

Drying Shrinkage and Durability Studies on Alkali Activated Slag Concrete Using Different Activators

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ABSTRACT: The production of OPC is responsible for about 7% of the world's CO₂ emissions, lead to the search for more environmentally viable alternative to cement. One of those alternative material is alkali-activated slag (AAS) where ground granulated blast furnace slag is used not as a partial replacement to cement but as a sole binder in the production of concrete. The durability performance of alkali-activated slag concrete with sodium silicate, with and without addition of hydrated lime, sodium hydroxide with lime and sodium carbonate as activators are studied. All the activators are used at 8% Na₂O (by weight of slag) and 4% of hydrated lime by total weight of solid binder content if used as a retarder. Drying shrinkage was measured up to 40 days. The durability potential of AAS concrete was investigated by testing for oxygen permeability and porosity. Among different AAS concrete Na₂CO₃ activated concrete (SC8) showed less shrinkage, which is little bit higher than OPC concrete with same w/c (CM2) but lower than OPC/Slag blended mix (SLG60) where as water-glass activated slag showed highest drying shrinkage. Among AAS concrete, L.S.S activated slag concrete without addition of lime (SS8) showed lower porosity, which was even little bit lower than the OPC concrete having same w/c ratio (CM2) at the age of 28days. NaOH activated slag concrete (SS8L4) showed high porosity than other AAS concrete in both water and dry curing conditions, whereas Na₂CO₃ activated slag concrete showed comparable porosity with OPC concrete (CM2). Therefore, the use of SC as an activator for slag concrete is recommended, since it results in adequate strength, comparable or lower shrinkage to control mixes and almost equal or comparable porosity.

KEYWORDS: Alkali activated slag concrete, durability, permeability, drying shrinkage and porosity.

NOTATIONS: AAS = Alkali activated slag, C-S-H = Calcium Silicate Hydrate, DC = Dry Curing, GGBS = Ground granulated blast-furnace slag, L.S.S = Liquid sodium silicate, Ms = Modulus of silica, OPC = Ordinary Portland cement, SC = Sodium Carbonate, SH = Sodium Hydroxide, UPV = Ultrasonic pulse velocity, WC = Water Curing, DC = Dry Curing.

I. INTRODUCTION

Portland cement clinker is made from calcinations of limestone and siliceous material where de-carbonation occurs according to reaction:



For every ton of cement clinker produced, an approximately equal amount of carbon-dioxide is released into the atmosphere (Davidovits, 1991). The world cement industry contributes some 7% to the total man-made CO₂ emission (Malhotra, 1999). This leads to the search for more environmentally viable alternatives to Portland cement. One of these alternative materials is alkali-activated slag (AAS), in which ground granulated blast-furnace slag (GGBS) is used

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not as a partial replacement for cement but as a sole binder itself in the production of concrete. This will produce an environmentally friendly concrete. The use of slag cement has advantages due to its excellent cementitious properties over ordinary Portland cement (OPC), but the disadvantage of the low early strength has limited its use. Various studies had investigated ways to enhance the reactivity of the slag. One of the economic ways of activation is alkali activation. Slag has latent hydraulic properties. If GGBS is placed in water alone, it dissolves to a small extent but a protective film deficient in Ca^{2+} is quickly formed, which inhibits further reaction. The reaction continues if the P^H is kept sufficiently high. The pore solution of a Portland cement, which is essentially one of alkali hydroxides, is a suitable medium. The supply of K^+ and Na^+ ions is limited, but these ions are only partially taken up by the hydration products, and the presence of calcium hydroxide ensures that the supply of OH^- ions is maintained. The slag can be similarly activated by OH^- ions supplied in other ways such as addition of sodium hydroxide or silicate (Taylor, 1997).

II. MATERIALS AND MIXES

Ordinary Portland cement, manufactured by Blue Circle Industries (Hope Works), was used in this investigation. The chemical properties of the cement, as provided by the manufacturer are given in Table 3.1.

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	LOI	FCaO	Cl	EqNa ₂ O
Percent	17.1	4.7	2.2	6.3	0.9	2.9	0.2	0.54	7.7	1.8	0.02	0.7

Table 2.1- Chemical properties of Cement*

The chemical properties of slag used are given in Table 3.2 and is provided by the manufacturer.

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O
Percent	35.84	13	0.55	39.53	8.28	0.1	0.35	0.5

Table 2.2 - Chemical properties of GGBS

Liquid Sodium Silicate (Water-glass)

Liquid form of sodium silicate was used in the investigation. The sodium silicate solution was obtained from the Fisher Scientific. It has a molecular ratio SiO₂: Na₂O (M_S) = 3.21 with 29.2% of SiO₂ and 9.1% of Na₂O by weight.

Sodium Hydroxide Pellets

Sodium hydroxide pellets was obtained from the Fisher Scientific. It is 98.86 % pure. The pellets are used to make solution of required dosage in water.

Sodium Carbonate

Sodium carbonate powder was obtained from the VWR. it is almost 100% pure. The powder is used to make solution of required dosage in water.

Aggregates

Medium graded sand and 10-mm natural round uncrushed gravel was used in this investigation.

III. MIX PROPORTIONS AND NOTATIONS

The notation for the mixes is as follows:

CM1: PC control mix with w/c=0.55.

CM2: PC control mix with w/c=0.48.

SLG60: 60% GGBS + 40% OPC mixture with w/c=0.48.

SS8: Liquid sodium silicate -activated slag concrete mixture with modulus of silica (M_S) = 0.75 and Na₂O content of 8% by weight of slag with w/c=0.48.

SS8L4: Liquid sodium silicate -activated slag concrete mixture with modulus of silica (M_S) = 0.75 and Na₂O content of 8% by weight of slag and with the addition of 4% of hydrated lime by total weight of solid binder content with w/c=0.48.

SH8L4: Sodium hydroxide-activated slag concrete mixture with Na₂O content of 8% by weight of slag and with the addition of 4% of hydrated lime by total weight of solid binder content with w/c=0.48.

SC8: Sodium carbonate-activated slag concrete mixture with Na₂O content of 8% by weight of slag and with w/c=0.48.

IV. RESULTS AND DISCUSSION ON DRYING SHRINKAGE OF CONCRETE

The loss of free water contained in hardened concrete, does not result in any appreciable dimension change. It is the loss of water held in gel pores that causes decrease in the volume. This shrinkage is represented usually by the length change expressed in micro strains ($\times 10^{-6}$). This section presents the test carried out and the results obtained for the AAS concrete mixes in comparison with OPC and OPC/slag (control) mixes. The results are discussed and the conclusions are derived from the results.

A. Test procedure: Three 75 x 75 x 280 prisms were prepared for each of the AAS mixes and the control OPC and OPC/Slag mixes. The prisms were cured for 7 days in water. The prisms were then left to dry in the controlled temperature ($20 \pm 2^\circ\text{C}$) for three days. The shrinkage was measured using a length comparator in accordance with BS 812: Part 120: 1989.

The results for drying shrinkage for WC samples are shown in Fig 1. They are presented to show the behavior of the mixes in terms of drying shrinkage and the effect of early curing condition on the behavior of the AAS mixes.

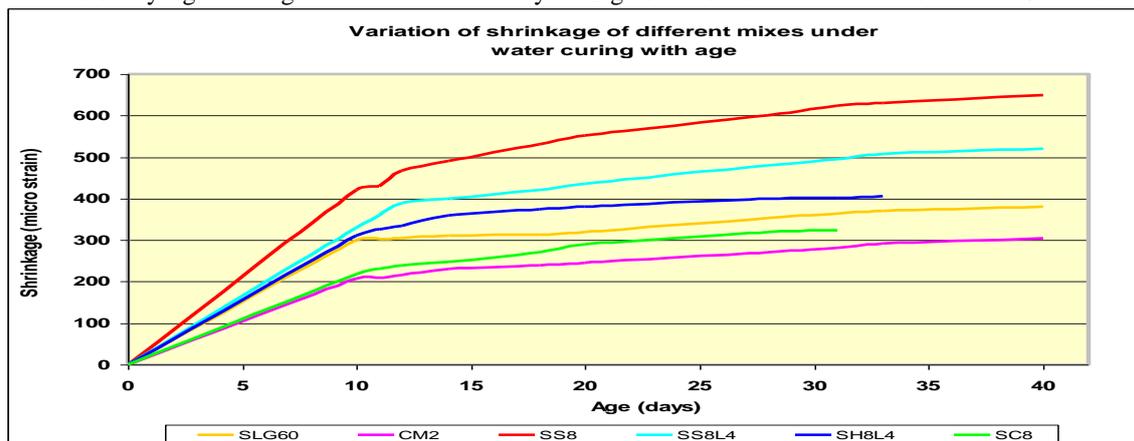


Fig 1- Drying shrinkage variation with age for different concrete mixes

The development of drying shrinkage with age for the samples cured in water for 7 days prior to drying is shown in Fig 1. The replacement of OPC by GGBS gave a higher shrinkage compared to the control mix having the same w/c ratio. SLG60 had shrinkage of 350 micro strain after 28 days compared to 270 micro strain for CM2 control mix is in agreement with previous results (Neville, 1995). The SS8 mix exhibited the highest drying shrinkage up to 601 micro strain at 28 days followed by SS8L4 mix with 480 micro strain, SH8L4 mix with 400 micro strain. The highest drying shrinkage in water-glass activated concrete is due to the presence of more silica gel which dries due to the loss of water (Saud, 2002; Shi et al., 2006). The decrease in shrinkage for SS8L4 when compared to SS8 mix is due to the presence of lime content, which decreases the cracking tendency and drying shrinkage (Saud, 2002; Shi et al., 2006). Among AAS concrete, SC8 exhibited lowest drying shrinkage (320 micro strain) but exhibited high shrinkage than OPC control mix CM2 (270 micro strain) and lower shrinkage than OPC/Slag blended mix (SLG60), which is in agreement with the previous results (Shi, 2006). The shrinkage difference in AAS concrete and OPC control mixes can be explained by the differences in pore structure. Pore size distribution can be characterized by r_s , which is defined as the radius of the pores where the meniscus forms; i.e., the pores whose radii are smaller than r_s are assumed to be filled with liquid water while pores larger than this are dry. As the drying progresses, the parameter r_s would decrease. The smaller the pore size is, the larger the capillary tensile forces set up at the meniscus, hence higher the resulting shrinkage. AAS concrete usually exhibits lower porosity than Portland cement concrete, but the former mainly contains

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pores with less radii than the later. The smaller the pore size is, the larger the capillary tensile forces set up at the meniscus, hence higher the resulting shrinkage for AAS concrete (Shi et al., 2006).

V. RESULTS AND DISCUSSION ON DURABILITY RELATED PROPERTIES OF CONCRETE

The durability of concrete can be defined as serviceability, ability to retain its quality and to resist deterioration throughout its life. Among the concrete properties that play a major role in the durability are the porosity of concrete and the permeability. In this chapter finally relationship between compressive strength and porosity of all the mixes under WC and DC is also included by making regression.

A. Apparent Porosity (water absorption method): Porosity is a measure of the proportion of a matrix occupied by pores and usually expressed in percentage of the total volume. The influence of the pores on the permeability depends whether these pores are interconnected or not. If the porosity of the matrix is high and the pores are connected the permeability is also high. On the other hand, a high porous material may also have a low permeability if the pores are discontinuous. The measure of the open porosity or apparent porosity through water absorption is one of the simplest methods. The porosity can be determined by immersing the sample in water for prolonged periods or by boiling the sample in water for several hours. It is probably the best method of measuring the porosity as it facilitates the real situation encountered by concrete structure. However, Neville (1995) reported that it is doubtful that these methods archive total or full saturation due to the presence of large number of very fine pores (as compared to rocks) and bottlenecks of some of the pores. In order to reach full saturation, it is necessary to empty the pores first from a air and water (by vacuuming) before allowing the water to penetrate them (Lynsdale, 1989). RILEM CPC-11.3 (1984) also recommended a test method based on evacuation of air from the samples then allowing the water to fill in the pores under vacuum to reach total saturation. The section presents the work carried out to measure the porosity using water absorption.



Fig 2- Vacuum saturation apparatus for measuring porosity

The porosity of concrete specimens investigated was determined using a vacuum saturation apparatus (RILEM CPC-11.3, 1984), which is shown in Fig 2. The test consists of weighing the sample in three different conditions: W₁= dry weight, W₂= saturated weight under water and W₃= saturated surface dry weight. The apparent porosity is calculated by the following formulae:

$$P = \frac{(W_3 - W_1) \cdot 100}{(W_3 - W_2)}$$

The formula above can be used if the sample is fully saturated; otherwise the porosity results are underestimated. To check the problems of partial saturation, the samples were broken after the test to check whether they were completed

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wet. The water saturation was achieved under vacuum for 24 hours. To calculate the dry weight, the samples were oven dried at $105 \pm 5^\circ \text{C}$ for 24 hours prior to testing, when constant weight was achieved.

B. Results and discussion on Porosity

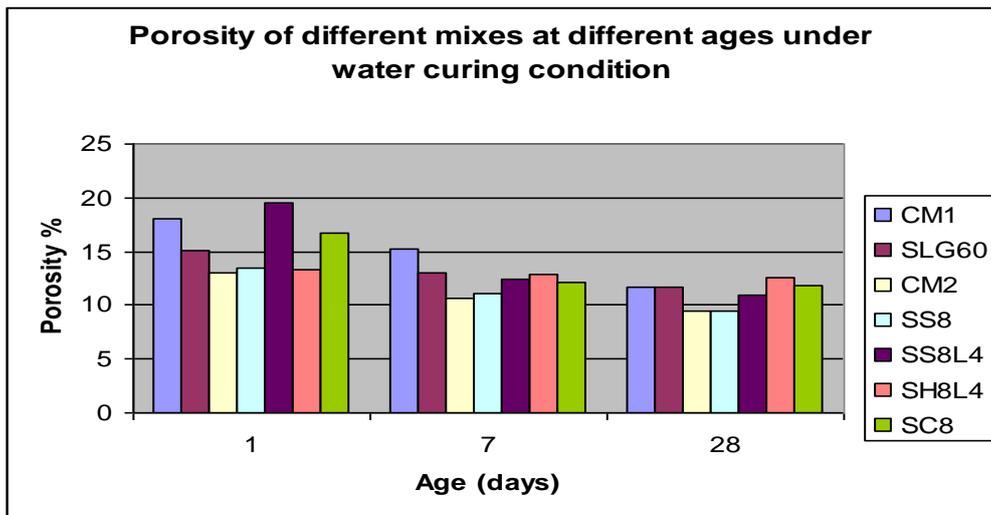


Fig 3- Porosity at different ages under water curing condition

Results of porosity test are presented in Fig 3 and Fig 4. From Fig 3 it is obvious that CM2 has the lowest value of porosity at all ages presented. Replacing 60% of the cement by GGBS (SLG60) resulted in an increase in the porosity of concrete compared to the OPC concrete mix (CM2) with the same w/c of 0.48. But when comparing the slag mix with the OPC control mix having w/c of 0.55 (CM1), the results show that SLG60 mix having lower porosity. These results are in agreement with results presented by *Lynsdale and Sit* (1992) where they indicated that 70% slag replacement of OPC resulted in an increase in porosity in the early age up to 28 days. The porosity of all concrete mixes reduced generally with age as is clearly shown in the results. For all the mixes under dry curing condition there is higher porosity value when compared to water cured samples, which obviously says that good curing results in less porosity.

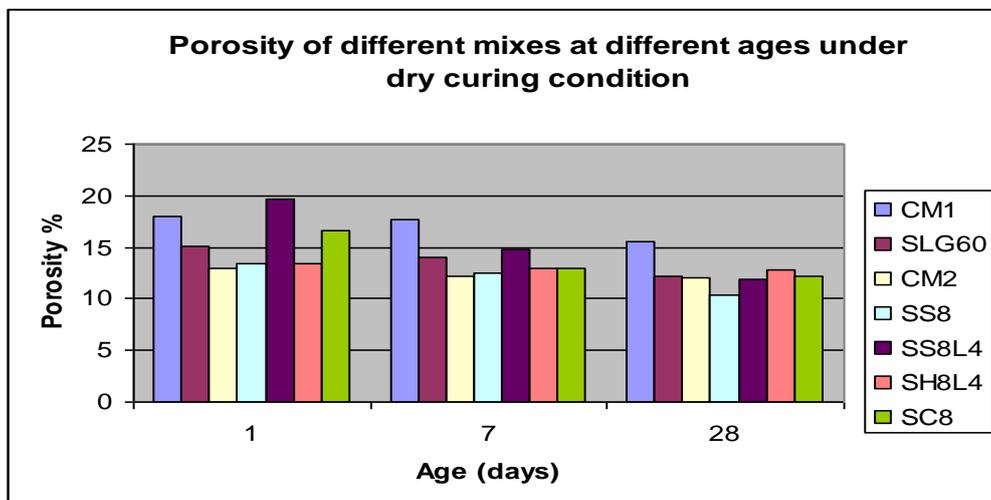


Fig 4- Porosity at different ages under dry curing condition

Among all the AAS concrete sodium carbonate activated concrete (SC8 mix) showed highest porosity at 1 day. This can be explained by the softness of the SC8 mix concrete at 1 day where the concrete is so soft even in demoulding some of the material removed, which obviously says that the hydration at 1 day is very low. The loss of material while demoulding is observed because of late hydration as the dosage of Na_2CO_3 might be high for the given chemical and physical composition of slag. This was reasoned by (Older et al. 1976) as when the dosage is low, sodium carbonate can be used as accelerator of cement hydration, but acts as a retarder in early age when the dosage is high. Looking at the results of dry cured AAS mixes it can be noted that the L.S.S activated slag concrete exhibits a lower porosity than all other mixes at the age of 28 day. At the age of 1, 7 and 28 day L.S.S activated concrete has almost same porosity as control mix (CM2) under water curing condition. At the age of 7 and 28 days the NaOH activated mix shows higher porosity around 13% in water cured condition.

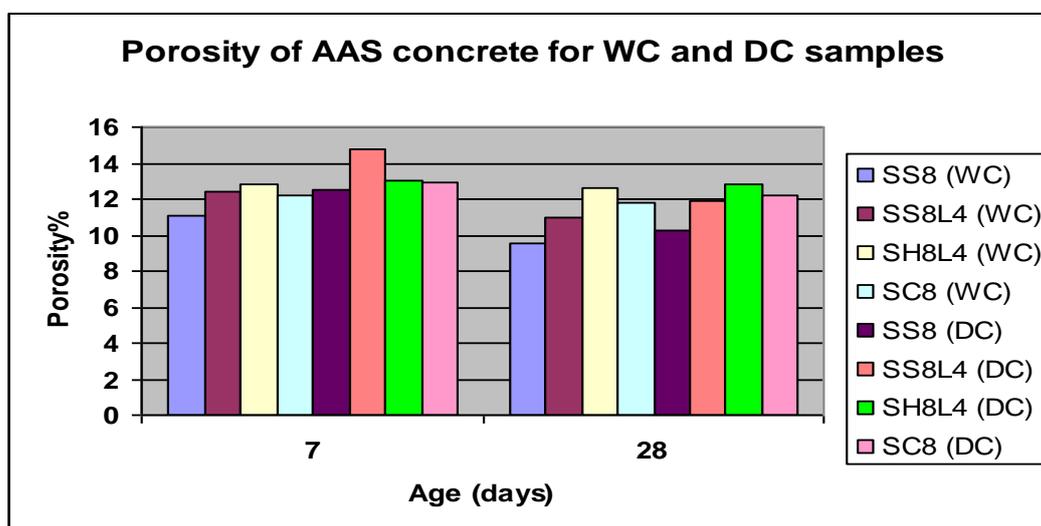


Fig 5- Effect of dry curing on porosity of AAS specimens

The Fig 5 shows the effect of dry curing on AAS mixes. For sodium hydroxide activated concrete the porosity change from 7 to 28 day is very less. This might be due to the early strength contribution and early hydration (before 7 day) of NaOH based concrete both in case of WC and DC samples.

C. Oxygen Permeability: Permeability of concrete can be defined as the ease with which external elements such as liquids and gases (fluids) penetrate concrete. The permeability is a very important property used to evaluate the durability of concrete and mortars. Water permeability is one of the methods used for evaluating the permeability of concretes and mortars. Nevertheless, the problems associated with the use of water permeameters include the difficulties to establish a steady condition and the duration of the tests, which take weeks rather than days (Basheer, 2001). One of the simple ways to measure the permeability of pastes, mortars or concrete employs the use of gases, such as oxygen (Basheer 2001; Cabrera and Lynsdale 1988; Khan and Lynsdale 2002). Oxygen permeability has the advantage of examining the permeability of samples collected “in situ”. The samples are usually smaller and therefore, the equilibrium condition is rapidly achieved, reducing the time of testing (Cabrera and Lynsdale 1988). The gas permeability of cementitious systems is greatly affected by the progress of hydration, curing condition, w/c ratio and moisture content (Neville 1995). The permeability increased with an increase of the w/c ratio and reduced with curing age (Khan and Lynsdale 2002).

D. Preparation of Specimens: In this investigation, the specimens that were cast were 100 mm diameter x 50 mm height. They were cured under water curing and dry curing and tested at ages 1, 7 and 28 days. Oxygen permeability was performed on two samples of each concrete specimen at 1, 7 and 28 days. Prior to testing samples were taken out of the curing environment at the required testing age. The samples were then dried in an oven at $105 \pm 5^{\circ} \text{C}$ for minimum 24 hours, until constant weight was reached.

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E. Testing procedure: The parameter used for the test was designed by Cabrera and Lynsdale (1988) and it is schematically shown in Fig 6. The sample is held tightly by a rubber O-ring, while kept inside a stainless steel capped cell. Oxygen is forced to flow vertically through the sample and a bubble flow meter records the flow rate (V) after the steady state is achieved (approximately 20 minutes after the beginning of the test).



Fig 6- Oxygen permeability test set-up

The following equation, derived from Darcy's law of flow, is used to calculate the intrinsic permeability of sample:

$$k = \frac{2P_2 V l \times 2.02 \times 10^{-16}}{A(P_1^2 - P_2^2)}$$

Where:

k is the intrinsic permeability (m²)

l is the length of the specimen (m)

A is the cross-sectional area of specimen (m²)

V is the flow rate (cm³/s)

P₁ is the absolute applied pressure (bar)

P₂ is the pressure at which the flow rate is measured, usually 1 bar.

The coefficient 2.02×10^{-16} transforms the units of the viscosity of oxygen and provides the final result in m². The condition of the sample, i.e., its moisture content, affects the oxygen permeability considerably, so that the results are specific to the drying regime used prior to testing. Another parameter that affects the oxygen flow is the inlet pressure employed (P₂) and the intrinsic permeability varies for the different pressures. Here for this permeability measurement throughout the investigation the inlet pressure used (P₂) was always 1 bar.

F. Results and discussion on Permeability of different alkali activated concretes: The results of Oxygen permeability of AAS mixes and control mixes under water curing investigated, having a w/c=0.48, are presented in Fig 7 and Fig 8.

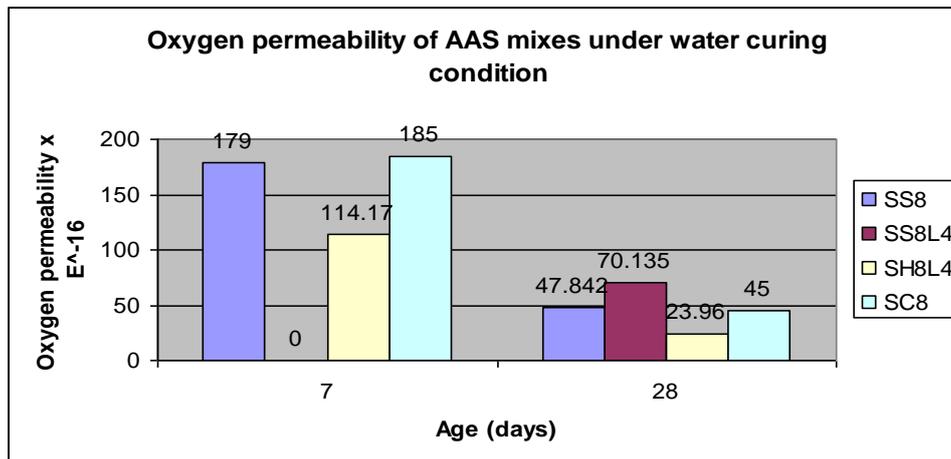


Fig 7- Oxygen permeability of control mixes at different ages investigated

The effect of curing on oxygen permeability for the OPC and OPC/Slag mixes are shown in Fig 7 and Fig 8, where it is clear that oxygen permeability under dry curing was over 10 times higher compared to water curing. Fig 8 shows the results of control mixes (CM2 and SLG60) under water and dry curing conditions. The CM2 mix had the lowest oxygen permeability results starting from $1 \times 10^{-16} \text{ m}^2$ at 7 days decreasing to 0.8×10^{-16} at 28 days, under water curing condition whereas there is no significant change in Oxygen permeability with age (from 7 to 28 days) in dry curing condition as investigated. So this shows that proper curing is essential to get better hydration and less permeability. The OPC/Slag mix (SLG60) had oxygen permeability higher than CM2 with a value of around $2 \times 10^{-16} \text{ m}^2$ at 7 days decreasing with age to around 0.9×10^{-16} at 28 days in case of water curing condition. It is noticed that the results of oxygen permeability for the AAS mixes are much higher in comparison with OPC and OPC/Slag mixes. From the Fig 7 it is visible that SH8L4 mix shows less permeability among AAS mixes followed by SS8 and SC8 mixes, which all are having more permeability until 28 days. All the AAS mixes (SS8, SS8L4, SH8L4 and SC8) under dry curing condition and at the age of 1 day are so high that measuring the oxygen permeability is not possible. The rate of flow was very high and could not be measured. From the Fig 7 it can be noticed that the SS8 mix shows a value of $179 \times 10^{-16} \text{ m}^2$ at 7 days and then decreasing drastically to $47 \times 10^{-16} \text{ m}^2$ at 28 days. Even sodium carbonate activated slag mix (SC8) shows more or less same value of oxygen permeability within the range of age investigated (until 28 days). For SS8L4 mix at 7 day the oxygen permeability is not measurable, whereas at 28 day it is around $70 \times 10^{-16} \text{ m}^2$. For SH8L4 mix at 7 day the oxygen permeability is $114 \times 10^{-16} \text{ m}^2$, decreasing drastically to $23 \times 10^{-16} \text{ m}^2$, which is the lowest permeability among AAS mixes within the range of age investigated. For all the AAS concrete mixes done in this work the gas permeability is not measurable in case of DC samples. The high permeability results for the AAS mixes bring into question the reliability of the test especially with relation to the drying of the specimens prior to testing. This technique received a lot of criticism in the literature related to normal concrete. The preconditioning treatment carried out in the test has some disadvantages although drying is required to remove moisture, which can influence the result. Usually the temperature of drying affects the permeability result with higher values in the case of higher temperature (Lynsdale, 1989). The 105°C was widely used with OPC concrete on the thought that it is much representative as all water was assumed to be removed. Mills (1985) reports that the differences in permeability due to the type of treatment and is ascribed to the transformation of the fine-pore structure into a coarser one due to the tensile stresses. Sanjuan and Munoz-Martialay (1996) stated that the variation in results of air permeability due to preconditioning temperatures (from $20\text{-}80^\circ\text{C}$) was low in the case of w/c ratios between 0.42 and 0.47 implying that there is no effect on the results for such concrete due to the variation in preconditioning temperature. A RILEM committee (1999) issued a paper on the gas permeability test recommending setting the preconditioning temperature to 50°C provided that the preconditioning continue until the calculated loss of water (variation in mass) is attained within a 5 percent level of accuracy. Although the preconditioning at 105°C was acceptable for control mixes, the high results of oxygen permeability for AAS concrete were probably due to the effect of drying. This is in agreement with Saud (2002) who

suggested that extreme drying could cause micro cracking and damage in the C-S-H gel. Since the main hydration product for AAS concrete is C-S-H and this justification is relevant.

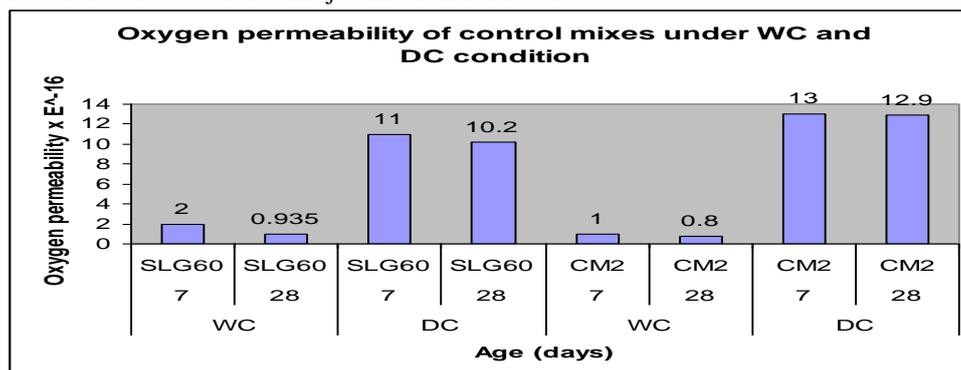


Fig 8 - Oxygen permeability of control mixes at different ages investigated

VI. CONCLUSION

Among AAS concrete, L.S.S activated slag concrete without addition of lime (SS8) shows lower porosity, which is even little bit lower than the OPC concrete having same w/c ratio (CM2) at the age of 28 days. NaOH activated slag concrete (SS8L4) shows high porosity than other AAS concrete in both water and dry curing conditions, whereas Na₂CO₃ activated slag concrete shows comparable porosity with OPC concrete (CM2). OPC/Slag mix with 60% replacement has oxygen permeability higher than that OPC mix having the same w/c ratio in case of water curing condition whereas in dry curing it is not so until the age of 28 days investigated. Pre-drying the specimens at 105°C resulted in an increase in oxygen permeability of AAS concrete to very much higher levels than those of OPC and OPC/Slag mixes. Dry curing increases the oxygen permeability to such a level that in case of all AAS concrete the rate of flow is not measurable. Among different AAS concrete Na₂CO₃ activated concrete (SC8 mix) showed less shrinkage, which is little bit higher than OPC concrete with same w/c (CM2) but lower than OPC/Slag blended mix (SLG60). The AAS concrete porosity test results are so high at early ages than the control mix due to the drying effect of preconditioning the specimens at 105°C. Dry curing increases the porosity and permeability of all the concrete mixes. The use of Na₂CO₃ as an alkaline activator for the slag used in this study was recommended since the Na₂CO₃ activated slag mix has comparable drying shrinkage and porosity to OPC concrete

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