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Using "Heat Treatment" Method for Activation of OPC-Slag Mortars

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Additional information is available at the end of the chapter

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1. Introduction

1.1. General

It is well known that a lot of ground granulated blast furnace slag (ggbfs) is produced in the steel-iron industry every year throughout the world. By utilizing this by-product it would help reduce the environmental problems and also provide significant economic benefits. The results of several researches have also shown that the use of replacement materials in mortars and concretes improves durability, which is crucial for structures built in aggressive environments, e.g. in marine structures and structures such as large tunnels and bridges with long life spans. For every ton of Portland cement manufactured, approximately one ton of CO₂, in addition to greenhouse gases, is released into the atmosphere. Therefore, if the part of the Portland cement can be replaced by waste materials, e.g., slag, then the amount of cement needed and hence, the amount of CO₂ released into the atmosphere can be reduced (Lodeiro, Macphee, et al., 2009). Consequently, ggbfs is being widely used as a cement replacement in Portland cement mortar and concrete for improving mechanical and durability properties.

The use of ggbfs has certain advantages because of its excellent cementitious properties over OPC and it is sometimes used due to the technological, economic and environmental benefits. However, the use of slag has been limited because of the disadvantage of its low early strength (Bougara, Lynsdale, et al., 2009). The major factors affecting the early age strength development of mortars and concrete are as follows:

- Mortar mixture proportions including water-binder and sand-binder ratios and the use of supplementary cementing materials such as ggbfs,
- Kind of formwork and size of structural elements, and
- Environmental conditions (Barnett, Soutsos, et al., 2006).

Concretes made with ggbfs have many advantages including improved durability, workability and economic benefits.

Mortar is a workable paste used to bind construction blocks together and fill the gaps between them. The blocks may be stone, brick, cinder blocks, etc. Modern mortars are typically made from a mixture of sand, a binder such as cement or lime, and water. Based on (ACI, 2006) there are four different types of mortars commonly used in building projects namely Type N, Type M, Type S, and Type O. Type N mortar is a medium strength mortar, which means that it is suitable for use both on indoor projects and on outdoor projects that are above grade. Type M mortar is a high strength mortar. Due to the strength of Type M it is usually used in heavy load bearing walls, although it is also sometimes used in other heavy duty applications like masonry that is below grade or that comes in contact with the earth such as retaining walls or foundations. Type S mortar is also a relatively high strength mortar which is suitable for below grade projects and heavier outdoor projects. Type O mortar is the lowest strength mortar and is suitable only for indoor, lightweight applications. The most commonly used mortars for most home improvement projects are Type N and Type S. Type N is chosen for lighter weight or indoor projects, and Type S for projects that require a heavier duty mortar. All of the different types of mortar are made with the same ingredients. The only difference is the proportions of each ingredient in the mix whether for availability considerations or for minimizing the number of different mortar types on the job site. The OPC-slag mortars can be classified into three groups as OPC mortars (OMs), slag mortars (SMs), and OPC-slag mortars (OSMs).

1.2. Research significance

Based on the related literature review there is not much research work regarding activation of OPC-slag mortars, and this is the main purpose of this investigation. In this study, the thermal activation method was used. The compressive strength loss was studied in this research at early and later ages. Strength development of OPC-slag mortars without and with use of activation method was also studied for duration up to 90 days and some regression relationships was suggested. Using the suggested relationships a criterion to forecast the strength behavior of OPC-slag mortars at later ages is established.

1.3. Research objectives

The objectives of this research are as follows:

- To determine the optimum replacement level of slag and the control ordinary Portland cement-slag mortar mix.
- To investigate the effects of thermal activation method on both the early and ultimate compressive strengths and also strength loss of the control ordinary Portland cement-slag mortar.

1.4. Scope of work

The objective of this study is to use higher percentage of replacement slag as possible without any reduction in mechanical properties of mortar such as compressive strength.

Different amounts of slag as replacement for cement were used and the optimum level of replacement was determined. In this investigation only one source of slag was used and the optimum level of replacement was used throughout the study. The optimum level was based on high early strength and lowest strength loss for the mortar mixes.

The focus of this project is to activate OPC-slag mortars using heat treatment (thermal activation method). In this activation method different temperatures within the range of 40 °C to 90 °C were used for activation. The mortars were heated for duration of 2 to 26 hours. The mechanical properties studied in this research are compressive strength at the both early and later ages, strength loss and strength development at later ages of OPC-slag mortars in duration of up to 90 days. The compressive strengths throughout the study were tested for the specimens at 1, 3, 7, 28, 56, and 90 days.

2. Determination of optimum level

2.1. Introduction

Before discussing the activation method, the determination of the optimum level of replacement of slag in OPC-slag mortars is discussed.

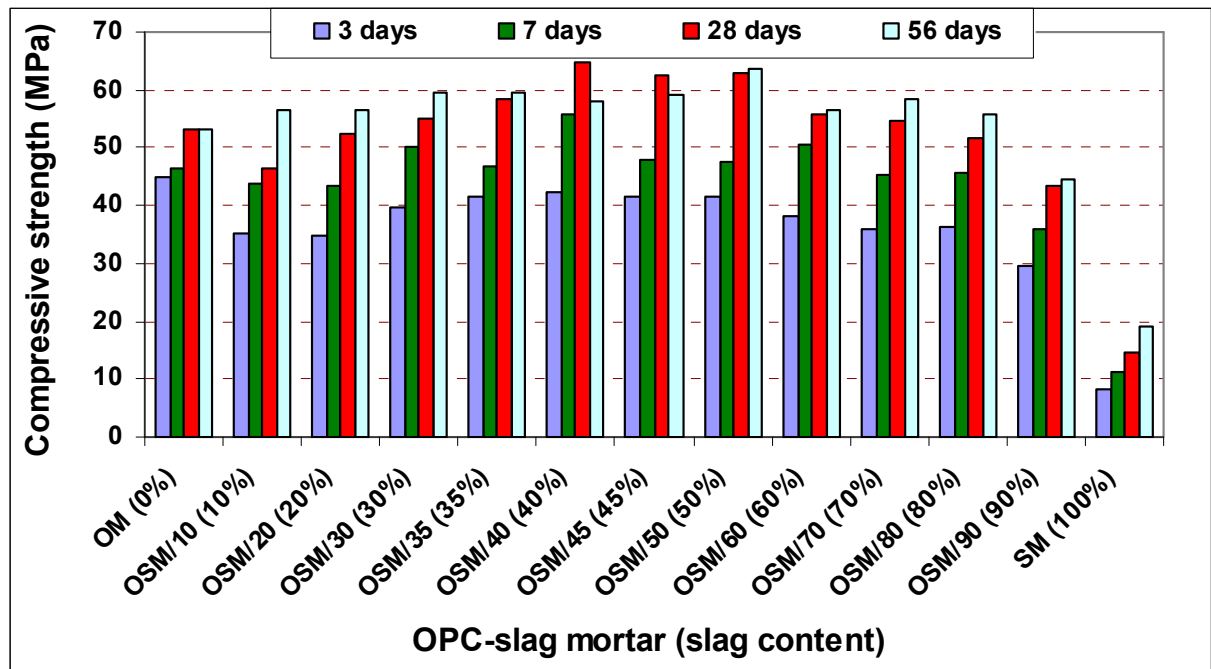
2.2. Replacement level

This section reports on the testing of fourteen OPC-slag mortars (OSMs) and two control OPC mortars (OMs) and slag mortars (SMs). The main aim is to determine the level of cement replacement with slag to achieve higher early strength with reasonable flow. The variable is the level of ggbs in the binder. Graded silica sand was used in all mixes. It was determined that the optimum level of replacement slag is within the range of 40% to 50% of OPC (Ahmed, Ohama, et al., 1999). The optimum level is defined as the replacement level of slag with the highest compressive strength, when used in the mortar while strength loss is the lowest.

2.2.1. Optimum replacement level

It is intended to find the optimum cement at replacement level with slag that gives the highest early strength at 7 days and especially 3 days without the use of any activation method. From Figure 1 it is clear that whenever the level of replacement is more than 40% the early strength at 3 days will be reduced. It can also be seen that although for replacement levels 10%, 20%, and 30% the early strength at 3 days is approximately the same, but, generally with an increase in the slag level from 10% to 40% the early strength increases. However, the early strength at the 40% level is the highest, i.e. 42.4 MPa. Based on the results obtained it can be seen that by increasing the replacement level to more than 40%, the early strengths decreased significantly. This shows that the optimum level of replacement is 40% at 3 days. The same variations for 7-day strengths were observed albeit with slight changes. At 40% optimum level of replacement slag, the strength at 7 days is 55.8

MPa, which is 20% higher than that of OPC mortar. Furthermore, this will continue to gain strength with age.



Notes: OSM/i= OPC-slag mortar for i% replacement with slag, OM= OPC mortar, SM= slag mortar.

Figure 1. Variations of compressive strength for different slag contents (%)

The use of different amounts of slag in OPC-slag mortars results in different compressive strengths. Before applying the proposed activation method it is required that several mortars are made with the use of different levels of slag to obtain indication of strengths. Based on this, it was decided to cast samples with 0, 10, 20, 30, 35, 40, 45, 50, 60, 70, 80, 90, and 100% replacement with slag to ascertain the optimum level. The specimens were prepared and hardened samples were tested at 3, 7, 28, and 56 days for compressive strength. Finally, it was revealed that the highest compressive strengths were obtained for samples having replacement level in the range of 40% and 50%. The results showed that there was some strength loss at 56 days compared to 28 days when 40% replacement level was used. In this case, the strength at 28 days was 64.9 MPa, which reduced to 57.9 MPa at 56 days giving a 10.8% loss in strength. In contrast no strength loss was observed at later ages when 50% replacement level was used. A comparison of the results obtained for both percentage levels of slag showed that 50% is the optimum. The strengths for both levels of replacement were very close but, the 50% replacement with slag did not show any subsequent loss in strength. Consequently, in the continuation of the research work this level was taken as the optimum and used for the preparation of mortar samples.

2.3. Summary

In this section it was attempted to determine the best replacement level for slag. Different levels of replacement slag were used to make mixes, i.e. 10, 20, 30, 40, 50, 60, 70, 80, and 90%.

Finally, with comparison of the results obtained for compressive strengths the best level of replacement was determined at 50%. In determination of the best level two factors are considered i.e. early strength at 3 and 7 days and also strength loss that should be minimized. In following parts of the research the best level i.e. 50% was used as the optimum level.

3. Thermal activation method

The objective of this research is to produce a data inventory of the early age mechanical properties, namely the compressive strength of mortars cured at different temperature, as well as the relationship between compressive strength with temperature and the relationship between the compressive strength of specimens cured in air and water at room temperature for 3 and 7 days, for 40% and 50% levels of cement replacement with slag. Thirty-seven mixes of OPC-slag mortars and two OPC mortars were prepared as control. For each mix, two factors are important for consideration. First, using a higher percentage of slag is desirable as it has some economic and environmental advantages and in addition, it helps to improve the durability of the mortars. Secondly, for early strength, it is clear that increasing the level of replacement slag causes early strength to be reduced, as the ggbfs has lower initial heat of hydration than that of OPC. In addition, for early strength the use of a low level of replacement slag is neither economic nor durable. Thus it is desirable to ascertain the optimum temperature and its duration that will give the highest early strength at 3 and 7 days. All the mix proportions made for water-binder and sand-binder ratios of 0.33 and 2.25, respectively, for 40% and 50% replacement level with slag.

3.1. Optimum temperature

In this investigation the effects of different temperatures i.e. 50 °C, 60 °C, and 70 °C were studied on the early strengths at 3 and 7 days of OPC-slag mortars by using 50% replacement with slag. The results are shown in Figure 2. It is clear that 60 °C provided the most enhancements on early age strength therefore; it is selected as the optimum temperature.

The results obtained in the study for compressive strength based on duration of heat curing are given in Table 1. Based on this, it can be seen that the specimens have higher strengths at 3 and 7 days without use of heat curing and with use of heat curing for duration of 2 hours when they are cured in water compared to curing in air under room temperature.

This has been proven for both OPC-slag mortars with 50% OPC replacement with slag and OPC-slag mortars with 40% OPC replacement with slag. However, as soon as the duration of heat curing is increased to 4 hours and above, the aforesaid statement is reversed. The strength of specimens cured in the air under room temperature is improved compared to those cured in the water. It seems that this is due to the air temperature and high relative humidity of the room's air. As it will be seen in following study, it can be said that both combined effects of temperature and relative humidity are more efficient in strength improvement. Hence, it seems that probably the effect of temperature for duration of at least 4 hours beside the high relative humidity of room's air results in the higher strength for the specimens cured in the air under room temperature after heat curing. This fact is shown in Figure 3 (a) and (b).

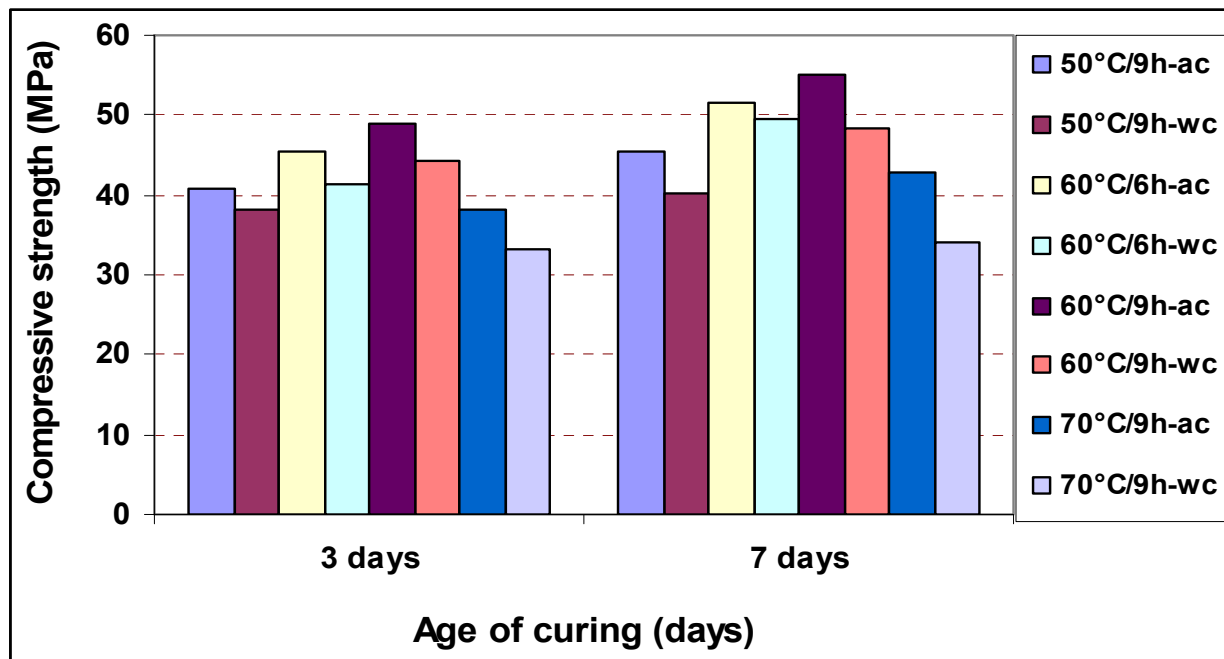
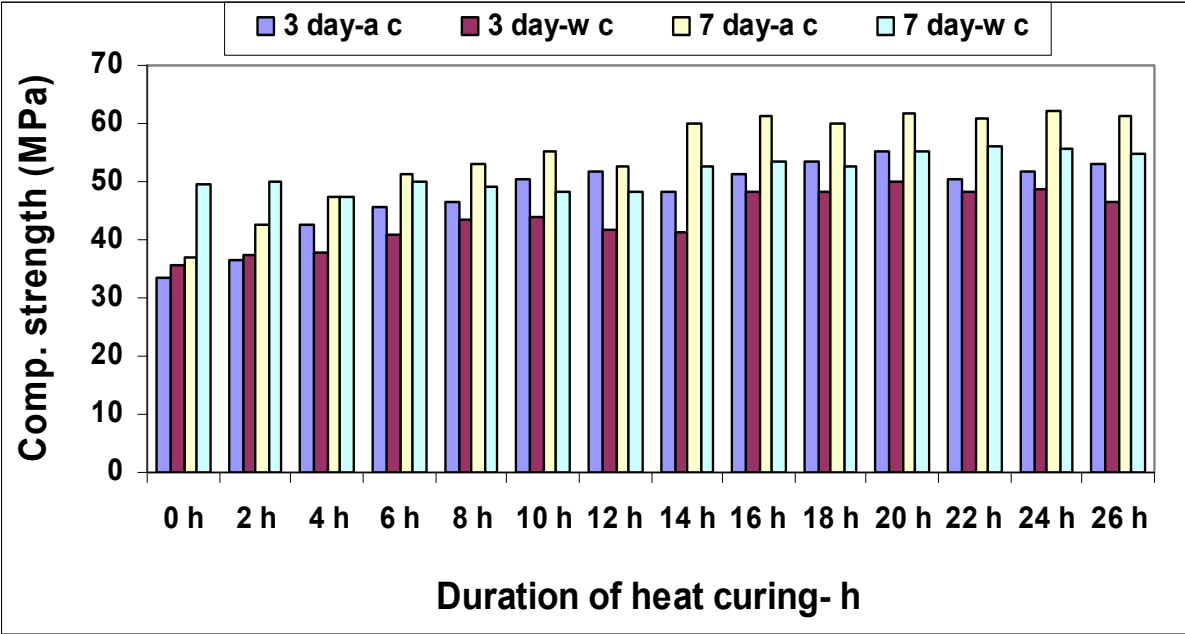


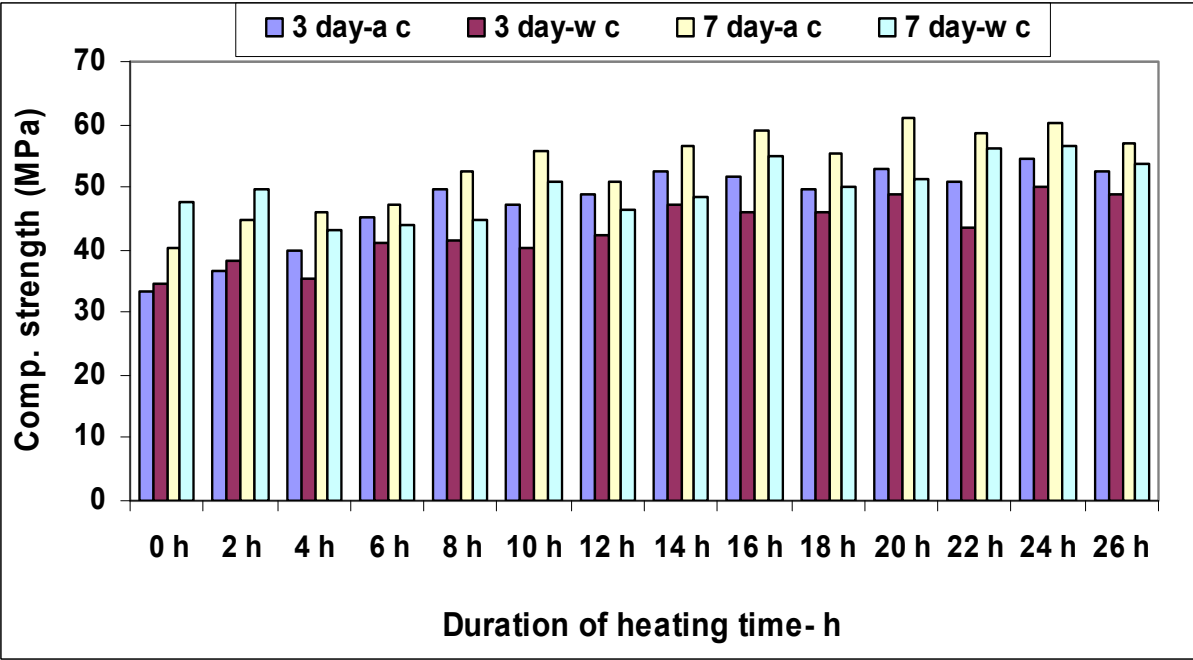
Figure 2. The effects of different temperatures on early age strength of OSMs/50

Duration (hours)	For OSMs/40				For OSMs/50				
	3 days		7 days		3 days		7 days		
	ac	wc	ac	wc	ac	wc	ac	wc	
0	33.2	34.5	40.3	47.4	33.6	35.6	37.0	49.6	
2	36.6	38.4	44.8	49.8	36.4	37.6	42.5	50.0	
4	39.7	35.4	46.2	43.2	42.6	37.7	47.5	47.3	
6	45.0	41.2	47.2	44.0	45.5	40.9	51.5	49.8	
8	49.6	41.6	52.3	44.8	46.4	43.4	53.1	49.0	
10	47.3	40.4	55.6	50.9	50.4	44.0	55.0	48.4	
12	49.0	42.5	50.9	46.4	52.6	41.8	57.6	48.3	
14	52.6	47.0	56.4	48.5	48.3	41.2	60.0	52.5	
16	51.7	45.9	59.0	54.8	51.2	48.4	61.2	53.4	
18	55.2	46.1	59.7	50.2	53.5	48.4	59.9	52.4	
20	53.1	49.0	61.1	51.2	55.3	49.9	61.6	55.3	
22	50.7	43.7	58.8	56.0	50.5	48.4	61.0	56.0	
24	54.6	50.1	60.4	56.8	51.5	48.5	62.3	55.6	
26	52.5	49.0	57.1	53.7	53.0	46.6	61.2	54.8	
For optimum OSM/50 at six ages- air cured under room temperature									
f ₁ = 15.5	f ₃ = 55.1		f ₇ = 61.4		f ₂₈ = 71.2		f ₅₆ = 69.6		f ₉₀ = 73.6
ac= air curing under room temperature; wc= water curing; all strengths are in MPa.									

Table 1. Compressive strength (f) versus duration of heat curing for OSMs/40 and OSMs/50 at 60 °C



(a) OPC-slag mortar with 50% OPC replacement with slag at 60 °C



(b) OPC-slag mortar with 40% OPC replacement with slag at 60 °C

Figure 3. Compressive strength versus duration of heat curing of OSMs/50 and OSMs/40 cured in water and air under room temperature.

Based on the results given in Table 1, it can be seen that heat curing at 60 °C for 20 hours, the maximum early strength of 61.6 MPa is attributed to OPC-slag mortar with 50% replacement with slag. It can be seen that the increment percentage is by about 0.62% when compared to a heating time of 16 hours. This shows that if the duration of heat curing is

increased over 20 hours, the increase at 7 days strength is not appreciable. Hence, it can be deduced from Figures 3 and 4 that heat curing at 60 °C for 20 hours is the optimum heat curing for the materials used in the study.

3.2. Temperature and humidity effects

From the H- 3 sets mix as shown in Table 2 the effects of ambient temperatures and relative humidity were considered on the strength improvement of the specimens. The specimens were first made and demoulded 24 hours after casting, and then heated at 60 °C for 20 hours and finally, cured in three curing regimes, i.e. at room temperature, in water at 25 °C - 26 °C, and in water at 32 °C. After 3 and 7 days the strength of specimens was determined. The results are given in Table 2.

Based on the given data in Table 2, it is clear that for curing regimes with different temperature and the same relative humidity, higher strengths are attributed to higher temperature regimes. For curing regimes with different relative humidity and the same temperature, higher strengths are attributed to lower relative humidity regimes. From the comparison of the three curing regimes it is seen that the strengths of specimens cured in water at 32 °C are less than those cured at room temperature. This shows that the increase of early age strength is a function of both temperature and relative humidity effects. In fact, it can be deduced that neither relative humidity nor temperature has the highest strength improvement alone, but rather a combination of both effects are effective on strength development. From the results it is clear that the trend of strength development at 7 days is similar to that at 3 days. It is seen that the strength of specimens at 7 days cured at room temperature and in water at 32 °C are the same, which shows that the effect of relative humidity over duration of 3 to 7 days is more than that of temperature. It is seen that the highest strengths at 3 and 7 days are attributed to curing at room temperature.

The percentages of strength growth (i) for duration of 3 to 7 days can be obtained as follows: For air cured specimens under room temperature $i = 0.97$, for water at 25 – 26 °C $i = 0.90$, and for water cured at 32 °C $i = 0.89$; where $i = \text{ratio } f_3/f_7$. These results show that whenever the specimens are cured at room temperature, about 97% of the strength is achieved at 3 days. This is a major advantage in the precast concrete industry when the specimens are cured at room temperature. For the specimens cured at room temperature, the maximum relative humidity attainable was 85%, while for the specimens cured in water it was 100%.

Based on the results obtained it is evident that the effect of temperature on the strength improvement at 3 and 7 days is higher than that of relative humidity. This is because the strengths of the specimens cured at room temperature and in water at 32 °C are higher than those in water at 25 °C at 3 and 7 days by about 14% and 10%, respectively.

It can be seen that with the use of heat curing at 60 °C for 20 hours the strength at 3 days is, on average, about 97% of the strength at 7 days for specimens cured at room temperature, while, on average, this ratio is about 90% for curing in water at 25 °C and 32 °C. This shows that curing at room temperature after heat curing improves the early strength at 3 days

extensively, which is very cost effective and applicable in the precast concrete industry. This result also shows that the heat treatment is a useful and efficient method for the activation of ordinary Portland cement-slag mortars and concretes which requires only slat duration and without the use of water to cure the specimens. An elevated curing temperature accelerates the chemical reaction of hydration and increases the early age strength. However, during the initial period of hydration an open and unfilled pore structure of cement paste forms which has a negative effect on the properties of hardened concrete, especially at later ages (Fu, Y., 1996; Neville, A.M. , 2008). Hardened mortars and concretes can reach their maximum strength within several hours through elevated temperature curing. However, the ultimate strength of hardened mortars and concretes has been shown to decrease with curing temperature (Carino, 1984). It was found that by increasing the curing temperature from 20 °C to 60 °C and the duration of heat curing to 48 hours causes a continuous increase in compressive strength (Brooks & Al-kaisi, 1990). Studies by (Hanson, 1963; Pfeifer & Marusin, 1991; Shi, 1996) have shown that there is a threshold maximum heat curing temperature value in the range of 60 °C to 70 °C, beyond which heat treatment is of little or no benefit to the engineering properties of concrete.

Based on the given data in Table 1 it can be seen that the highest strengths at 3 and 7 days of OPC-slag mortars for 40% replacement with slag and OPC-slag mortars for 50% replacement with slag is attributed to the specimens cured in air under room temperature as:

Curing regime	air curing under room temperature	Water 32 °C	Water 25 -26 °C
f_3	58.2	53.3	48.9
f_7	59.9	59.9	54.5
f_3/f_7	0.97	0.89	0.90
f_i are strength in MPa.			

Table 2. Compressive strengths (f) at 3 and 7 days for three curing regimes of H- 3 sets mix

OPC-slag mortars for 40% OPC replacement with slag: f_3 = 55.2 at 18 hours and f_7 = 61.1 MPa at 20 hours; OPC-slag mortars for 50% replacement with slag: f_3 = 55.3 and f_7 = 61.6 MPa, the both for 20 hours. The 3 and 7 days strengths of OPC mortars' specimens cured at room temperature and in water are f_3 = 45.4, and f_7 = 51.4 MPa, and f_3 = 43.8, and f_7 = 47.8 MPa, respectively. It is noted that the maximum 3 and 7 days strengths of OPC-slag mortars for 40% replacement with slag and OPC-slag mortars for 50% replacement with slag specimens are 21.7% and 19.0% which are 21.8% and 20.0% more than those of OPC mortars' specimens cured at room temperature at the same age, respectively. It is seen that there is strength loss at 56 days compared to 28 days by about 2.2%. This has been previously reported by other researchers (Kosmatka, Panarese, et al., 1991). The main objective of elevated temperature curing is to achieve early strength development. However, it is generally acknowledged that there is also strength loss as a result of heat curing (Bougara, Lynsdale, et al., 2009). Another mix proportion of OPC-slag mortars for 50% replacement with slag was made by using the optimum heat curing at 60 °C for 20 hours and specimens were tested at ages of 1, 3, 7, 28, 56, and 90 days. To determine the trend of strength

development for the mentioned mortar cured at room temperature, the regression technique was used. The equations obtained for this mortar and also OPC mortar cured in water are as below:

$$f_{T-ac} = 5.2128 * \ln(t) + 50.644 \text{ with } R^2 = 0.9305 \quad (1)$$

For OPC-slag mortar cured in air under room temperature, made using optimum heat curing at 60 °C for 20 hours, and

$$f_{OM-wc} = 6.1673 * \ln(t) + 35.141 \text{ with } R^2 = 0.9738 \quad (2)$$

For OPC mortar cured in water; where f is compressive strength in MPa, ac and wc denote air and water curing under room temperature, respectively and t is the age of the specimen in days. The best fit curves are shown in Figure 4.

No	Age (d)	Binomial relationships	Linear relationships	CR
For OPC-slag mortars for 40% replacement with slag, i.e. OSMs/40				
1	3	$f = -0.0455X^2 + 1.865X + 32.921$; $R^2 = 0.9261$	$f = 0.6825 X + 38.651$ $R^2 = 0.7545$	air
2	3	$f = -0.0131X^2 + 0.8806 X + 34.825$; $R^2 = 0.8502$	$f = 0.5391 X + 36.191$ $R^2 = 0.8252$	water
3	7	$f = -0.0347 X^2 + 1.5959 X + 40.621$; $R^2 = 0.9107$	$f = 0.6927 X + 44.234$ $R^2 = 0.8068$	air
4	7	$f = 0.0163 X^2 - 0.0222 X + 46.327$; $R^2 = 0.6305$	$f = 0.4011 X + 44.634$ $R^2 = 0.5815$	water
For OPC-slag mortars for 50% replacement with slag, i.e. OSMs/50				
5	3	$f = -0.0487 X^2 + 1.9196 X + 34.298$; $R^2 = 0.9271$	$f = 0.6526 X + 39.366$ $R^2 = 0.7212$	air
6	3	$f = -0.0184 X^2 + 0.9857 X + 35.234$; $R^2 = 0.8492$	$f = 0.5066 X + 37.154$ $R^2 = 0.7954$	water
7	7	$f = -0.0479 X^2 + 0.1079 X + 38.56$; $R^2 = 0.9598$	$f = 0.8628 X + 43.54$ $R^2 = 0.8291$	air
8	7	$f = 0.0115 X^2 + 0.0108 X + 48.789$; $R^2 = 0.7742$	$f = 0.3089 X + 47.597$ $R^2 = 0.7232$	water
X= heat duration in hours, f is compressive strength in MPa, R^2 is coefficient of determination, CR= curing regime, d= days				

Table 3. Relationship between compressive strength (f) versus heat duration

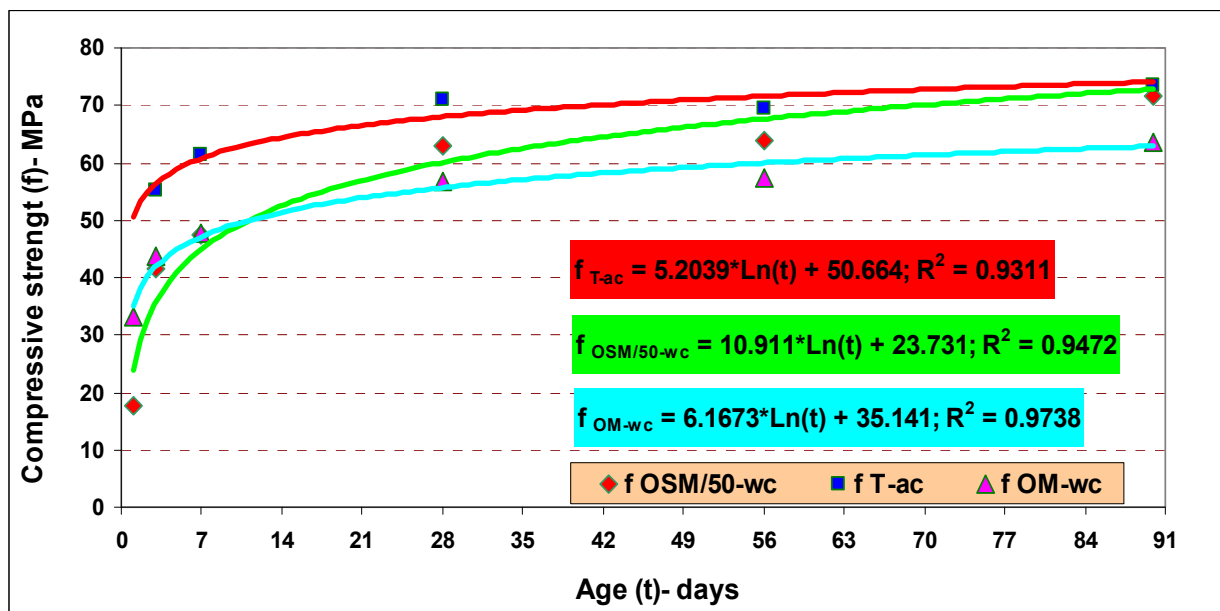
The relationships between compressive strength and duration of heat curing at room temperature and in water for OPC-slag mortars at 40% OPC replacement with slag and OPC-slag mortars at 50% OPC replacement with slag is shown in Table 3. It can be seen that the best equations are binomial and attributed to the specimens cured at room temperature.

It is also seen that the best fit curve at 3 and 7 days strengths are power equations. According to the results obtained in the study, it can be said that thermal activation is one of the best techniques for the activation of OPC-slag mortars.

No	Age (days)	Power regression relationship	Curing
1	3	$f_{\text{OSM}/50} = 1.3991 \cdot x^{0.9147}$; $R^2 = 0.8857$	air
2	7	$f_{\text{OSM}/50} = 0.4548 \cdot x^{1.2047}$; $R^2 = 0.9334$	air
3	3	$f_{\text{OSM}/50} = 2.098 \cdot x^{0.8064}$; $R^2 = 0.7349$	water
4	7	$f_{\text{OSM}/50} = 5.9897 \cdot x^{0.5511}$; $R^2 = 0.6897$	water

x= compressive strength of OSM/40 in MPa, R^2 = coefficient of determination.

Table 4. Relationships between compressive strengths (f) of OSMs/50 and OSMs/40



Notes: OM= OPC mortar, T= OPC-slag mortar for 50% OPC replacement with slag made by using the optimum heat curing, f= Compressive strength of the specimens in MPa, ac and wc denote air and water curing under room temperature, respectively.

Figure 4. Strength development for OPC mortar and OPC-slag mortar made by using the optimum heat curing

Based on the results presented in Table 4, it can be seen that there is an acceptable power relationship at 3 and 7 days strengths between OPC-slag mortars for 50% OPC replacement with slag and OPC-slag mortars for 40% OPC replacement with slag for the specimens cured at room temperature.

The given relationships in Table 4 were determined by using the regression technique. Based on the relationships it is seen that the coefficient of determination R^2 of regression for relationships between the strengths at 3 and 7 days of water cured OPC-slag mortars for 50% OPC replacement with slag and OPC-slag mortars for 40% OPC replacement with slag is small. This shows that there is no acceptable relationship between the strengths of water cured specimens. However, there is a proper relationship for those cured at room temperature.

This appears to be due to the behaviour of specimens cured in water, which are still not clear and that is specific for the duration of 7 days. This means that the effect of water on the strength of water cured specimens is different for the durations of 3 and 3 to 7 days.

3.3. Ettringite formation

In this study four extra sets of specimens were tested for SEM images and EDX analyses of H- 4 sets mix given in Table 5 after curing for 7 days. SEM images and EDX analyses were tested on four specimens of the four sets. Each specimen was analyzed to determine if ettringite was present. If ettringite was detected its morphology was noted. It is impossible to quantify ettringite because of the scaling factor and depth of uncertainties of the electron microscope. However, it is significant to note that very small samples measuring approximately 5 mm in diameter yielded large quantities of ettringite. Ettringite was identified visually and from the EDX analysis. The morphologies of the observed ettringite are summarized in Table 5. Samples typically produced ettringite with similar morphologies including lamellar and needles. Ettringite was found in cavities and in the cement matrix.

Set No.	Curing regime	Ettringite formation
Set 1	With heat curing, air cured	Thick and long needles
Set 2	With heat curing, water cured	Needles with lamellar
Set 3	Without heat curing, air cured	Needles with lamellar
Set 4	Without heat curing, water cured	Needles with dense lamellar
Heat treatment was done after demoulding at 60 °C in duration of 20 hours.		

Table 5. Summary of scanning electron microscopy morphology for H- 4 sets mix after 7 days curing

The mechanism of DEF expansion is a highly debated issue. Ettringite Crystal Growth Theory and Uniform Paste Expansion Theory are the two predominant theories. (Shoaib, Balaha, et al., 2000) and (Wang, Pu, et al., 1995) suggested the Ettringite Crystal Growth Theory, which attributes the expansion to pressure exerted by the growing ettringite crystals in the micro cracks between the cement paste and the aggregate.

(Wang, Scrivener, et al., 1994) proposed the Uniform Paste Expansion Theory, which suggests that the concrete expands and then the ettringite forms in the newly created gaps. (Yang, R., Lawrence, et al., 1996) found no evidence to support the Uniform Paste Theory concluding that the ettringite present in the mortar produced the expansion. (Lewis, M. C. & Scrivener, 1996) suggests that both mechanisms are possible and depending on the environmental condition one may be more prevalent. Although other mechanisms have been suggested, expansion most probably results from crystal growth pressure. There are differences of opinion as to whether expansion in mortars or concretes is driven by growth of ettringite crystals at aggregate interfaces or by processes occurring in the paste. If the later expands, gaps will be formed around aggregate particles (Wang, Scrivener, et al., 1994), and ettringite or other phases may recrystallize in them, simultaneously or subsequently. Based on the paste expansion theory the widths of peripheral cracks are proportional to aggregate size; cracks at the interfaces are initially empty. Assuming that expansion occurs through

crystal growth pressure, significant growth pressures could not be obtained in relatively large cracks and pastes expand, albeit slowly. Three factors influencing expansion will be considered namely chemistry, paste microstructure, and mortar or concrete microstructure. Proportionality between crack width and aggregate size, which can only be explained by paste expansion, was first reported by (Johansen, Thaulow, et al., 1993). For the H- 4 sets mix 24 hours after casting, the first two sets of the specimens were demoulded and without heat curing but were cured in water and air under room temperature for 7 days, respectively. Another two sets were heated at 60 °C temperature for 14 hours and then cured the same as the former sets. Ettringite was observed in all of the sets of specimens i.e. with and without heat curing, but with different amounts of crystal size. The results are shown in Figure 5.

The morphology and crystal size of ettringite varies under the different curing conditions the specimens were subjected to. Most of the SEM observation shows that ettringite is normally a slender, needle-like crystal with a prismatic hexagonal cross-section. Its size depends on w/c ratio, that is, the effective space that ettringite is able to occupy (Barnett, Soutsos, et al., 2006). It can grow up to 100 μm long for high w/c ratios. The particle-size of Al-bearing agents is also a main factor affecting the size of ettringite. Large particles of Al-bearing agents form a large amount of small ettringite crystal and the period of expansion can last longer. Small particles will produce large size crystals quickly at any early stage because of the large surface area and fast reaction rate. The ettringite crystals will also be smaller in size in the presence of calcium hydroxide (CH) (Barnett, Soutsos, et al., 2006). The form of ettringite is relevant to studies of the mechanism of expansion. (Lerch, Ashton, et al., 1929) reported that synthetic ettringite consists of long slender needles that often form sphere-like. (Mehta, P.K., 1976) also reported the presence of spheroid ettringite. (Ogawa & Roy, 1982) found that during the hydration, the ettringite formed as very small irregular particles around Al-bearing particles in the early stage, and then changes to long needle-like crystals arranged radially around the Al-bearing particles. This formation was associated with the start of expansion (Barnett, Soutsos, et al., 2006).

Comparison between Figure 5 (a), (b), (c) and (d) show that for each test and in all curing regimes ettringite crystals were detected. For the four sets of tests, the ettringite crystals formed under room temperature curing, i.e. Figure 5 (a), and were more and bigger than those under water curing. Probably, this is the reason for the higher strength improvement of the specimens cured in air under room temperature compared to curing in water. It can be observed that the thickest and the longest ettringite crystals are attributed to the specimens that were cured in the heating process at 60 °C for 14 hours after casting, and then cured at room temperature for 7 days as shown in Figure 5 part (a). The strength obtained at 7 days for the specimens was about 64 MPa. With the comparison of SEM images and the strengths obtained from the four sets of specimens, it can be deduced that heat curing at 60 °C for 14 hours increases the rate of ettringite formation and thus the early strength. Whenever there is a greater quantity of ettringite formed it contributes to the higher strength. The details obtained for the compressive strengths at 7 and 28 days, of H- 4 sets mixes are given in Table 6.

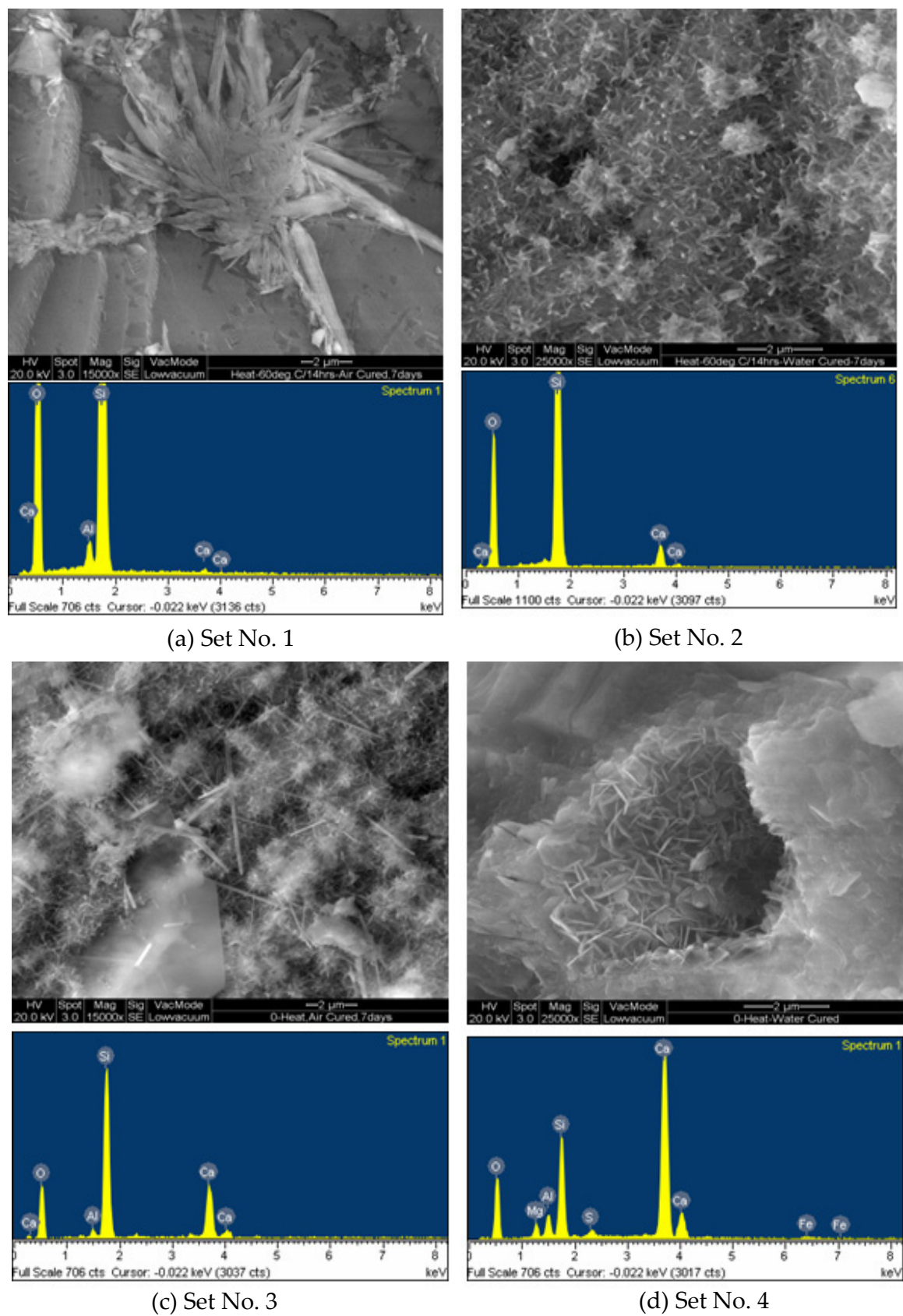


Figure 5. SEM images and EDX analyses for H-4 set mix specimens

Curing & Strength (MPa)	Type of curing regime			
	Without use of heat curing		Heat curing at 60 °C for 14 hours	
	Water cured (25 °C - 26 °C)	Room temp. Cured (32 °C)	Water cured (25 °C - 26 °C)	Room temp. Cured (32 °C)
f_7	53.7	38.7	57.6	64.0
f_{28}	61.6	44.5	63.2	64.2
f_7/f_{28}	0.87	0.87	0.91	1.00
f_{28}/f_7	1.15	1.15	1.10	1.00
SEM images	Figure 5, (d)	Figure 5, (c)	Figure 5, (b)	Figure 5, (a)

Table 6. Compressive strength (f) at 7 and 28 days for H- 4 sets mix

Based on the given data in Table 6 it is evident that the highest strength is obtained from air curing at room temperature after heat curing at 60 °C for 14 hours. Generally speaking, the results obtained can be discussed according to the different curing regimes.

The strength of specimens cured in air at room temperature after subjected to heat curing is the highest. This is followed by the specimens cured in water after heat curing and specimens cured under the same conditions but without heat curing. The lowest strength was obtained for the specimens cured in air at room temperature without heat curing. It was also observed that the thickest and the longest ettringite crystals were formed in the specimens with the highest strength as shown in Figure 5.

Similar to the strength at 7 days, it is seen that among the four curing regimes the strength of specimens at 28 days is the highest for those cured at room temperature after heat curing at 60 °C for 14 hours. From Table 6 it can be seen that the strength growth (i) at 28 to 7 days in different curing regimes is as follows: for without heat and curing in air at room temperature $i = 1.15$ and for curing in water $i = 1.15$. Use of heat curing at 60 °C for 14 hours followed by curing in air at room temperature $i = 1.00$ and for curing in water; $i = 1.10$.

From the results observed it is clear that in the cases without use of heat curing, the strength growth of 28 to 7 days is noticeable for both curing at room temperature and in water. The relative strength growth is on average about 1.15. This shows that there is a continuous hydration process progression for duration of 7 to 28 days. It seems that the latent potential of the specimens is gradually released, whilst whenever the specimens are heated, the whole latent potential is suddenly released during the initial days (in duration of the first days) due to the temperature effect. In the case with heat treatment, it is seen that the strength gain is completely different for the specimens cured at room temperature and in water. It is

observed that there is no significant strength growth at 28 days compared to 7 days. In fact, it can be said that when the specimens are cured at room temperature after heat curing, the highest strengths are achieved during the first 7 days. This is a major advantage to the precast concrete industry. However, when the specimens are cured in water after heat curing more time is needed to achieve maximum strength. This shows that curing in water is not the best way to cure the specimens after heat treatment from the standpoint of strength gain at early ages. Hence, the comparison of curing regimes at room temperature and in water shows that the best curing regime after heat curing is air curing under room temperature, especially for the precast concrete industry. It is also seen that in the case without heat curing, on average, the strength at 7 days is 87% of the strength at 28 days for both curing at room temperature and in water, and with heat curing this ratio is increased on average to 95%. This means that heat curing improves the strength at 7 days by an average of about 9%.

3.4. Strength loss

The compressive strength loss of mortars and concretes containing supplementary cementitious materials is quite common. In the process of this research, strength loss has been observed several times for some of the mortars prepared. This is because for a variety of reasons; some of the observed reasons in the study are as follows:

Several researchers reported that a high temperature improves strength at early ages (ACI, 2001; Al-Gahtani, 2010; Shariq, Prasad, et al., 2010). At later ages, the important number of formed hydrates had no time to arrange suitably and this caused a loss of ultimate strength. This behaviour has been called the crossover effect (Powers, T.C., 1947). For OPC it appears that the ultimate strength decreases nearly linearly, with curing temperature (Ramezaniapour & Malhotra, 1995).

Generally, the reason for the loss of strength can be due to internal or external factors. The internal factors are those linked to the chemical composition of the reacted products. The most efficient external factors are due to the variability of specimens and testing procedures. Another factor having high importance is the effect of temperature. The initial curing temperature has a significant effect and can reduce or increase strength at later ages. It seems that the main reason for strength loss at later ages is due to lack of inside water in specimens to complete the hydration and pozzolanic process progression. Usually for the duration of 1 to 28 days, the inside water due to the mixing water is available and adequate for the hydration process; however, beyond 28 days it is reduced and then insufficient for the process of hydration and pozzolanic reaction to progress; hence, strength loss occurs.

3.5. Strength development

Customarily, whenever it is wanted to understand the behaviour of a phenomenon, it is accepted to model its behaviour by the use of a diagram or mathematical relationship. Using

the relationships can approximately be forecasted the behaviour of the phenomenon at the later ages. In this research based on the results obtained for the OPC-slag mortars with 50% OPC replacement with slag under thermal activation method have been determined to forecast the variations of compressive strengths versus age of curing.

Comparison of all the relationships shows that the most appropriate form of equation to describe the variations of strength versus age of curing is a logarithmic function in the form of $f = a \cdot \ln(t) + b$; where R^2 is the coefficient of determination, a and b are constants for a specified mortar, f is compressive strength in MPa and t is the age of specimens in days.

The best fitted curves strength developments are shown in Figure 6.

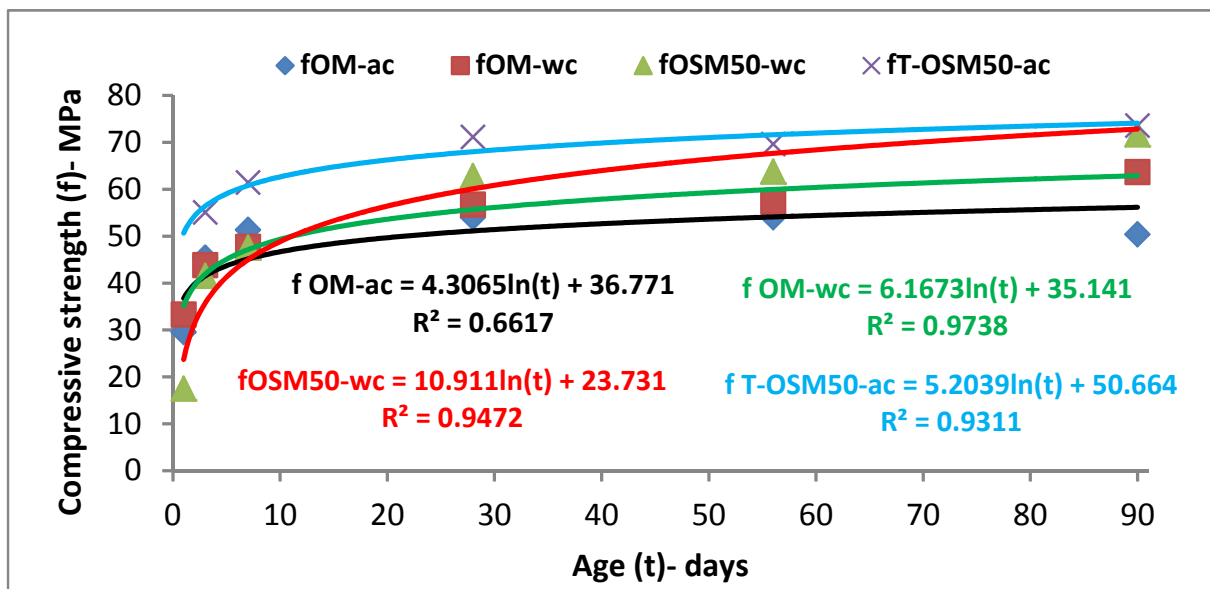


Figure 6. Strength development curve fitting for the optimum OPC-slag mortar activated using thermal activation method and OPC mortars

3.6. Summary

For different duration several temperatures such as 50 °C, 60 °C, and 70 °C were used. Based on the results obtained it can be deduced that heat curing at 60 °C for 20 hours is the optimum. It should be noted that 40% and 50% levels of replacement of OPC with slag were used and the results were compared. In addition to temperature, the effects of relative humidity were also studied. Finally, the results of compressive strength at 3 and 7 days versus heat curing were determined for 40% and 50% levels of replacement. It was recognized that the formation of long and thick crystals of ettringite was the main reason for significant strength improvement at early ages when the specimens were heated and then cured in air under room temperature. The flowchart of thermal activation method is shown in Figure 7.

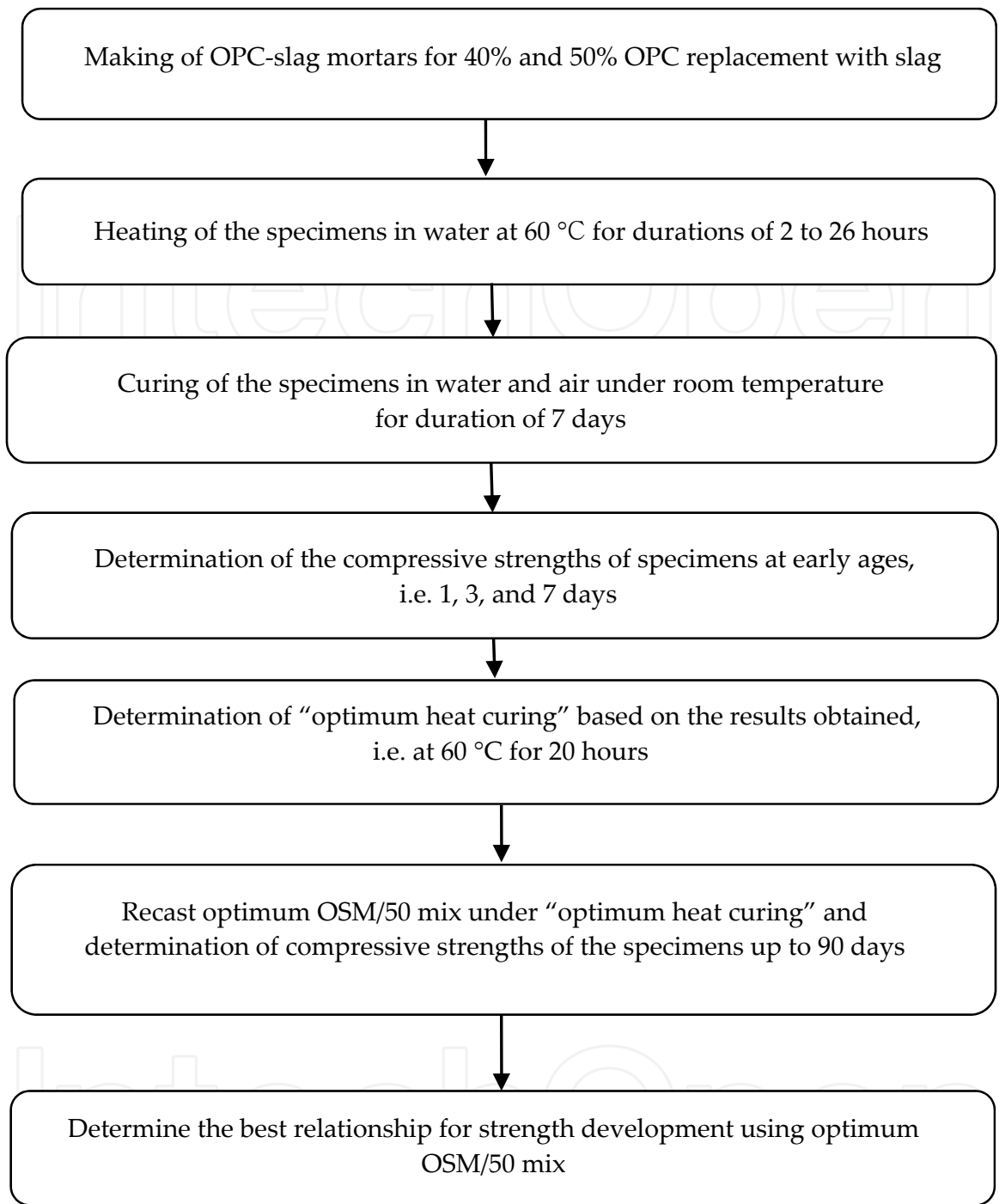


Figure 7. Thermal activation work phase

4. Conclusions and recommendations

4.1. Conclusions

4.1.1. Introduction

In this study 50% level of slag as the optimum was used as replacement for OPC. The thermal activation method (T) was used to activate ordinary Portland cement-slag mortars (OSMs).

4.1.2. Optimum replacement

For the slag used in this investigation, the optimum cement replacement level from viewpoint of high early strength was proven within the range of 40% - 50%. By using the optimum level of replacement slag, i.e. 50%, noticeable strength levels of OPC-slag mortars are achievable without the use of any activation method. In contrary to OPC-slag mortar for 40% replacement with slag, the other mortars made with different levels of replacement slag have shown higher 56-day strength compared to strength at 28-day, but the OPC-slag mortar for 40% OPC replacement with slag gives less 56-day strength compared to strength at 28-day by about 10.8%; namely compressive strength loss about 10.8%. Thus this strength loss phenomenon needs to be further investigated.

The highest growth of 56-day strength compared to 28-day are attributed to slag mortars and OPC-slag mortars for 10% replacement with slag (OSM/10) as 31.5% and 21.7%, respectively. This shows that the strength of mortar including the highest level of replacement slag will be improved at later ages more than others. However, it is well known that the ultimate strength of slag mortars is not significant compared to the strength of the others at the same ages. Therefore, it can be deduced that the slag mortars are the best only from viewpoint of durability.

4.1.3. Thermal activation method

It has been shown that the strengths of specimens cured in water at 3 and 7 days for OPC-slag mortar with 40% and 50% OPC replacement of slag, without and with use of heating for duration of 2 hours, are more than those cured in air under room temperature. However, as soon as the heating duration is increased to 4 hours and more, this effect is reversed. This is a new finding with a major advantage in precast concrete industry and also has many advantages in arid regions for curing of concrete structures.

Based on the experimental results obtained in the study, it can be concluded that there is an optimum temperature for each specific material to obtain high early strength. It was determined that 60 °C is the optimum. Heating duration is also very important for obtaining high early strength. For the slag used in the study, duration of 20 hours is optimum. Usually, as heating time increases towards the optimum, the compressive strength will be increased.

The maximum strengths obtained at 3 and 7 days for OPC-slag mortar with 50% OPC replacement of slag cured in air under room temperature are 55.3 and 61.6 MPa, respectively. It can be seen that these are 21.8% and 20.0% more than those of OPC mortar specimens cured in air under room temperature, and 26.1% and 29.0% more than those of OPC mortar specimens cured in water, respectively.

It was proven that whenever the mortar is heated larger than the optimum duration, it could be seen that this will not lead to an increase in early strength. According to the results of the study and other researches, it can be deduced that the thermal activation is one of the most efficient and applicable techniques for activation of OPC-slag mortars. This is well known specially in precast concrete industry.

The results obtained show that the best relationship of compressive strengths versus heating duration of the specimens cured in air under room temperature and water for OPC-slag

mortars at 40% and 50% OPC replacement with slag are power equations. A relationship exists between the compressive strength of the specimens cured in air under room temperature and water for OPC-slag mortar at 40% and 50% OPC replacement with slag at 3 days, but not at 7 days. Generally, comparison of OPC mortars heated in water bath or oven showed that water bath heat treatment gave better results than those of oven heated. It was also observed that the specimens gave higher strengths in air under room temperature compared to water curing after heating in the water bath at 60 °C for a duration of 20 hours. This is a significant finding with a major advantage in construction and is also economic since water bath heating can be practically implemented. However, oven heated pre-curing, results in higher strengths whenever the specimens are cured in water after heating.

4.2. Recommendations for future works

Among the mixes prepared by using different levels of OPC replacement with slag, mixtures with 50% ggbfs and 50% OPC show the highest strength in the absence of any activation method.

Based on the extra work done in this research by use of “mining sand” instead of “silica sand”, it was revealed that using mining sand is preferable to silica sands for activation of slag mortars, thus it is suggested to conduct a new study using mining sand.

In this study a single source of ggbfs was used throughout. It is recognized that other sources may have somewhat different chemical compositions. Thus other sources of the material need to be evaluated in order to determine the influence of the activation methods used in this study to be generalized.

Appendix

Mix proportions

No	Mix name	OPC (g)	Slag (g)	Flow (mm)	SP (g)	Water (g)
1	OM-wc	1200	0	225	40	421.11
2	OSM/10-wc	1080	120	225	68	421.11
3	OSM/20-wc	960	240	220	65	421.11
4	OSM/30-wc	840	360	220	60	421.11
5	OSM/35-wc	780	420	230	40	421.11
6	OSM/40-wc	720	480	210	48	421.11
7	OSM/45-wc	660	540	235	40	421.11
8	OSM/50-wc	600	600	235	40	421.11
9	OSM/60-wc	480	720	220	35	421.11
10	OSM/70-wc	360	840	230	35	421.11
11	OSM/80-wc	240	960	225	33	421.11
12	OSM/90-wc	120	1080	220	27	421.11
13	SM-wc	0	1200	220	30	421.11

Table 7. Mix proportions for determination of optimum level of replacement slag

No	Mix name	OPC (g)	Slag (g)	Water (g)	SP (g)	Flow (mm)
For OPC mortars, room temperature and water cured						
1	OM- ac	1800	-----	631.7	28	230
2	OM- wc	1800	-----	631.7	30	230
For OPC-slag mortars for 40% replacement with slag , cured in water and air under room temperature						
3	H0/0	720	480	421.11	28	225
4	H60/2	1440	960	842.22	82	230
5	H60/4,6	1440	960	842.22	90	230
6	H60/8,10	1440	960	842.22	79	230
7	H60/12,14	1440	960	842.22	79	230
8	H60/16	1440	960	842.22	82	230
9	H60/18,20	1440	960	842.22	73	230
10	H60/22,24,26	2160	1440	1263.33	70	220
For OPC-slag mortars for 50% replacement with slag, cured in water and air under room temperature						
11	H0/0	600	600	421.11	35	230
12	H60/2	1200	1200	842.22	76	235
13	H60/4,6	1200	1200	842.22	91	225
14	H60/8,10	1200	1200	842.22	90	235
15	H60/12,14	1200	1200	842.22	73	235
16	H60/16	1200	1200	842.22	76	235
17	H60/18,20	1200	1200	842.22	62	225
18	H60/22,24,26	1800	1800	1263.33	60	220
OSMs/50 test for three sets of specimens in cured room temperature and in water 25 °C and 32 °C after heat treatment 60 °C for duration 20 hours						
19	H- 3 sets mix	900	900	631.7	35	225
OSMs/50 test for four sets of specimens cured in room temperature and in water after with and without use of heat treatment 60 °C for duration 14 hours						
20	H- 4 sets mix	1200	1200	842.22	50	225
For optimum OSM/50 at six ages, only room temperature cured						
21	H60/20	900	900	631.7	43	230
H60/i,j,k means 60 °C temperature with duration i, j, and k hours						

Notes: OSMs/50= OPC-slag mortars for 50% replacement with slag, OMs= OPC mortars, SP= super plasticizer, ac= cured in air under room temperature, wc= water cured.

Table 8. Mix proportions of OPC-slag mortars for thermal activation method

Abbreviations

Abbreviation	Statement
OPC	ordinary Portland cement
ggbfs	ground granulated blast furnace slag
OM	ordinary Portland cement mortar
OSM	ordinary Portland cement-slag mortar
SM	slag mortar
OSM/i	OPC-slag mortar for i% replacement with slag
OSM/50-wc	control mix/ mortar
f_i	compressive strength at i days in MPa
i	relative strength or strength ratio
T	thermal activation method
Wc	water curing
Ac	curing in air under room temperature
SD	standard deviation
R^2	coefficient of determination
XRD	X-ray diffraction
SEM	scanning electron microscopy
EDX	energy dispersive X-ray analysis
XRF	X-ray fluorescence
ASTM	American Society for Testing and Materials
ACI	American Concrete Institute
BSI	British Standards Institute
SCA	Slag Concrete Association
MIA	Mortar Industry Association
CSA	Canadian Standards Association
GSD	grain size distribution
FM	fineness modulus
SSA	specific surface area
SAI	slag activity index
SP	super plasticizer
s/b	sand-binder ratio
w/c	water-cement ratio
w/b	water-binder ratio
DEF	delayed ettringite formation
EEF	early ettringite formation
SEF	secondary ettringite formation
ASR	alkali silica reaction
C-S-H	calcium silicate hydrates
CH	hydroxide calcium
LOI	loss on ignition
RH	relative humidity
AF _m ($C_3A.CaSO_4.12H_2O$)	aluminate-ferrite-monosubstituted or
AF _t ($C_3A.3CaSO_4.32H_2O$)	aluminate-ferrite-trisubstituted or ettringite

Abbreviation	Statement
CaCO_3	calcite
$\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$	ettringite
$\text{C}_3\text{A} \cdot \text{CaSO}_4 \cdot 13\text{H}_2\text{O}$	imonosulphate
C_2ASH_8	gehlenite hydrate
$\text{M}_6\text{Al}_2\text{CO}_3 (\text{OH})_{16} \cdot 4\text{H}_2\text{O}$	hydrotalcite
$\text{Ca}_2\text{SiO}_4 \cdot \text{H}_2\text{O}$	α - C_2SH

H-3 sets mix is a control mix, which the specimens were first made and demoulded 24 hours after casting, and then heated at 60 °C for 20 hours and finally, cured in three curing regimes, i.e. at room temperature, in water at 25 - 26 °C, and in water at 32 °C.

H-4 sets mix is a control mix, which the specimens were first made and demoulded 24 hours after casting, and then pre-cured without and with the use of heat at 60 °C for 14 hours and finally, each set of specimens was cured in both curing regimes, i.e. at room temperature and in water at 25 - 26 °C.

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