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ELECTRICAL PROPERTIES AS AN INDICATOR FOR RHEOLOGICAL PROPERTIES DEVELOPMENT OF FRESH CEMENT PASTE

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ABSTRACT

Concrete rheological properties are important in hardening and workability assessment. These properties can be measured by the method standardized in ASTM C1749. However, determining these properties is not often practiced on job-sites. Hence, developing a practical technology to determine the development of the rheological properties of a mixture can be beneficial. Alternatively to ASTM C1749, the test method ASTM D4648 (originally developed for saturated clay soils) can measure the Vane shear resistance of cement paste. However, it is a laboratory test that does not consider the job site conditions. The other physical parameter that develops in the time frame that the Vane shear strength develops is setting. Setting is measured in this project, according to ASTM C109 to plan the time frame of testing for the shear strength development.

Proposing a test method which not only estimates the shear strength, but also considers the actual job site conditions is essential. In this project, development of shear strength (with the Vane shear method) is studied from the mixing time until the initial setting of twelve cement paste mixes. At the same time, the electrical resistivity of the pastes is also measured with a designed technology. Correlation between the changes in the electrical resistivity of the cement pastes with the rheological and setting properties development can result in a practical method for estimating the in-place properties. It is seen that the changes in the electrical properties (e.g., resistivity) of fresh concrete can be used as an indicator for the shear strength development.

Key Words: *cement paste, shear strength, miniature Vane shear test, initial setting time, final setting time, fresh stage electrical resistivity, electrical properties*

1. INTRODUCTION

Concrete is a complex composite material, whose micro and macro structures and properties change over time. The process, rate, and quality of cement hydration (as a complex physical-chemical process) the properties of concrete. These properties are important for concrete construction as concrete performance and long-term durability are determined and analysed by those properties. Among all properties of concrete, beginning of the hardening time (time the mixture starts gaining strength) is one of the most important ones. As concrete begins hardening, its workability reduces. Determining the hardening time (which is different and before the setting time) can help engineers organizing the construction plan. However, there is no standard test to measure the beginning of the concrete hardening time and determine the rate of hardening, cast in-place. In addition to the hardening time, the setting time is the only standard factor in planning the finishing and curing time of a concrete pouring. Monitoring the setting time of concrete by the only available standard testing methods for cement pastes (ASTM C191) or mortars (ASTM C403) is neither adoptable, nor completely applicable to the actual on-site projects.

This research project presents an innovative, practical, and non-destructive technique to estimate the actual beginning of the hardening and development in the shear strength of concrete in a reasonable amount of time, non-destructively. In this novel method, the electrical resistivity (independent from specimen geometry and device electrodes) of cement products is monitored over the time of hydration. Since the factors influencing the electrical resistivity of the cement paste (responsible for electrical properties of the concrete in the fresh stage) are similar to the factors influencing other properties of the cement matrix of the concrete, it can be theoretically concluded that the electrical property can be used as an indicator for other concrete properties (e.g., hardening in this project). This technique will be employed to estimate the beginning of the hardening time and further shear strength development (as the indication for the workability).
Ten mix designs are studied in this project with four types of cement, common in the concrete industry, with three water-to-cement ratios.

2. TEST PLAN AND METHODOLOGY

Theoretically, it can be assumed that the electrical properties of fresh concrete (mixture from mixing time to the 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 10^6-10^{10} times greater than that in fresh cement pastes (Xiao et al, 2008). Therefore, the electrical resistivity 9inverse of conductivity) of a fresh concrete is similar to the electrical resistivity of its fresh cement paste (McCarter and Puyrigaud, 1995). Since all properties tested in this research project are for fresh concretes (e.g., electrical resistivity, setting, and hardening), the project focused on cement pastes, only.

A wide (and practical) range of W/CM ratios and cement types are selected, as presented in Table 1.

Table 1: Cement paste mixtures properties

Cement Type	Supplementary Cementitious Material (% replacement)	W/CM ratio		
		0.37	**0.40**	**0.43**
General Use *(Type 10)*	<%5 Limestone	X	X	X
High Early *(Type 30)*		X	X	X
Silica Fume Blended *(GUbSF)*	5.5 - 7.5	X	X	X
Limestone Cement *(GUL)*	12 - 15	X	X	X

All cement paste mixtures were mixed in four steps according to the standard method described in ASTM C305. Three tests were performed to study the electrical and physical properties of each cement paste.

2.1. Rheology Vane Shear

A cement paste starts hardening before the initial setting time. However, ASTM C191 is not able to determine the hardening time (it shows 40 mm needle penetration at any time prior to the initial setting). The beginning of the hardening process is important, as this is the time the solid state in the cement paste matrix starts forming and the quantity of the pore solution starts reducing. In construction, the beginning of the hardening time is the point when the mixture starts losing its workability and further mixing, pouring, or consolidating is not recommended. Therefore, this crucial time is needed to be determined. In this project, the development of shear strength (as a rheological property) of the fresh cement paste mixture is studied and used as an indicator for the beginning of the mixtures hardening. Besides hardening, workability of concrete and cement pastes can be studied by focusing on the rheology properties. A modified test procedure described in ASTM D4648 is used to determine the development of shear strength of the cement paste mixes. A Ø50 X 100 mm cement paste cylinder is cast for each mix design to measure rheology properties at different times (the test apparatus is shown in Figure 1).

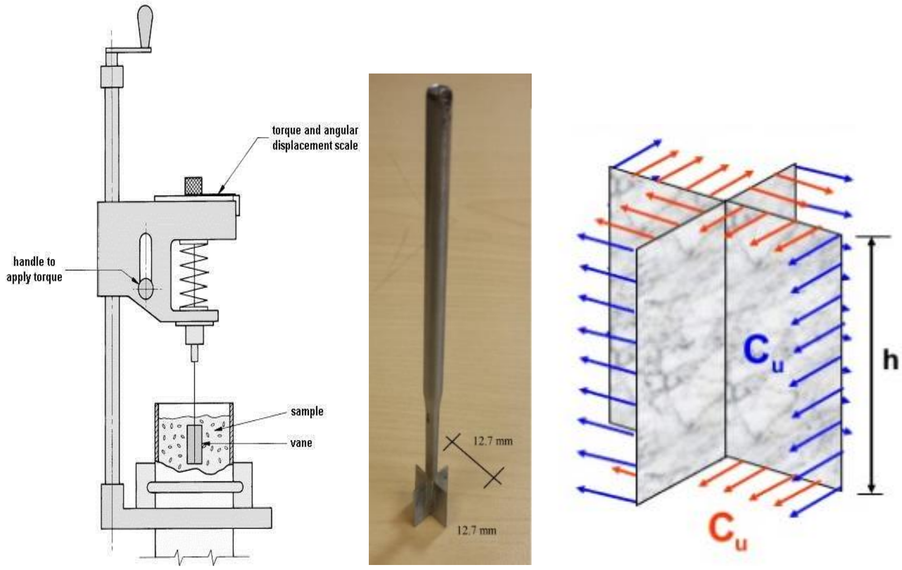


Figure 1: Vane shear test machine (left), 1:1 blade (middle), and uniform shear stress distribution on the Vane shear blades (right)

By placing the blade in the specimen (penetration depth is 50 mm), a constant torque (with a constant rate of 60 to 90°/min) is applied by the machine to a stiff shaft and a calibrated torque spring. By the time the torque applied by the spring overcomes the shearing resistance of the cement paste, the Vane blade rotates rapidly to bring the paste to total failure. All of these processes can be measured by monitoring the rotation angle of the shaft during the test. Based on the rotational angle difference (Δ_f-Δ_i), the shear strength (τ) is calculated. Assuming the distribution of the shear strength is uniform across the ends of the failure cylinder and along the shear blade (schematically shown in Figure 1), the Vane shear constant K (m^3) can be calculated as follows:

$$K = \frac{\prod D^2 H}{2\times10^9}\left[1 + \frac{D}{3H}\right] \ (SI\ Units) \quad [1]$$

where, D is the measured diameter of the Vane in mm and H is the measured height of the Vane in mm.

The maximum measured torque (T) which occurs at the yield point (Figure 2) can be calculated as in Equation 2.

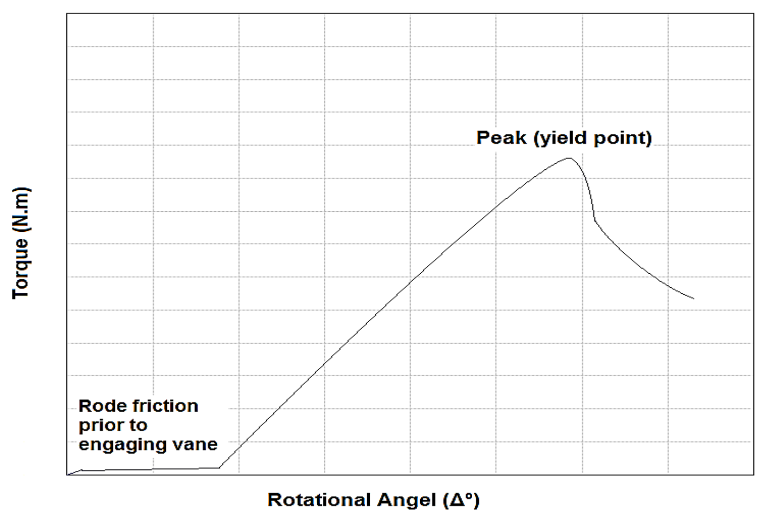


Figure 2: Typical Vane shear test torque and rotational angle relation

$$T = K_{spring} (\Delta_f - \Delta_i) \quad [2]$$

where, Δ_f is the final rotational angle, Δ_i is the initial rotational angle, and K_{spring} is the spring constant which is the inverse of the slope of the calibration curve in °/N.m (based on the typical Vane shear test results, if the applied torque is constantly measured as in Figure 2).

Finally, the undrained shear strength (τ) in Pascal, the point where a significant plastic deformation or yielding occurs due to an applied shear stress, is calculated from the following equation:

$$\tau = \frac{T}{K} \quad [3]$$

where, K is the Vane constant (m^3) from Equation 1 and T is the measured torque (N.m) in Equation 2..

2.2. Fresh Stage Electrical Resistivity

The bulk resistivity of fresh cement paste is measured continuously until the final setting. The test setup is similar to the Consensor measurement technique setup. In this test setup electrodes are embedded in the sample right after casting the mixture. The embedded electrodes setup is the test setup used for fresh cement paste resistivity measurement in this research project. It has been concluded from previous studies that a frequency between 5 kHz and 15 kHz (depending on sample resistance) was normally sufficient to reduce the electrode effects (McCarter and Puyrigaud, 1995). Here, 110 mm long stainless steel electrodes are in contact with the cement paste) in an Ø100X200mm cylinder) and the electrical property of the paste in between the electrodes is measured (Figure 3). To avoid any of the polarization effects described before, AC impedance is employed with a frequency of 10 kHz. The electrical meter can be connected to a cell phone via Bluetooth or WiFi.

Figure 3: Electrodes setup for fresh cement paste electrical resistivity measurement (photo courtesy of Giatec Scientific Inc., 2015)

The electrical resistance (impedance) measured by the meter is converted to electrical resistivity, which is independent from the sample geometrical properties. Electrical resistivity, ρ_C, is obtained by multiplying the measured resistance, R_c, by a conversion factor, called the cell constant, *m*. By considering the current density flux, electrodes size/spacing, and the assumption that the electrical field around the electrodes are in the form of a half cylindrical surface, the conversation factor, *m*, was mathematically calculated as

$$m = \frac{\pi h}{\ln(r)} \quad [4]$$

In the test setup illustrated in Figure 3, h=110 mm and r =50 mm. Therefore, m= 88 mm. This mathematical conversion factor has been calibrated with experimental results by comparing the electrical resistivity and resistance of a known solution.

2.3. Setting Time

Initial and final setting times are the indicating stages of concrete setting, workability, and finishability. Initial and final setting times of cement paste can be determined by the test method described in ASTM C191. The initial setting time is the time taken by cement paste to stiffen enough so that the 1-mm needle of the Vicat's apparatus penetrates by 5-7 mm from the bottom of the mould. The initial setting time is an indication of the critical stage after which cement paste fresh properties such as workability decrease. Final setting is the time when the apparatus needle (1 mm diameter) does not form any visible indentation on the test specimen surface.

3. Test Results and Discussions

3.1. Rheology Vane Shear

At the preliminary testing stage of the project, all cement paste mixtures were tested for the beginning of the hardening time according to the method described in Section 2.1. The time hardening starts in cement paste is the time the shear strength starts developing. To determine the beginning of the hardening development time, cement pastes were cast in Ø50X100 mm cylinders and tested with the Vane shear after the mixing. Since the Vane shear strength may be greatly influenced by the rate at which shear occurs, the torque was applied using a motorized Vane device (instead of a hand crank manual torqueing). Based on the results tabulated in Table 2, 120 minutes (average) is considered as the time that the cement paste hardening process starts.

Table 2: Early stage Vane shear test results to determine the hardening time of the cement pastes

Mix Design		**Δ_f - Δ_i ° at the Time of Testing** (min)							
Cement Type	**W/CM**	**30**	**60**	**90**	**100**	**110**	**120**	**130**	**140**
GU	**0.37**	0	0	0	0	0	4°	NT	NT*
	0.40	0	0	0	0	0	0	3°	NT
	0.43	0	0	0	0	0	0	2°	NT
HE	**0.37**	0	0	0	1°	NT	NT	NT	NT
	0.40	0	0	0	0	3°	0	NT	NT
	0.43	0	0	0	0	2°	0	NT	NT
GUL	**0.37**	0	0	0	0	0	7°	NT	NT
	0.40	0	0	0	0	0	4°	NT	NT
	0.43	0	0	0	0	0	2°	NT	NT
GUbSF	**0.37**	0	0	0	0	2°	NT	NT	NT
	0.40	0	0	0	0	2°	NT	NT	NT
	0.43	0	0	0	0	0	2°	NT	NT

*NT= Not Tested (as the beginning of the hardening time was determined)

One hundred and twenty (120) minutes also meets the requirement described in CSA A23.1 for the maximum allowable time for pouring and finishing a cement-based batch, if the mixture temperature does not exceed the limits in Table 14 of CSA A23.1 (2014). This is the time a cement-based mix starts hardening and cannot be placed and consolidated, properly.

Based on the results, the shear strength of all cement pastes are studied at 2 hours after mixing and at initial setting. The rotational strength (the Vane shear strength) of the cement paste, which can be an indicator for the workability in the mix, is calculated according to the relations described in ASTM D4648, and the results are shown in Figure 4**.**

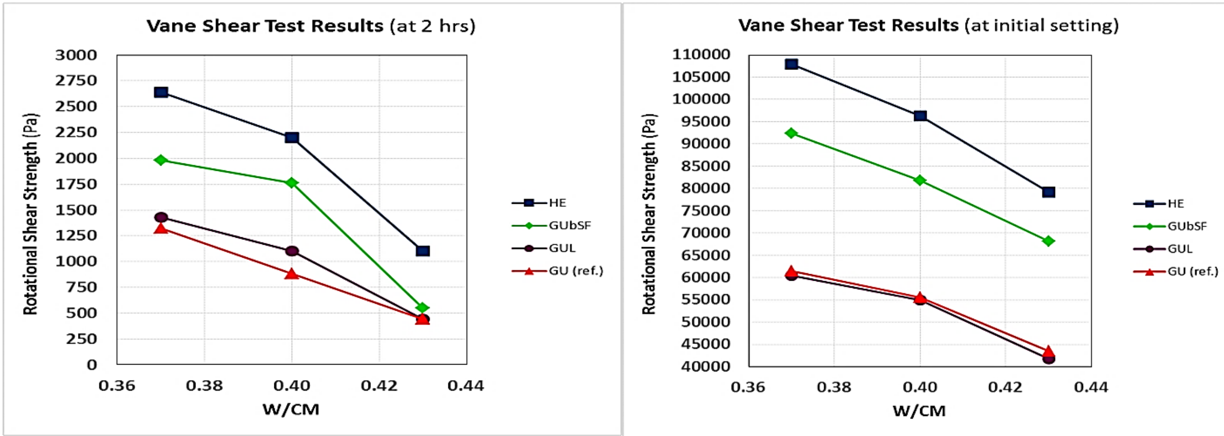


Figure 4: Rotational undrained shear strength development of cement pastes at 2 hours of hydration (left) and initial setting (right)

As the W/CM ratio increases, the shear resistance of the mixtures decreases. This reduction is significant in the HE cement paste mixture compared to the GU mixture, which is used in this study as the reference mixture. It is seen that the presence of silica fume (which was not hydrated at 2 hours) resulted in higher shear strength. At initial setting, it can be said that having limestone does not influence the workability of the mixture, significantly, while silica fume decreases the workability of the mixtures. Also, HE mixtures lost their workability faster than other mixtures.

3.2. Fresh Electrical Resistivity and Setting Time

The initial and final setting times of the mix designs studied are shown in Figure 5.

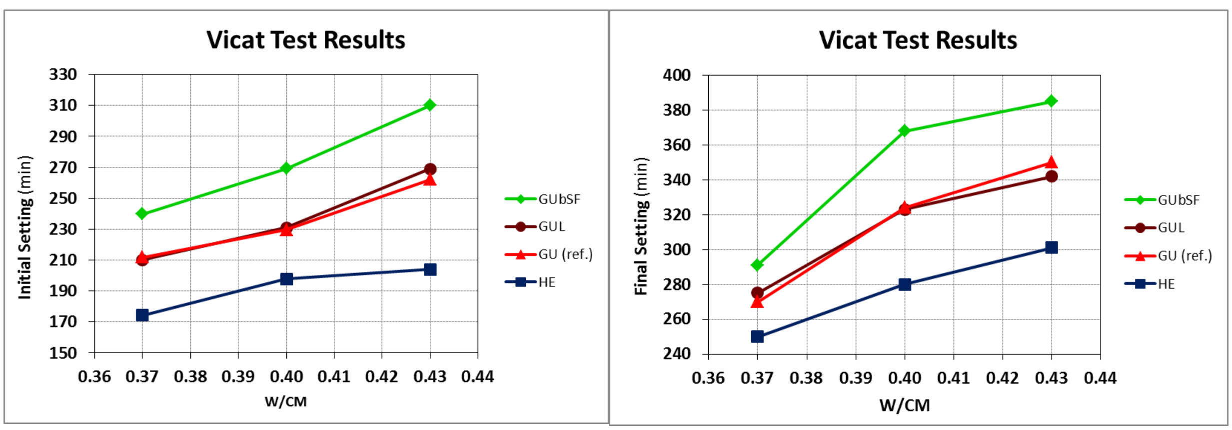


Figure 5: Initial (left) and final (right) setting time of cement paste mixtures

It can be seen that both initial and final setting times were influenced by the W/CM ratio and the cement type. From Figure 5, it is noted that the initial setting time increased as the water content of the mixture increased. However, the rate of the increase in the reference mixture (GU cement) was more than 30% than that in the HE mixtures.. The HE initial setting time at W/CM ratios greater than 0.40 was not significantly influenced by the addition of water, while the final setting time for these mixtures was more affected by the extra water. In addition, it is seen that the mixtures containing silica fume showed longer setting time which can be related to later contribution of silica fume to the setting properties.

The electrical resistivity of fresh cement paste was measured from time zero (immediately after mixing) until the final setting time. Figure 6 illustrates the electrical resistivity evolution with time for each of the four mixes categorizes (four types of cement).

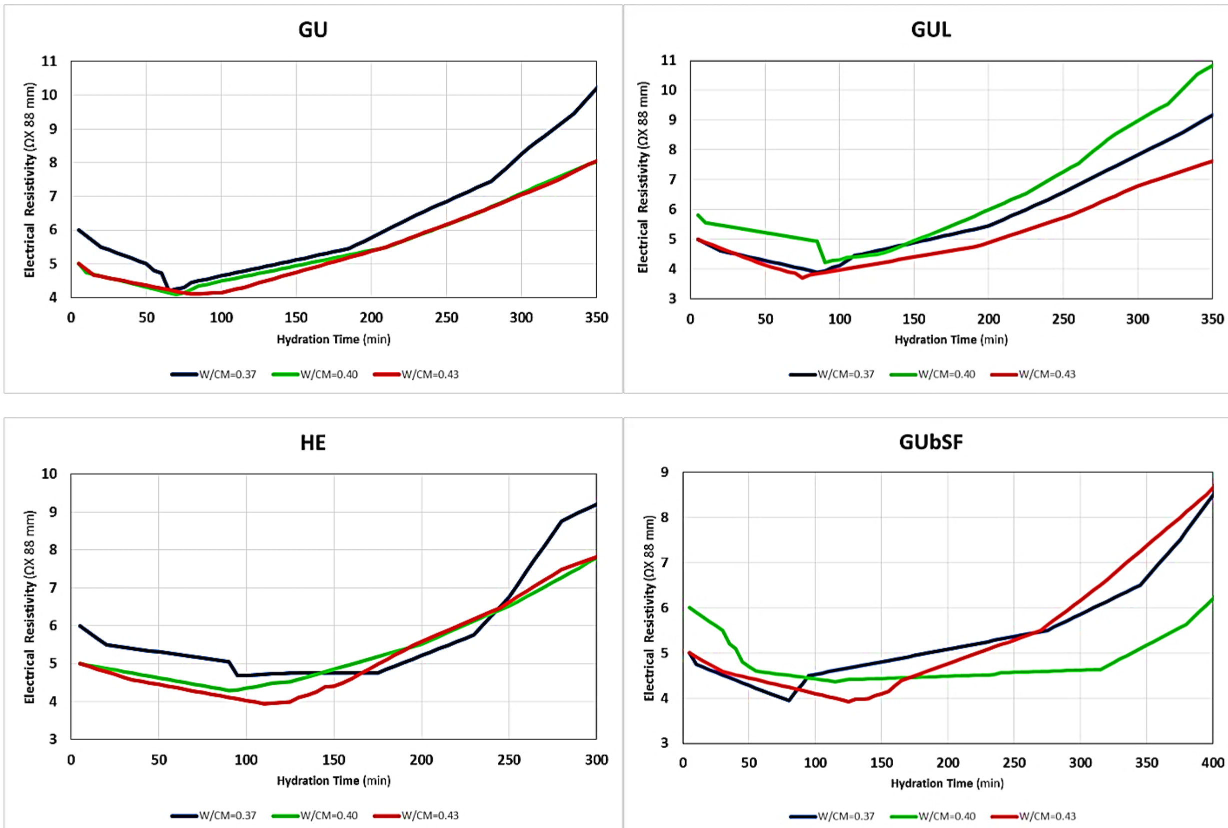
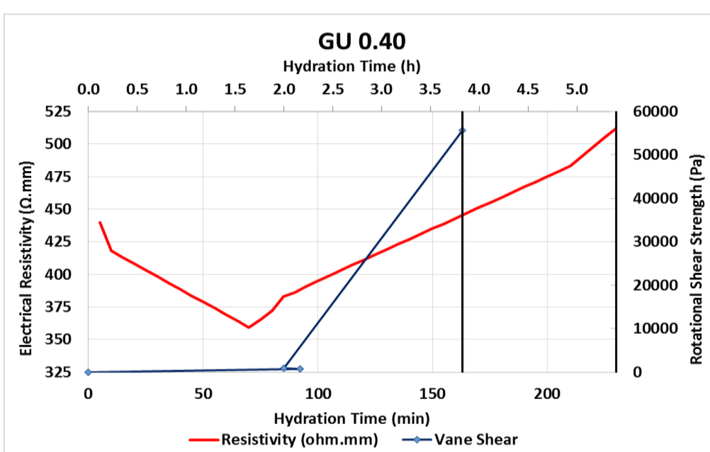


Figure 6: Electrical resistivity changes of cement paste mixtures (W/CM=0.37, 0.40, and 0.43)

It is seen that after a drop in the fresh cement paste electrical resistivity at the beginning of the hydration due to dissolution of ions in the pore solution (Wei and Xiao, 2014), electrical resistivity of the pastes increased over time. High early cement pastes showed higher electrical resistivity followed the mixtures containing silica fume and then the General Use mixes. Although silica fume does not contribute into the secondary hydration in the early stages of hydration (Neville, 2008), the presence of silica fume particles in the fresh paste matrix resulted in higher electrical resistance than similar mixtures without it.

3.3. Electrical Property as an Indicator

Graphs in Figure 7 show the development in the electrical resistivity and the shear strength (for mixes with W/CM=0.40) from the mixing time to the final setting time (main focuses of the project).



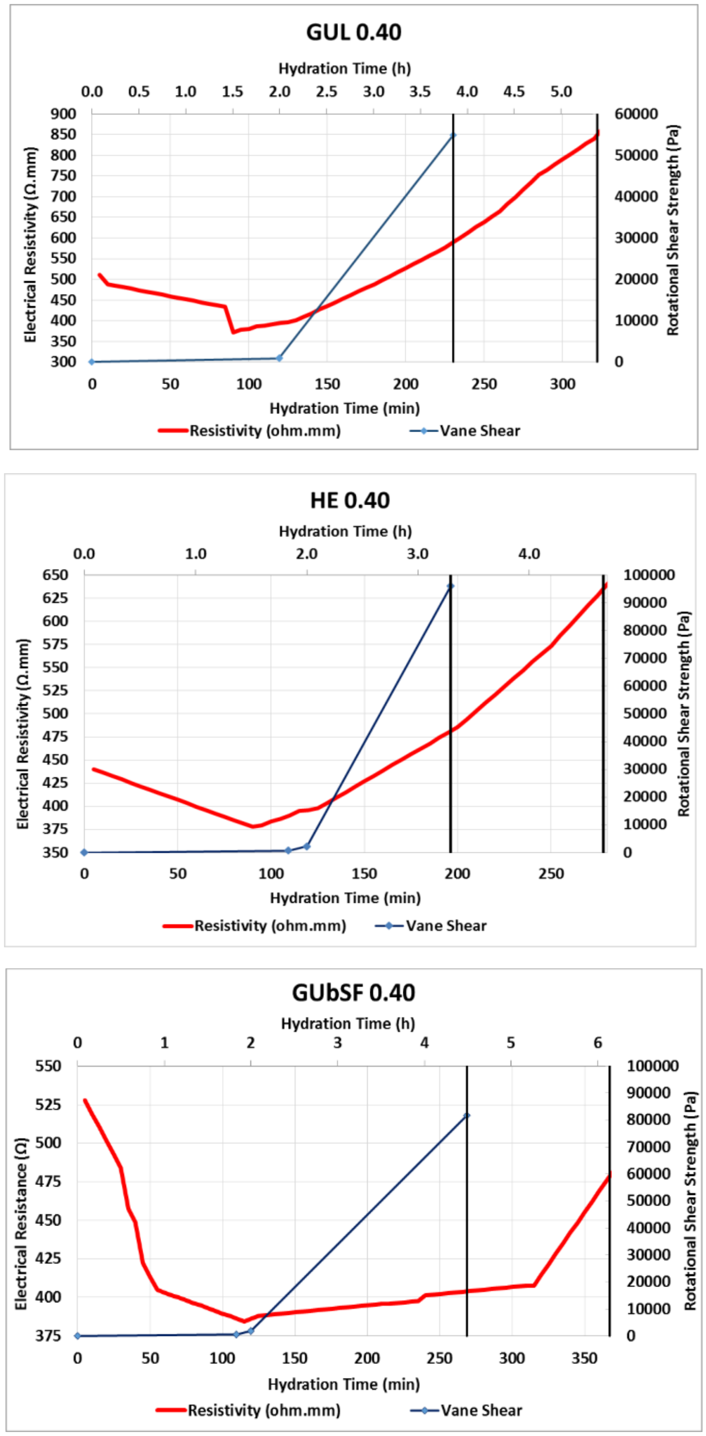


Figure 7: Electrical resistivity and Vane shear strength for cement mixtures with W/CM=0.40 (vertical lines: initial and final setting times)

It is seen that the time when the electrical resistivity starts increasing is closed to the time that the cement hardening begins (where the Van shear strength starts developing). This time is about 2 hours. The resistivity values at that time can be used as indicators for the beginning of the hardening process. This is a crucial time for the concrete construction as concrete pouring should be completed by that time, since concrete starts gaining shear strength (reduction in workability starts at this point).

In addition, it is seen that the electrical resistivity values of similar mixtures (with different water-to-cement ratio) are statistically similar at the initial setting time. In other word, the average resistivity values can be used to determine the initial setting time. The average electrical resistivity values are tabulated in Table 3.

Table 3: Average values representing cement-based mixtures properties

Mix Design	Resistivity at the Beginning of Hardening (Ω.mm)-CoV%		Resistivity at Initial Setting (Ω.mm)-CoV%		Resistivity at Final Setting (Ω.mm)-CoV%		Shear Strength at Initial Setting (kPa)-CoV%	
HE	387	9	468	9	670	4	94	15
GUbSF	381	8	478	16	573	22	80	15
GUL	387	5	543	8	715	17	52	18
GU	407	5	534	5	671	5	54	17

CoV: Coefficient of Variation

The average resistivity values for each mixture (regardless of the W/CM ratios as presented in Table 3) can be used as the indicator for the other properties (setting time, begging of the hardening and shear strength). Statistical analysis has shown that the average values are good representatives for similar cement type mixtures properties in fresh stage.

Besides, the average shear strength values at the time of initial setting (the last column in Table 3) can be considered as the shear strength for the time that a cement-based mixture is not workable anymore. The electrical-resistivity technique can be used to determine that time and the shear strength value at that time. This value can help engineers plan the time that concrete construction must be completed and surface finishing must be started.

4. CONCLUSIONS

The following conclusions are drawn from the research project:

1- As the water content of the mixes is increased, the electrical resistivity decreases. This reduction can be explained by the changes in the physical characteristics of the pore network and the ionic concentration in the pore solution (to be further studies in the future).
2- The W/CM ratio directly influences the setting time of cement paste mixtures. Mixtures with higher water content have longer setting time (initial and final).
3- The shear strength of the mixtures is affected by water content of the mixtures. Higher W/CM ratio resulted in lower shear strength.
4- By using the Vane shear test method (usually used for saturated clay soils), it is seen that all mixtures start hardening at about 2 hours from mixing. It meets the requirement described in CSA A23.1 for the maximum allowable time for pouring and finishing a cement-based batch. The Vane shear (measuring the yield shear strength is a reliable test to determine the hardening time and later, the workability of a mixture).
5- Beside water content, the cement type has a significant effect on the shear strength and setting time of the cement-based mixtures. Mixtures with the High Early cement (Type 30) showed higher shear strength and lower setting time (initial and final), followed by GUbSF (general use cement with 5.5-7.5% silica fume replacement). General used mixtures (GU and GUL) showed the least shear strength values.
6- Measuring the changes in the electrical resistivity of the mixture (as a factor independent from the probe sizes and shapes) can be used as a technique to estimate the setting and beginning of the hardening time of the mixtures. Placement of the resistivity-meter on site (as designed in the project) will result in considering the job site conditions.

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