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Effects of temperature and alkaline solution on electrical conductivity measurements of pozzolanic activity

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ABSTRACT

The influence of alkaline solutions (which are unsaturated lime, saturated lime and ordinary Portland cement solution), reaction temperatures (40 °C, 60 °C and 80 °C), and quantity of tested sample in 200 mL of alkaline solution on the pozzolanic reaction of silica fume (SF) is considered on electrical conductivity measurements. The results reveal that raising the reaction temperature accelerates the pozzolanicity and 80 °C is a recommended reaction temperature for the rapid evaluation of pozzolanic activity. An ordinary Portland cement (OPC) solution is proposed and used as an alkaline solution for pozzolanic activity evaluation. Increasing the amount of SF also increases the chemical reaction rate. It was found that using 1.0 g of SF/200 mL of OPC solution yield the best results. This study could be used as a basis for further development of a more accurate, rapid evaluation of pozzolanic activity.

1. Introduction

Due to the advantages of using pozzolans as cement replacement materials – namely durability and strength – pozzolans are being increasingly used in concrete as partial substitutes for ordinary Portland cement. By definition a pozzolan is "a siliceous, or siliceous and aluminous, material which in itself possesses little or no cementitious value; but in finely divided form and in the presence of moisture, it chemically reacts with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties" [1]. The pozzolanic reaction differs in behavior from the hydration reaction of Portland cement with water in regard to the formation of calcium silicate hydrate (C–S–H). The pozzolanic reaction is slow relative to that of cement and water. However, the pozzolanicity could be accelerated by using elevated reaction temperature techniques [2–6]. Therefore, to find a more rapid evaluation method of pozzolanic reaction, an elevated reaction temperature should be considered.

Since, the electrical conductivity of the pozzolan-lime solution (electrolyte) depends on amount of ions in the solution [7] while the pozzolanic reaction results in the increase of C–S–H at the expense of $Ca(OH)_2$ [2,4–8]. In other words, the interaction between both Ca^{2+} and OH^- ions and pozzolan in pozzolan-lime solution to form reaction products resulting in a reduction of ions and hence the decrease in the electrical conductivity. Hence, pozzolanic reaction could be monitored by measuring the electrical conductivity of the pozzolan in lime solution [3,7,9,10]. Previous researches obtained the results of pozzolanic activity by conductimetric technique in a saturated or unsaturated lime solution [3,7,9,10]. However, pozzolanicity is still imperfectly understood. It has been suggested that, in addition to reacting with $Ca(OH)_2$,pozzolans react also with C_3A or its products of hydration [5,6]. Therefore, investigating pozzolanicity of pozzolan used as a cement replacement material based on pozzolan – the hydration product solution should be studied to compare with pozzolan-lime solution.

In the case of Portland cement it has been found that, when mixed with water, cement produces a solution supersaturated with $Ca(OH)_2$ and containing concentrations of calcium silicate hydrate and other products [6]. Normally the concentration of an alkaline solution affects the hydration of pozzolan: higher concentrations create a faster reaction [11]. Therefore it is of interest to use the OPC solution as an alkaline solution for the evaluation of pozzolanic activity. Besides, using an OPC solution represents a more realistic condition, which occurs in concrete mixtures containing pozzolan.

Regarding the time required to prepare the alkaline solution: the rapid evaluation of pozzolanic activity using a saturated lime solution to activate the reaction was first introduced by Luxan et al. [9]. Even though the solution was simple and easy to prepare, the time required for saturation equilibrium was at least 24 h. Subsequently, Paya et al. [3] proposed using an unsaturated lime solution, which took about 1 h to prepare. This study proposes a new type of alkaline solution for use in the rapid evaluation of pozzolanic activity by using an ordinary Portland cement (OPC) type I

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solution. This solution, which will be called an OPC solution, can be easily prepared within 1 h and could simulate the actual chemical reaction between pozzolan and a cement–water product.

The aim of this study is to present the influence of the type of solution, the reaction temperature, and the quantity of tested pozzolan on its electrical conductivity for the evaluation of pozzolanic activity of tested pozzolan. Two types of lime solution (unsaturated and saturated) and an OPC solution will be used; and three different suspension temperatures – namely 40, 60 and 80 °C – will be attained in an experimental program. Since silica fume (SF) contains about 90% silica by mass and is an excellent pozzolan (used to make high-strength concrete) it was selected as the tested pozzolan in this study. The quantity of SF tested varied from 1.0 ± 0.5 g, whereas the volume of the OPC solution was kept constant at 200 mL. The electrical conductivity of the suspension under various conditions was monitored by using an electrical conductivity meter through a personal computer. Finally, the result obtained will be used to develop a rapid evaluation of pozzolanic activity of pozzolan.

2. Experimental investigation

2.1. Materials

Ordinary Portland cement (Elephant brand complying with ASTM type I, manufactured by Siam Cement Co., Ltd.) and pure calcium hydroxide powder were used to prepare lime solutions. Condensed silica fume, Force 10000D, was obtained from W.R. Grace & Co., Thailand, to be used as tested pozzolan.

2.2. Testing apparatus

This research used the 250 mL flask and rubber cork with hole for conductivity meter probe and a magnetic stirrer which allows adjustment of the base plate temperature and stirring rate, as shown in Fig. 1a. Rubber cocks was used as carbonation protectors when installed on the glass stopper. The electrical conductivity and the temperature were measured using a WTW (Cond 340i) conductivity meter, as shown in Fig. 1b. In addition, the temperature and conductivity readings were fed, through an interface module, to a computer-based data acquisition system.

Fig. 1a. Magnetic stirrer with hotplate.

Fig. 1b. Digital electrical conductivity meter.

2.3. Preparation of alkaline solutions

There are three types of alkaline solution used in this study: saturated lime solution, unsaturated lime solution, and a specially prepared solution hereinafter referred to as OPC solution. These solutions were prepared as follows:

I. Saturated lime solution was first proposed by Luxan et al. [9]. This solution is an alkaline solution for rapid evaluation of pozzolanic activity by conductimetric technique. This type of solution can be obtained by gradually adding calcium hydroxide powder to distilled water, and shaking every few hours for a period of 24 h until the solution becomes saturated (by observing that dissolution of the calcium hydroxide powder has terminated). The saturated lime solution in this experiment had an electrical conductivity of approximately 7.6 mS/cm at the reference temperature of 40 °C (with nonlinear temperature compensation according to EN 27 888).

II. Unsaturated lime solution was prepared following Paya et al. [3] in order to solve the low dissolution rate of $Ca(OH)_2$. This solution had a concentration of 800 mg $Ca(OH)_2$ per liter of de-ionized water, and an electrical conductivity of approximately 4.0 mS/cm at the reference temperature of 40 °C (with nonlinear temperature compensation according to EN 27 888).

III. This solution is proposed by the authors [12] in order to simulate the actual interaction between pozzolan and a cement–water product. In addition, this procedure allowed preparation of an OPC solution within 1 h. The OPC solution was obtained by mixing approximately 350 g of ordinary Portland cement type I and 1 L of tap water. The OPC solution was stirred and stored for about 30 min to allow precipitation to take place. After the mixture became clear, it was poured (without dregs) and kept in a sealed container to protect against carbonation. The electrical conductivity of this solution will vary with the particular cement used.

Hence for this experiment the initial electrical conductivity of the solution was fixed in the range of 10–11 mS/cm at the reference temperature of 25 °C. In order to avoid the influence of initial concentration of the potential activity in the solution, it could be adjusted by adding distilled water or more concentration of OPC solution. This range of initial electrical conductivity was selected because it is a supersaturated solution with $Ca(OH)_2$ [6] and it is difficult to raise its electrical conductivity beyond this concentration level.

All solutions were used without dregs to avoid non-homogenous properties, and were kept in a sealed container after preparation in order to prevent carbonation.

2.4. Testing program

The details of this experimental program are shown in Table 1. Eleven series – designated as A, B, C, D, E, F, G, H, I, J and K – were tested to investigate the influence of the solution type, temperature and quantity of testing sample. This experiment was originally aimed to carry out experiences at temperatures in the 0–100 °C range. However, low-temperature experiences (0–40 °C) were not conducted because of slow reaction time. Secondly, temperatures higher than 80 °C brought technical difficulties due to high water-vapor pressure and loss of water in the testing flask. Therefore the reaction temperatures used in this study were 40, 60 and 80 °C. In addition, three types of alkaline solution – namely saturated lime solution, unsaturated lime solution and the proposed OPC solution – were tested in order to investigate and compare the performance of the proposed alkaline solution. For all series, the electrical conductivity of suspension was recorded with non-linear temperature compensation according to EN 27 888 by using a personal computer at 1-min intervals for a duration of 8 h.

2.5. Testing procedures

The testing for each series was carried out according to the following procedure:

- Two-hundred milliliter of the prepared solution was poured into a flask. And then, rubber cork was plugged tightly to protect the solution from carbonation reaction during the measurement.
- The temperature of the solution was set until the required temperature was reached.
- After the solution temperature had stabilized (usually about 15–30 min, depending on the required temperature) the SF was added at one time into the solution and the measuring of electrical conductivity started.
- The suspension was continuously stirred and the suspension temperature was controlled within ±1 °C of the reaction temperature throughout the experiment.
- The electrical conductivity of the suspension at the required temperature, expressed in terms of millisiemens per centimeter (mS/cm), was measured by using an electrical conductivity meter (model Cond 340i, WTW brand) with serial interface (RS232) output. The data was sent to a personal computer and recorded.

3. Experimental results and discussions

Three parameters – namely suspension temperature, type of alkaline solution, and quantity of tested sample – were experimentally investigated. The results for each parameter are presented and discussed as follows.

3.1. Effect of suspension temperature level

In this study the influence of suspension temperature on the electrical conductivity of the suspension was experimentally investigated. Fig. 2 demonstrates the relationship between normalized electrical conductivity and elapsed time for three different alkaline solutions, having temperatures of 40, 60 and 80 °C. It should be noted that these results were obtained using a quantity of SF equal to 0.50 g. Despite the electrical conductivity of a suspension increasing with temperature, in this case normalized electrical conductivity was used instead of using temperature-compensated. Normalized electrical conductivity is defined as the ratio of the measured electrical conductivity at any time and its initial electrical conductivity. For an unsaturated lime solution, it can be observed in Fig. 2a that the normalized electrical conductivity of the suspension slowly decreased with elapsed time when the suspension temperature was 40 °C. For a suspension temperature of 60 °C, it was found that the rate of decrease in normalized electrical conductivity was faster than that having a suspension temperature of 40 °C. Finally, at 80 °C the decreased rate of normalized electrical conductivity was very fast as compared with those having lower temperatures. It is of interest to note that the normalized electrical conductivity of a suspension at 80 °C became stabilized after approximately 3.5 h of elapsed time. This indicated that the chemical reaction between SF and the unsaturated lime solution was more or less terminated.

For saturated lime and OPC solutions, similar trends were observed at suspension temperatures of 40, 60 and 80 °C, as shown in Fig. 2b and c respectively. It can be concluded from the results obtained in Fig. 2 that the optimum suspension temperature for the pozzolanic activity evaluation of SF by measuring the electrical conductivity was 80 °C. This can be explained by the fact that at 40

Table 1
Details of experimental program.

Series	Type of solution	Suspension temperature (°C)	Quantity of SF (g)	Investigated parameters
A	Unsaturated lime	40	0.5	*T*[a]
B	Unsaturated lime	60	0.5	*T*
C	Unsaturated lime	80	0.5	*T* and *S*[b]
D	Saturated lime	40	0.5	*T*
E	Saturated lime	60	0.5	*T*
F	Saturated lime	80	0.5	*T* and *S*
G	OPC	40	0.5	*T*
H	OPC	60	0.5	*T*
I	OPC	80	0.5	*T* and *S* and *Q*[c]
J	OPC	80	1.0	*Q*
K	OPC	80	1.5	*Q*

[a] *T* = Suspension temperature.
[b] *S* = Solution type.
[c] *Q* = Quantity of test sample.

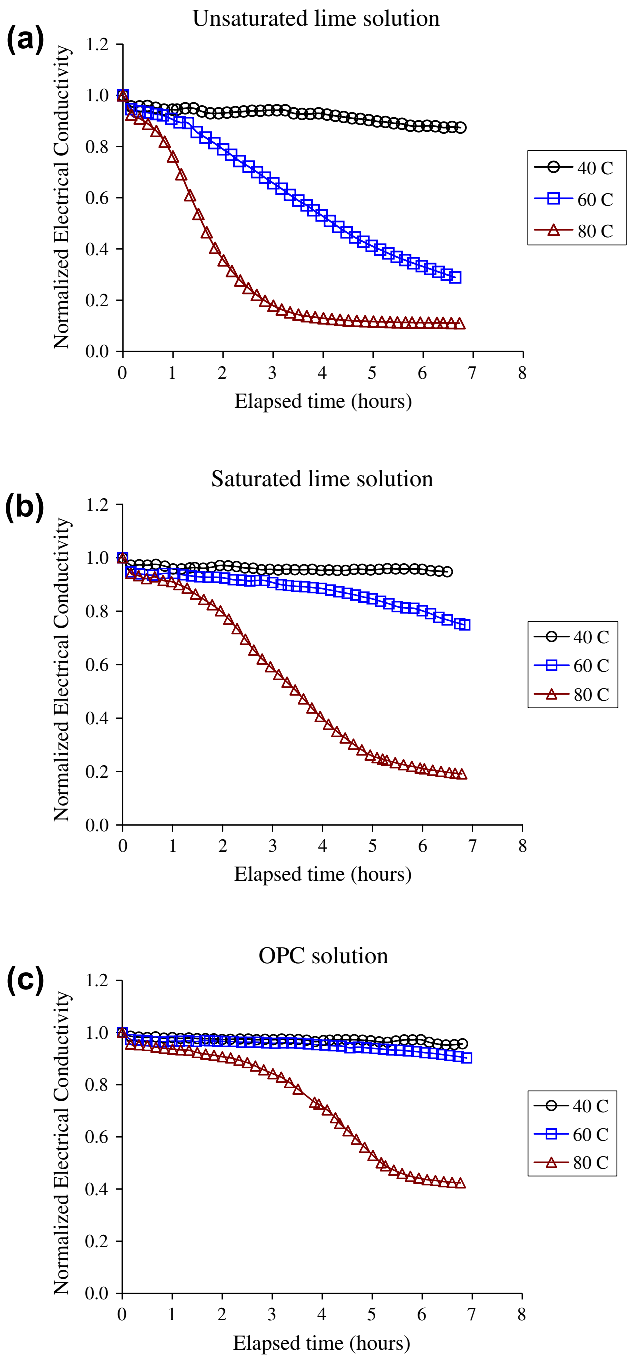

Fig. 2. (a–c) Influence of suspension temperature on normalized electrical conductivity for different types of alkali-activated solutions and 0.5 g of SF.

and 60 °C the electrical conductivity of the suspension kept decreasing, whereas at 80 °C the electrical conductivity of the suspension was more or less stabilized within an elapsed time of 7 h. In other words, the chemical reaction between SF and the alkaline solution was stopped. Moreover, raising the suspension temperature showed a high loss in electrical conductivity and illustrated a more measurable of the evaluation parameter. Hence, a suspension temperature of 80 °C is appropriate for the rapid evaluation of pozzolanic activity of pozzolan.

3.2. Effect of alkaline solutions

This study investigated the pozzolanic reaction of SF using three different types of alkaline solution: namely, unsaturated lime solution, saturated lime solution, and OPC solution at the recommended suspension temperature of 80 °C obtained from the previous section. The amount of SF used was kept constant at 0.50 g and the solution volume was 200 mL. The relationship between electrical conductivity and elapsed time using different types of alkaline solution having a temperature of 80 °C is shown in Fig. 3. The initial values of electrical conductivity of the unsaturated lime solution, saturated lime solution and OPC solution at 80 °C were found to be 3.3, 4.5 and 6.7 mS/cm respectively. It should be noted that the electrical conductivity of a solution varies with the suspension temperature; hence the initial values for the three alkaline solutions were different from those mentioned in Section 2.3. For each type of solution it was found that the electrical conductivity gradually decreased with elapsed time, and after a certain period of time the electrical conductivity became more or less unchanged. After stabilization the final electrical conductivity values of the unsaturated lime, saturated lime and OPC solutions were found to be 0.36, 0.84 and 2.85 mS/cm respectively. It is of interest to note that the electrical conductivity of the unsaturated lime solution became stabilized after approximately 3.5 h. For the saturated lime and OPC solutions, the times required for more or less complete chemical reaction were found to be about 5 and 6 h respectively. The criteria which should be used in the selection of the optimum type of alkaline solution are explained in a subsequent section.

It is of interest to note from Fig. 3 that the relationship between electrical conductivity and elapsed time can be divided into four stages, as discovered by McCarter and Tran [10]. During the first 10 min, which represents the first stage, for all types of alkaline solutions the electrical conductivity significantly decreased. This indicated some initial chemical reaction on the particles. Although the results were obtained from different sample preparation techniques, it could be inferred from other previous research [9,10] that the loss in electrical conductivity during this stage could itself be taken as an indicator of pozzolanic activity: the greater the reduction, the better the pozzolanicity of the material. However, this could not explain the overall pozzolanic reaction of the sample [3,10]. The second stage showed a period where the rate of change of electrical conductivity remained relatively constant and attained a low value. It could be denoted as a dormant period. Such a low rate of change in electrical conductivity would indicate a decrease in chemical activity within the suspension in comparison to stage one. A significant decrease in electrical conductivity was observed in the third stage, which indicated an increase in chemical activity after the dormant period. Electrical conductivity was finally stabilized within 6 h. The stabilization of electrical conductivity is attributed to a slowing down in chemical reactions.

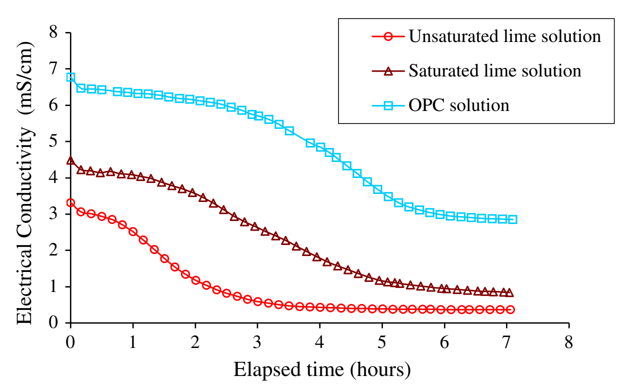

Fig. 3. Relationship between electrical conductivity and elapsed time of SF 0.5 g in different alkali-activated solutions at 80 °C.

3.2.1. Loss in electrical conductivity

Table 2 illustrates the initial, final and loss values of the electrical conductivity of different types of alkaline solutions at a suspension temperature of 80 °C. The loss in electrical conductivity is obtained by taking the difference between the initial and final values of the electrical conductivity of the suspension, and represents the degree of chemical reaction between silica fume and $Ca(OH)_2$ available in the solution. In this experiment, the losses of electrical conductivity in unsaturated lime, saturated lime and OPC solutions were found to be 2.95, 3.65, and 3.92 mS/cm respectively. As mentioned earlier, the reduction of electrical conductivity in an alkaline solution reflects the degree of pozzolanic reaction between silica fume and the amount of $Ca(OH)_2$ available in the solution. The electrical conductivity of a $Ca(OH)_2$ solution would be decreased by the interaction between both Ca^{2+} and OH^- ions and tested pozzolan. It is evident from Table 2 that the loss of electrical conductivity in the unsaturated lime solution was lowest, whereas that of the OPC solution was highest. This indicates that for the unsaturated lime solution, the main chemical reaction was stopped due to the depletion of Ca^{2+} and OH^- ions in the solution. Since both saturated lime and OPC solutions contained higher amounts of $Ca(OH)_2$, the loss values of electrical conductivity were higher than for the unsaturated lime solution. This means that an amount of SF still remained for the chemical reaction. In other words, different types of alkaline solutions contained different initial amount of reactants. In addition, the mechanism of lime solution and OPC solution are different. As hydration progresses, the portlandite initially present in blended cements re-dissolves and participates in further C–S–H production and it would seem reasonable to expect a similar mechanism to pozzolanic reaction [13]. It can be concluded that since the proposed OPC solution yielded a more measurable and pronounced response, it should be selected as the optimum type of alkaline solution for the pozzolanic activity evaluation of pozzolan.

Although, the initial electrical conductivity of the prepared OPC solution was controlled and checked to be fixed in the range of 10–11 mS/cm before using, sensitivity of the results to cement composition did not include in this study. However, to determine the element concentration of OPC solution, X-ray fluorescence technique (XRF) was applied. The result of XRF shows that OPC solution contains calcium, potassium and sulfur elements are 1.69%, 0.15% and 0.12% by weight, respectively.

3.3. Effect of quantity of SF

Once the optimum type of alkaline solution (OPC solution) and suspension temperature (80 °C) are established, it is of interest to investigate experimentally the influence of SF quantity on the electrical conductivity of an OPC solution having a suspension temperature of 80 °C. In this case the amount of SF used varied from 0.5 to 1.0 g, whereas the volume of OPC solution was fixed at 200 mL. Fig. 4 shows the loss in electrical conductivity at 10 min versus the amount of SF in the sample. The close – almost linear – relationship between the loss of electrical conductivity at 10 min and the amount of SF was mentioned by Luxan et al. [9]. It was found that by increasing the amount of material the loss of electrical conductivity also increases. The explanation of this result is that during the initial stage only superficial reactions take place, but the more inner reactive parts of the pozzolan particles remain unaffected [3]. Thus, only considering the data over such a short time scale does not reflect the overall response of the material [10].

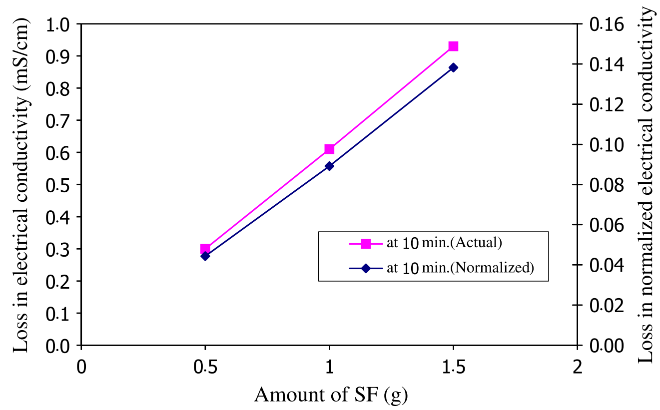

Fig. 4. Loss in electrical conductivity versus amount of SF at 10 min.

Fig. 5 depicts the results showing the influence of SF dosage on electrical conductivity of an OPC solution at a suspension temperature of 80 °C for periods extending over 8 h. The relationship between the electrical conductivity and elapsed time using different quantities of SF was generally found to be similar to that shown previously. In the case of 1.00 g of SF, the time required to stabilize the electrical conductivity is shorter than that for a SF dosage equal to 0.50 g, whereas the time required to stabilize 1.00 g is similar to that of 1.50 g of SF. The results showed that increasing the amount of pozzolan increased the initial rate of chemical reaction especially 0–2 h. It is significant that the different doses of SF yielded similar levels of normalized electrical conductivity: approximately 0.4 within an elapsed time of 8 h. This result is similar to that obtained by Paya et al. [3]. Little variation in the percentage of loss in electrical conductivity over time was measured for the higher amount of pozzolan sample. In other words, the existing reacted ions in the OPC solution were more or less completely consumed after 8 h by using different doses of SF. It could be suggested that for the case of 1.00 and 1.50 g of SF, there was an excess of pozzo-

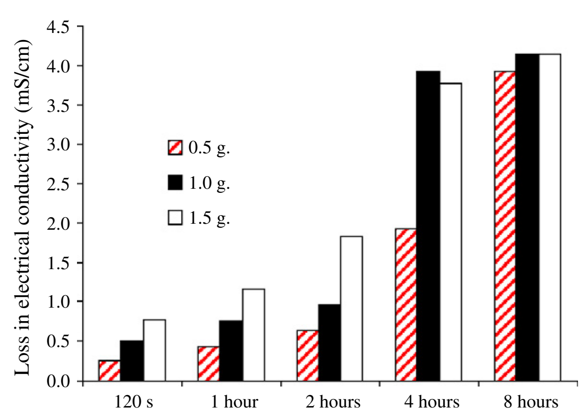

Fig. 5. Influence of SF dosage on electrical conductivity of an OPC solution having a suspension temperature of 80 °C at 120 s and 1, 2, 4 and 8 h.

Table 2
Loss of electrical conductivity of different alkali-activated solutions at a suspension temperature of 80 °C.

Type of alkali-activated solution	Unsaturated lime solution	Saturated lime solution	OPC solution
(*a*) Initial electrical conductivity (mS/cm)	3.31	4.49	6.77
(*b*) Final electrical conductivity after stabilization (mS/cm)	0.36	0.84	2.85
(*c*) Loss in electrical conductivity (mS/cm); (*c*) = (*a*) – (*b*)	2.95	3.65	3.92

Table 3
The lime: pozzolan mass ratio considered in the different studies.

Method	OPC:pozzolan mass ratio	The lime:pozzolan mass ratio
Strength activity index [1]	4:1	1:1
Frattini test [14]	4:1	1:1
Saturated lime test (Donatello et al. [13])	–	0.15:1
Unsaturated lime test (Paya et al. [3])	–	0.04:1
This study and the author's method [12]	70:1	17.5:1

lan with respect to available $Ca(OH)_2$ in the OPC solution. However, it was not actually possible to increase the available $Ca(OH)_2$ in the OPC solution because it was already supersaturated with $Ca(OH)_2$ [6]. It was also difficult to raise the concentration level of the solution greater than the solubility product.

This study aimed to use the smallest amount possible of SF in the rapid evaluation of pozzolanic reactivity. Therefore, 1.0 g of SF sample in 200 mL of OPC solution was suitable for evaluating pozzolanic activity. This dosage indicated a more rapid chemical reaction rate as compared with a dosage of 0.5 g, and the time required for stabilization was more or less the same as for 1.5 g dosage.

3.3.1. Lime:pozzolan mass ratios

Table 3 illustrates the lime:pozzolan mass ratios (L/P) considered in different studies. Beside, mass of $Ca(OH)_2$ in three methods, including strength activity index [1], Frattini test [14] and the author's method [12], were approximated as 25% of the OPC mass [13]. This table shows that the method used in this study is the highest L/P ratio (rich activator) while unsaturated lime test [3] is the lowest L/P ratio. Donatello et al. [13] discussed that when assessing the pozzolanic activity of a material, it is important to take into account the method used and the most important factor when comparing tests is L/P ratio while uncertainties in the absolute amount of $Ca(OH)_2$ in each sample of the low activator to pozzolan methods may introduce errors. In addition, the effect of increasing the L/P ratio can improve the correlation between strength activity index and electrical conductivity measurements of pozzolanic activity as revealed in previous study [12]. Therefore, the high L/P ratio of the method used in this study may appropriate in the rapid evaluation of pozzolan.

It is well known that silica fume (SF) is an excellent pozzolan so it was used as a sample of pozzolan to study the influence of alkaline solution types, reaction temperatures, and dosage of sample to use in electrical conductivity measurements of pozzolanic activity. Although, this article studies only SF, the research potentially applies to pozzolanic materials in general. The obtained results, discussion and limitations in this study could be used for further development of a more accurate, rapid evaluation of pozzolanic activity.

4. Conclusion

Based on the results obtained from this experiment, the following conclusions can be drawn:

(1) Ordinary Portland cement solution, which is produced from Portland cement type I and water, could be used as an alkaline solution for pozzolanic activity evaluation. It is observed that the highest losses in electrical conductivity occur for the OPC solutions compared with unsaturated and saturated lime solutions.
(2) Raising the suspension temperature accelerates the pozzolanic reaction of SF in an alkaline solution – the higher reaction temperature the higher losses in electrical conductivity. The optimum suspension temperature of 80 °C is appropriate and recommended for the rapid evaluation of pozzolanic activity of pozzolanic materials.
(3) Increasing the amount of SF increases the chemical reaction rate. Among the three dosages of silica fume tested in this study, it was found that using 1.0 g of silica fume and 200 mL of OPC solution was appropriate in the rapid evaluation of silica fume.
(4) The results obtained in this study could be used for the further development of accurate, more rapid evaluations of pozzolanic activity.

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