadequate determination of the concrete setting time will result in improper finishing, the formation of cold joints between layers of concrete, and curing time delay. As a result, short and long-term performance of concrete, concrete durability, and its life span will be affected. The concrete setting time proceeds from the initial stage (end of concrete pouring and consolidation) to the final stage (end of finishing process and beginning of curing) with the hydration of cement particles in the concrete matrix. Currently, there is no standard test to measure the beginning of the in-place concrete setting time by considering field conditions. Monitoring the setting time of concrete by the only available lab-based standard testing methods for cement pastes (ASTM C191) or mortars (ASTM C403) is neither adaptable nor completely applicable to actual field projects.

This paper presents a practical and non-destructive technique to estimate the actual concrete setting time by monitoring the development of its electrical resistivity over the time of hydration. Since the factors influencing the electrical resistivity of the cement paste (responsible for the electrical properties of the concrete in the fresh stage) are similar to the factors influencing concrete setting time, it can be theoretically concluded that the electrical resistivity can be used as an indicator for setting time. Three mix designs with three water-to-cement ratios using General Use (GU) cement were studied in this project. The development in electrical resistivity, temperature changes, and setting times of the mixtures are monitored from the time the cement particles become in contact with water until the first 24 hours, which is the conventional time of formwork removal in construction.

2. Testing Methodology

Theoretically, it can be assumed that the electrical properties of fresh concrete (mixture from mixing time to final setting) are influenced only by the electrical properties of the cement paste portion of the mixture. This assumption is based on the fact that the electrical resistivity of aggregates is 106-1,010 times greater than that in fresh cement pastes [1]. Therefore, the electrical properties of fresh concrete are similar to the electrical properties of its fresh cement paste [2]. In this study, the electrical resistivity, temperature, and setting times of GU cement pastes with water-to-cement (w/c) ratios of 0.37, 0.40, and 0.43 were monitored during its first 24 hours of hydration. All cement paste mixtures were mixed in four steps according to the standard method described in ASTM C305 [3].

2.1. Cement Paste Setting Time Test (ASTM C191)

The initial setting time is an indication of the critical stage after which cement paste fresh properties, such as workability, decrease. The final setting time is the time the cementing-based material is not finishable anymore and curing must be started. The initial and final setting times of 650 g of cement paste can be determined using the Vicat needle (Fig. 1) as described in the standard ASTM C191 [4]. The test is conducted at room temperature (22-23°C) and relative humidity (50%).

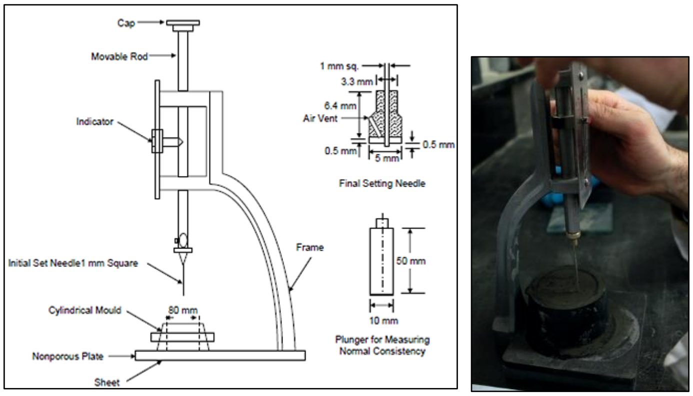

Fig. 1: Cement paste setting time test apparatus (ASTM C191).

The initial setting time is determined when the Vicat needle penetrates 25 mm into the cement paste sample. If the 25 mm record is missed, interpolation is required. The initial setting time is the beginning of the cement solidification. At this time, the cement paste becomes unworkable, so concrete placement and consolidation become difficult to perform [5]. Final finishing must be started at the initial setting time after placement, consolidation and initial finishing are completed. The final setting time is the time to the nearest 5 minutes at which the needle does not mark the specimen surface with a complete circular impression and does not penetrate the sample. This time is the completion of the cement paste solidification. Cement curing can start at this stage since the absorbed moisture cannot contribute to the mix design and increase the w/c ratio.

ASTM C191 [4] has several limitations; it does not consider environmental and construction conditions, aggregate effects, and additives influence the parameters. In addition, its results are influenced by the cement paste preparation and laboratory conditions. Also, human error is common during these tests, as all measurements are recorded by the visual reading of the operator. Therefore, replacing it with an in-situ measurement method is beneficial.

2.2. Fresh Stage Electrical Resistivity and Temperature

Among all electrical properties, electrical resistivity is independent of specimen and electrode geometry. In this study, 110-mm long stainless-steel electrodes were embedded in the cement paste after casting the mixture (in a Ø100×200mm cylinder), and the electrical resistance of the paste in between the electrodes was measured (Fig. 2). To avoid any polarization effects, AC impedance was employed with a frequency of 10 kHz. The meter was connected to a cell phone via Bluetooth to record the data in the designed application. At the same time, the Adiabatic Temperature (AT) of the cement paste was measured using the Fisher Scientific Traceable 255NA thermometer and recorded for the first 24 hours of hydration, as shown in Fig. 2. All mix design ingredients were at room temperature before starting the mixing procedure, so the initial temperature of the mixtures was 22-23°C.

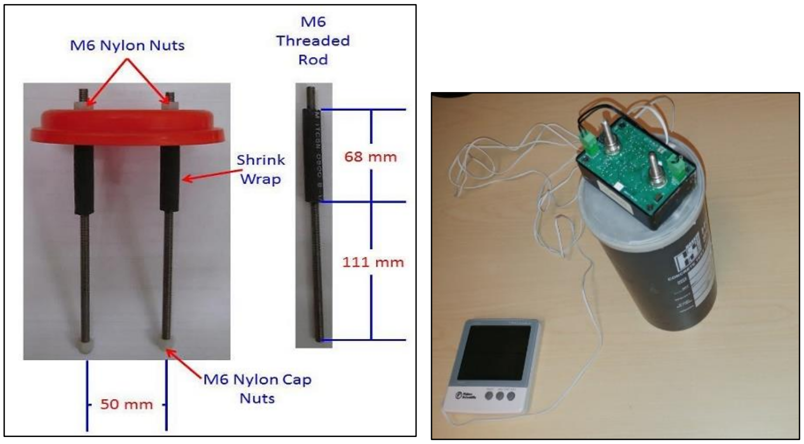

Fig. 2: Electrodes and fresh cement paste electrical resistivity and temperature setup (photo courtesy of Giatec Scientific Inc.).

The electrical resistance measured by the meter was converted to electrical resistivity, which is independent of the sample geometrical properties, by multiplying it by a geometric conversion factor *K*. By considering the current density flux, electrodes size/spacing, and the assumption that the electrical field around the electrodes forms a half-cylindrical surface, the conversion factor was mathematically calculated as per Eq. (1):

$$K = \frac{\pi h}{\ln r} \tag{1}$$

where h is the length of the electrode (h = 11 cm), and r is the spacing between the electrodes (r = 5 cm). Based on the test setup illustrated in Fig. 2, the conversion factor K = 21 cm. The calculated K was calibrated by comparing the electrical resistivity and resistance of a known solution of NaCl.

3. Test Results

3.1. Cement Paste Setting Time

The initial and final setting times of all GU cement paste mixtures were measured according to the method described in Section 2.1. For the initial setting time, the penetration was calculated as the average of ten measurements in a 35×35 mm area. The initial and final setting times of the GU mixtures are shown in Fig. 3. Both initial and final setting times were influenced by the w/c; both setting times increased as the water content of the mixture increased.

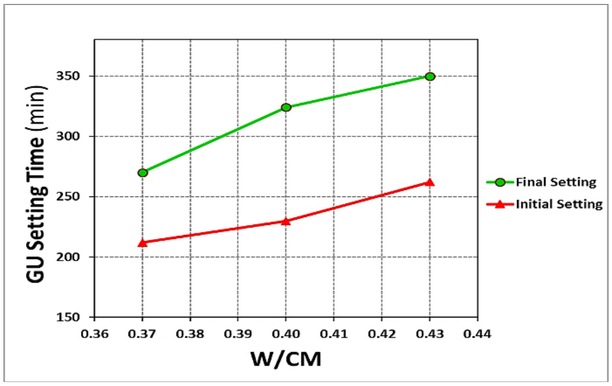

Fig. 3: Initial and final setting times of GU cement paste mixtures.

3.2. Cement Paste Electrical Resistivity and Temperature

The electrical resistivity of fresh cement paste was measured from time zero (immediately after mixing) until the final setting time. Figure 4 illustrates the electrical resistivity evolution with time for each of the three mixtures. It is noted that the electrical resistivity of cement pastes develops over time. At the beginning of cement hydration, a reduction in the electrical resistivity is observed, which translates into an increase in the conductivity of the cement paste mixture. This reduction indicates the dissolution of ions (Na^+, K^+, Ca^{++}, OH^-, SO_4^{2-}) from the cement into the pore solution [6]. In this stage, the un-hydrated cement particles contribute to the electrical resistance of the cement paste. This reduction continues until the leached ions start contributing to the formation of some cement hydration crystals. It is observed that as the w/c ratio increases, the electrical resistivity of the cement paste decreases. The highest resistivity was observed for the cement paste with the lowest w/c = 0.37. The effect of the w/c ratio was not significant at an early age for the first 6 hours, while the effect of water content in the mix design started influencing the electrical properties of the cement paste around the initial setting time.

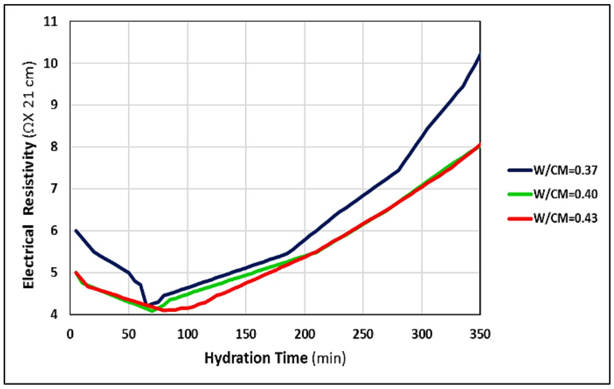

Fig. 4: Electrical resistivity changes of GU cement paste mixtures.

Since the initial temperature of all mixtures was the same (room temperature, 22-23°C), and it is assumed that there is not thermal energy transfer between the cement paste specimen and the environment, the only factor affecting the specimen temperature is the cement hydration exothermic energy. The internal temperature of the GU mixtures over the first day of hydration is illustrated in Fig. 5. It is observed that the temperature of the cement paste mixtures remained almost the same over the first 2-3 hours of hydration, corresponding to the dormant period of cement hydration. After the dormant period, the rate of the cement hydration (and the rate of increase in the samples' internal temperature) increased sharply. Close to the peak, the hydration becomes diffusion-controlled, and the rate of hydration slows down. After reaching a maximum, the temperature starts decreasing due to heat dissipation. Theoretically, the rate of cement hydration is very high during the first several minutes of hydration due to the dissolution of the ions into the mixing water [7]; the same reason for a rapid drop in the electrical resistivity of the mixture right after mixing. However, the rise in the initial temperature of the GU mixes was not significant during that time, although the first reading happened very early right after mixing, about 4 minutes. It can be concluded that the dissolution of ions in the water affects the electrical properties of the mixture much more significantly than the rate of hydration before the dormant period.

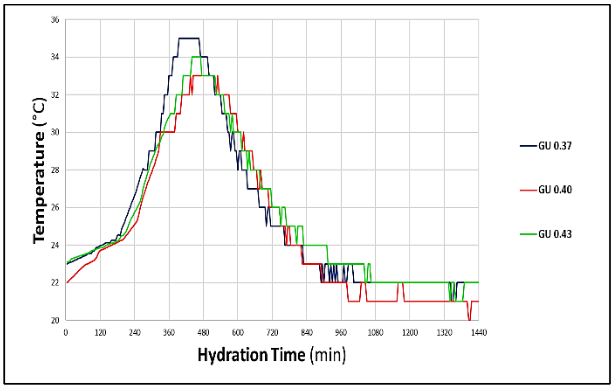

Fig. 5: Internal temperature of GU mixtures over the first 24 hours of hydration.

The rate of change in adiabatic temperature (dT/dt) of the mixtures, calculated from the first derivative of the temperature with respect to time, is tabulated in Table 1. It is also observed that mixtures with lower w/c ratio resulted in higher temperatures over the first 24 hours of hydration; however, the effect of w/c ratio on the dormant period was not significant. In addition, the peak of the Adiabatic Temperature Rise (ATR) was delayed as the w/c ratio increased.

Table 1: Rate of adiabatic temperature change over the first 24 hours of hydration.

Mix Design		Average dT/dt (°C/ min)	
Cement	w/c	Dormant to peak	Peak to control temperature
GU	0.37	0.041	-0.028
	0.40	0.034	-0.024
	0.43	0.031	-0.021

To review the effect of temperature, the measured electrical resistivity values can be normalized by method using Arrhenius law [10].

4. Discussion

By analysing the electrical resistivity and its Rate of Change (ROC) over the first 24 hours of cement paste hydration, four major stages of hydration can be detected. These stages, shown with vertical dash and solid lines in Fig. 6, are.

- Stage I – Dissolution and stiffening: from the mixing time to the first turning point in the electrical resistivity (transition point from decreasing to increasing resistivity), which is shown with the first dash line in Fig. 6.
- Stage II – Setting: stage over which resistivity starts to increase slowly. This stage starts from the turning point in the electrical resistivity to the final setting time (second solid line in Fig. 6). The initial setting time is indicated with the first solid vertical line in Fig. 6.
- Stage III – Hardening: this stage starts from the final setting time to the point where $y''_{\rho} = 0$, which is shown with the second vertical dash line in Fig. 6. This is the point when the momentum of the electrical resistivity value starts to decrease due to the discontinuity of the pores in the microstructure.

- Stage IV – Strengthening: from the second dash line to the end of the 24-h hydration period, in which the cementing-based material develops its mechanical strength. In this stage, the electrical resistivity increases at a very slow rate.

Similarly, a comparison between the electrical resistivity values and the AT of the cement paste samples over the first 24 hours of hydration shows similar stages of hydration:

- Stage I – Change in temperature: From the mixing time to almost the first 2 hours of hydration, indicated by the first vertical dash line in Fig. 6. During this stage, the temperature of the cement paste mixtures increased by 1.0 to 1.5°C. This stage is similar to the dissolution stage during hydration.
- Stage II – Heating: This stage starts around 2 hours of hydration to somewhere between the initial and final setting times (2 vertical solid lines in Fig. 6). In this stage, the temperature of the cement paste mixtures increases significantly due to the hydration of cementing materials particles. This stage is similar to the setting stage during hydration.
- Stage III – ROC reduction: from the final setting time to the peak in internal temperature (shown with the second dashed vertical line in Fig. 6). During this stage, the ROC starts to decrease; however, the AT is still increasing due to the hydration of the cementing material. This stage, which is similar to the hardening stage of hydration, ends when the AT of a cement paste mixture starts to decrease.
- Stage IV – Cooling: from the peak of the AT to the end of the measurement at 24 hours of hydration. During this stage, the temperature of the cement paste samples decreases until it reaches equilibrium at room temperature (22-23°C). This thermal behaviour indicates the strengthening stage of hydration.

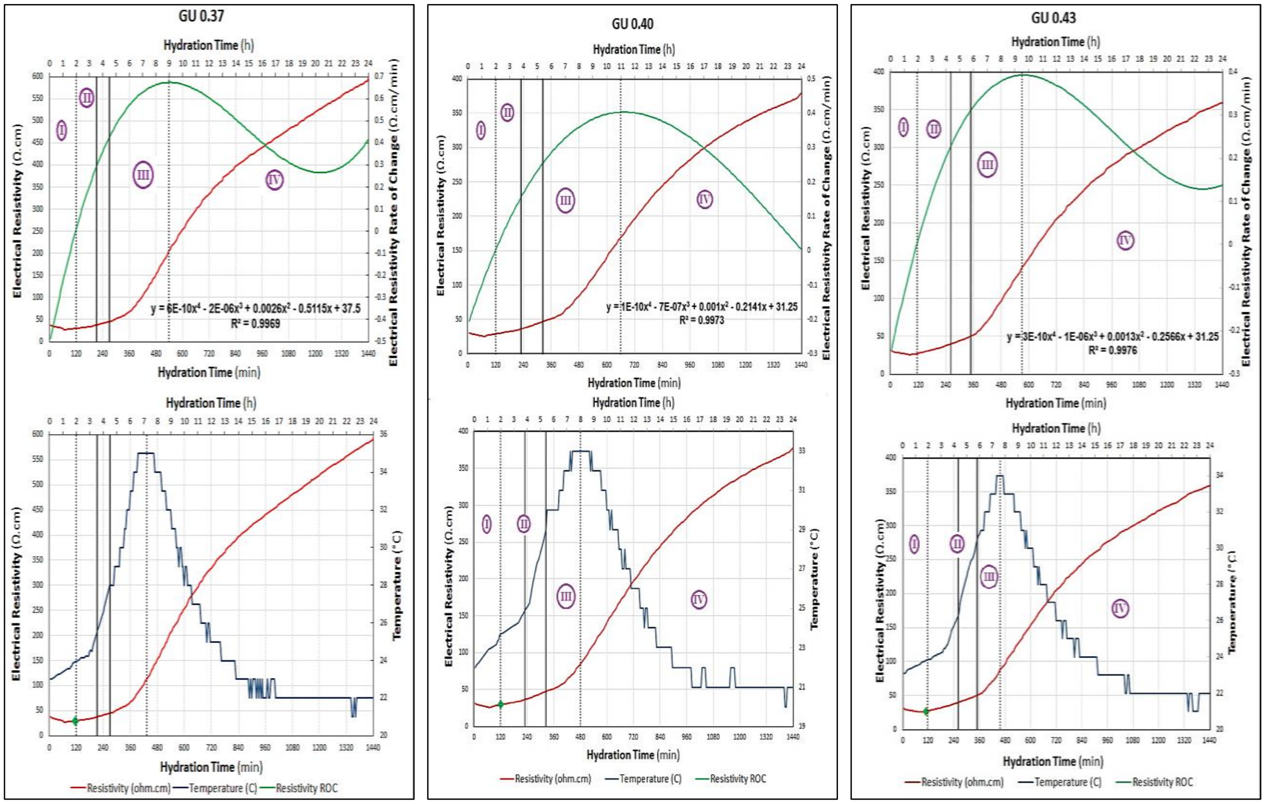

Fig. 6: Four stages of hydration based on the electrical resistivity ROC (top graph) and temperature development (bottom graph) during the first 24 hours of hydration.

During Stage I of hydration (the dissolution stage), as observed in both the electrical resistivity and the temperature graphs, the dissolution of ions resulted in a decrease in the electrical resistivity of the cement pastes, but no significant effect was measured in the internal temperature changes. Therefore, it can be said that ionic dissolution can be detected accurately by monitoring the electrical properties of the cement-based samples, but it cannot be detected by monitoring the internal temperature changes. At the end of Stage I (around 2 hours of hydration), the internal temperature of the mixtures starts mixtures starts increasing again, and the Stage II of hydration begins (setting stage). In Stage II of cement hydration, both the internal temperature and the electrical resistivity of the mixtures are influenced by the hydration. As it is observed in Fig. 6, during this stage (from the stiffening time until the final setting time), both the AT and electrical resistivity increase. From the electrical resistivity point of view, this important stage starts when the rate of change in the electrical resistivity value of the cement paste mixtures becomes zero. The same trend was noted in the temperature graphs, as the beginning of Stage II is the time when the temperature of the mixtures started to increase. The electrical resistivity and temperature values at the initial and final setting times are tabulated in Table 2.

Table 2: Electrical resistivity and internal temperature values at setting times of cement pastes.

Cement	w/c	Initial setting		Final setting	
		Electrical resistivity (Ω·cm)	Temperature (˚C)	Electrical resistivity (Ω·cm)	Temperature (˚C)
GU	0.37	37.88	25.53	45.31	28.08
	0.40	36.40	24.89	47.38	29.04
	0.43	39.84	26.47	50.35	30.49

At the end of Stage II, a sharp increase in the electrical resistivity of the cement paste is observed. This point is around the final setting time. During Stage III, the internal temperature of the mixtures raised significantly compared to that in Stage II. This stage is beyond the final setting time, and it can be said that the physical behaviour of the cement-based mixtures is changed from setting to hardening. In Stage III, the increase in the electrical resistivity value of the mixtures becomes more significant. As observed in Fig. 6, the ROC in the electrical resistivity starts to increase into positive values from zero at Stage II until the end of Stage III. If the electrical resistivity development is considered as a factor, Stage III ends when the slope of the ROC becomes zero. However, it has been observed that the AT is a better indicator for determining the end of Stage III at the maximum internal temperature value, where the ROC in the temperature value becomes zero. In Stage IV of hydration, the ROC in the electrical resistivity of the cement paste mixtures starts reducing, which means that the cement pastes become more electrical resistant, although at a lower rate.

As can be concluded from the results in Fig. 6, and also from the data tabulated data in Table 2, the electrical resistivity values in Stage II are not influenced significantly by cement hydration, despite the development of physical properties of the mixture (e.g., setting and stiffening). Based on the measured electrical resistivity values, the electrical resistivity of 38±2 Ω.cm and 48±3 Ω.cm can be used as the indicator for initial and final setting times of GU cement pastes, respectively. It is observed from Table 2 that the cement hydration reaction affects the electrical properties of the cement pastes more than the internal temperature. Therefore, it is concluded that a cement paste temperature of 26±1°C and 29±1°C can be used as the indicator for initial and final setting times, respectively.

5. Conclusions

The following conclusions are drawn from this study:

- Dissolution of cement ions during Stage I of hydration can be detected by the rapid reduction in the electrical resistivity of the paste, while the temperature of the paste does not change significantly.
- The first significant increase in the internal temperature of the mixtures is observed at the time the mixture starts stiffening (around 2 hours of hydration).
- Beyond the stiffening stage (Stage II of hydration), both the internal temperature and electrical resistivity of the cement paste are influenced by the cement hydration (both increase). However, the ROC of zero in the electrical resistivity is the indicator for detecting the beginning of Stage II (the setting stage).

- In the setting stage, the electrical resistivity and internal temperature of the cement pastes show a similar trend. The internal paste temperature of 26±1°C and 29±1°C can be used as the indicator for initial and final setting times, respectively.
- As hydration proceeds, the setting phase transitions to hardening. This is the beginning of Stage III, where a sharp increase in the electrical resistivity of the cement paste can be observed. This point corresponds approximately to the final setting time. In this stage as well, the internal temperature of the mixtures raises significantly compared to that in Stage II.
- In Stage IV of hydration, the ROC in the electrical resistivity of the cement paste mixtures becomes negative, which means that the cement pastes become more electrical resistant, but at a lower rate.
- Beyond Stage IV of hydration, the resistivity of the cement pastes increases (due to the formation of the pore structure), but at a slower rate as the ions content in the pore solution reduces by the hydration process.
- The significant increase in the ROC of the electrical resistivity can be used as an indicator for the beginning of the hardening stage (not setting stage). This stage is crucial for the concrete industry, as moist curing can be started at this time.
- The most reliable indicator for determining the setting time of the cementing-based mixtures is the internal temperature (not electrical resistivity of the mixture).

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