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Research Paper

Effect of different curing methods on the compressive strength development of pulverized copper slag concrete

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ABSTRACT

The results of laboratory studies conducted to evaluate the effects of different curing conditions on the compressive strength development of concrete made with pulverized copper slag as partial replacement for Portland cement (PC) is presented. Concrete cube specimens were made with basic material proportions ranging from 0, 2.5, 5, 10 and 15% by weight of PC under normal laboratory conditions and cured in three different conditions, namely; water, solar chamber and ambient air up 90 days. Test performed included X-ray Fluorescence (XRF) and sieve analysis respectively for the chemical oxide composition and fineness, for both the pulverized copper slag and Portland cement. Specimens were tested for compressive strength up to 90 days of curing. The experimental results indicate a significant drop in the compressive strength as the copper slag content increases for all curing methods. Moreover, for the control samples, the percentage decrease in the compressive strength for the 3-day curing for water cured sample, compared to the solar chamber and ambient air were respectively 31 and 28%.However, beyond 28 up to 90 days of curing, the water cured samples yielded a higher compressive strength, followed by the solar chamber and ambient air. The percentage increase in the compressive strength up to 90 days of curing for water cured specimen, compared to the solar chamber and ambient air were respectively 8.5 and 12%. This trend was similar for all percentage replacement of cement with the pulverized copper slag.

1 Introduction

Curing is the process used for promoting the hydration of cement. Curing consists of the control of temperature and moisture movement from and into placed concrete; with the aim of keeping the concrete saturated or as nearly saturated as possible, until the originally water-filled space in the fresh cement paste has been filled to the desired extent by the products of cement hydration [1]. Proper curing reduces the rate of moisture loss and provides a continuous source of moisture required for hydration. Thereby reduces the porosity and provides a fine pore size distribution in concrete [2].

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Curing may be applied in a number of ways and the most appropriate means of curing may be dictated by the site or construction method [2]. Curing is designed primarily to keep the concrete moist, by preventing the loss of moisture from the concrete during the period in which it is gaining strength.

Experimental study shows that concrete continuously cured in air have lower compressive strength compared to water cured concrete at all required age of testing [3, 4]. Additionally, compressive strength loss between 10-20% of concrete cubes cured in air was observed compared to cubes that are wet cured [5]. At 28 days, the compressive strength of concrete cube specimens continuously wet cured was 40% higher than those air cured and at 90 days, specimens continuously moist cured had compressive strength 20% higher than those of air cured cubes [6]. The effect of curing period and curing delay on the properties of concrete in hot weather was also studied by Al-Ani *et al.* [7]. The authors reported that, wet burlap curing method was an effective technique for maintaining the moisture in concrete. However, they recommended a minimum of 3 days of wet burlap curing for rich mixes, whereas 7 days for lean mixes. The 90-day compressive strength of PC and fly ash cement concrete was reported to be 67% and 50% respectively for continuously fog cured concrete specimens. However, 7 days moist curing improved these values to 95% and 82% of the fully cured concrete [8]. Another author suggested a minimum period of curing should be optimized in terms of several properties such as strength, permeability and the movement of aggressive gases and/or liquids from the environment [9].Concretes prepared with mineral admixtures are more sensitive to water curing than PC concretes, the minimum period of curing required for PC, fly ash and silica fume concrete mixtures were respectively 3, 3.75 and 6.5 days [9]. The durability of concrete is affected by a number of factors including its permeability and absorptivity. These factors are related to the porosity of concrete and whether the pores and capillaries are discrete or interconnected [10]. Whilst the number and size of the pores and capillaries in cement paste are related directly to its water-cement ratio, they are also related indirectly to the extent of water curing. Over time, water curing causes hydration products to fill either partially or completely, the pores and capillaries present help to reduce the porosity of the paste [10].

Copper slag is produced either by hydro-metallurgical or pyro-metallurgical production of copper from copper ores and contains materials like iron, alumina, calcium oxide, silica [11]. Until recently, metallurgical slags were traditionally considered a waste product. Current management options of slags are recycling and recovering of metal, production of value added products and the disposal in slag dumps, stockpiles or tailing dams. Over the years, rigorous environmental impacts have been associated with copper tailings dam failure. Approximately 2 million m^3 of mud containing heavy metals were spread over 4286 ha of land and surface water during the 1998 Aznalcollar tailings pond failure in Spain [12]. The year 2000 tailings spillage at Nchanga Copper Processing Plant in Zambia released high concentrations of heavy metals into the nearby surface water, thereby contaminating the local source of water supply [13]. Researchers have developed waste management strategies for specific needs which are sustainable. The integrated approach of working on safe disposal and utilization of industrial by-products can lead to advantageous effects on the ecology and environment and also as a tool for sustainable development.

2 Experimental Procedure

2.1 Materials

Type 1 Portland cement of strength class 52.5MPa at 28-days (CEM I, 52.5N) procured from local cement manufacturing company called PPC (Ltd), was used for this study.PPC (Ltd) type 1 Portland cement is among the most widely used cements in the construction industry in South Africa, made from high quality raw materials (limestones and clays or shales). The properties of the cement conforms to the requirement of SANS 50197-1 [14] and both the chemical oxide composition and glass content of the copper slag examined using the X-ray fluorescence (XRF) techniques and X-ray diffraction (XRD) pattern respectively are discussed in chapter three.

Granite aggregates are crushed hard rock of granular structure, being the most common on Earth. Granite rock comes from magma that erupted on the ground surface and then hardened [15]. Good properties of granite stones make it the most popular building material and in terms of its technical characteristics. Coarse granite aggregates between 18-20mm and fine granite aggregates between 75µm – 4.75mm particle sizes procured from Afrisam South Africa were used for this experimental work. The sampling of the aggregates was done in accordance with BS EN 932-1: 1997 and all the aggregates used for this experiment were air dried under standard laboratory conditions [16].

The copper slag used for this research work was brought from Katanga Province, Democratic Republic of Congo. Katanga is a province with several mining companies producing copper and cobalt. These companies include Electric Foundry of Panda (FEP), Electric Foundry of Kolwezi (FELCO) and STL Company all located in Lubumbashi. The physical appearance of the Copper slag is black, glassy and granular in nature with similar particle size range like sand, mostly between 4.75 and 0.075 mm in size as shown in Fig. 1.

(a) *(b)*

Fig. 1 – (a) Granulated copper slag (b) Pulverized copper slag

2.2 Chemical Composition

The mineralogical and glass content of the copper slag sample were obtained using the Rietveld X-Ray Diffraction (XRD) method. The elemental oxide composition of the copper slag was obtained using the X-Ray fluorescence spectrometer to determine all the major oxides present in the sample. The XRF of both the pulverized copper slag and cement were analyzed in South Africa by Lafarge Chemical laboratory (Pty), whiles the XRD was performed in Germany by Heidelberg Technology Centre.

2.3 Fineness test using sieves

25 g of the pulverize copper slag was carefully placed in a 45 µm sieve to avoid losses, the sieve was fitted with the lid and agitate by swirling, until no more fine materials pass through in accordance with EN 196-6: 2005[17]. The residue was removed, weighed, and expressed as mass percentage of the initial quantity first placed in the sieve to the nearest 0.1%. The experiment was repeated for 90 µm and 212 µm sieves for both Portland cement and the pulverized copper slag sample.

2.4 Mix proportions

A series of concrete mixtures were cast in cubic moulds of nominal size 100 mm with different proportions of pulverized copper slag ranging from 0, 2.5, 5, 10 and 15% respectively replacing Portland cement. Activation of the pozzolanic reaction of the pulverized copper slag was done using 1.5% hydrated lime ($Ca(OH)_2$) by weight of PC. The materials were weighed using a digital balance and mixed in a rotating pan mixer. The materials constituents are shown in Table 1.

Table 1 –Concrete mix proportions

Copper slag replacement (%)	0	2.5	5	10	15
Portland Cement (kg/m^3)	352	343	334	317	299
Fine aggregate (kg/m^3)	758	758	758	758	758
Coarse aggregate (kg/m^3)	995	995	995	995	995
Copper slag (kg/m^3)	0.0	8.8	17.6	35.2	52.8
$Ca(OH)_2$(kg)	5.3	5.3	5.3	5.3	5.3
Water (kg/m^3)	170	170	170	170	170
Water to binder ratio	0.5	0.5	0.5	0.5	0.5

2.5 Curing of concrete cubes

For air curing, the demoulded concrete cubes were cured at normal room conditions. The room environment records an average temperature of 24±1ºC and 21±1% average relative humidity. Concrete cubes after demoulding were placed in stacks and ambient air was allowed to freely flow to cure. The temperature and relative humidity were digitally recorded, until the required age of curing for compressive strength determination.

For water curing, the series of concrete cubes were demoulded after 24 hour curing in standard laboratory conditions. The demoulded specimens were moist-cured in a water tank, at an average temperature of 20±1 ºC, 100% relative humidity and tested at the required age of curing. The water curing encompasses the control of temperature since this affects the rate at which cement hydrates. The near constant water temperature was measured using a thermometer up to 90 days of curing the concrete cubes specimen in a water tank at an average temperature of 20±1ºC.

The solar chamber made of ordinary rectangular plastic container of dimensions; length 1300 mm, breadth 600 mm and height 700 mm; with a glass lid of average thickness 0.9 mm was used for curing. The chamber was painted white in color, to reflect the rays of the sun and help keep the concrete specimens at a uniform temperature during extreme hot weather conditions. Granite stones of average size 19 mm were placed at the bottom of the chamber to a depth of about 200±20 mm and filled with ordinary portable water 100 mm underneath to keep a higher constant relative humidity inside the chamber. The chamber was tightly sealed after placing the demoulded cubes and a digital probe was attached to monitor the interior temperature and relative humidity. The internal conditions of the chamber was not regulated, however, recorded an average 90-days temperature of 35±1ºC and 90±1% relative humidity depending on the climatic conditions.

2.6 Compressive strength testing

Concrete specimen were quickly removed and tested at the required curing age to prevent excess loss of heat during the removal process The axis of the specimen was aligned with the centre of thrust of the spherically seated platen and the platen adjusted gently by hand to achieve uniform contact prior to applying the load as par SANS 5863:2006 [18]. To determine the unconfined compressive strength, ninety concrete cubes of size 100 mm × 100 mm ×100 mm were cast for each mixture. For all three curing method used for this experiment, the specimens were kept for 3, 7, 14, 21, 28, 60 and 90 days before the compressive strength test were conducted. An average of three samples were tested at constant loading rate of 1.0kN/s at each required curing age in accordance with SANS 5863:2006 [18] using the Tinius Olsen compressive machine.

3 Results and discussion

3.1 Chemical analysis

The chemical analysis of Portland cement, procured from PPC (Ltd) and copper slag are presented in Table 2. The calcium oxide (CaO) contributes to nearly 63% of the chemical composition of the cement, whereas copper slag has a very low lime content of approximately 12%. This indicates that copper slag is not chemically a very reactive material to be used as a cementitious material since sufficient quantity of lime must be available in order to reach the required rate of hydration and to achieve the required early-age strength. On the other hand, copper slag has high concentrations of SiO_2 and Fe_2O_3 compared with PC. The SiO_2react with calcium hydroxide formed from the hydration of calcium silicate. The result of the hydraulic activity index for copper slag is approximately 0.5, which is less than 1, the recommended requirement to be used as constituent for cement [19]. The glass content of the copper slag is approximately 99.3%, similar to Ground Granulated Blast Furnace Slag (GGBS) glass content between 85 and 90% [20]. The glassy nature of a slag is responsible for its cementitious properties, with a linear relation to the late compressive strength development of concrete [21].

Table 2 - Chemical composition and glass content of PC and CS

Components	Portland cement (PC) %	Copper slag (CS) %
SiO_2	19.85	40.03
Al_2O_3	4.78	7.24
Fe_2O_3	2.38	24.40
CaO	63.06	12.53
MgO	2.32	6.62
K_2O	0.94	1.01
Na_2O	0.22	0.68
TiO_2	0.25	0.64
Mn_2O_3	0.05	0.11
P_2O_5	0.26	0.19
SrO	0.3	-
ZnO	-	-
SO_3	2.48	0.48
Loss on Ignition (LOI)	2.83	2.38
$(CaO + MgO)/SiO_2$	3.29	0.48*
Glass Content	-	99.3

* - hydraulic activity index in accordance with SANS 55167-1:2011

3.2 Fineness test

The residue of the pulverised copper slag and cement passing through the sieves were removed, weighed and expressed as mass percentage of the quantity first placed into the sieve to the nearest 0.1%. Three sieves of sizes 45μm, 90μm and 212μm were used. The results of the sieve analysis are presented below in Table 3.

Table 3 – Fineness of ordinary Portland cement and Copper slag

	Sieve sizes		
Materials	45μm	90μm	212μm
	Residue %		
Copper slag	1.9	0.2	0.1
Portland Cement	8.6	1.0	0.2

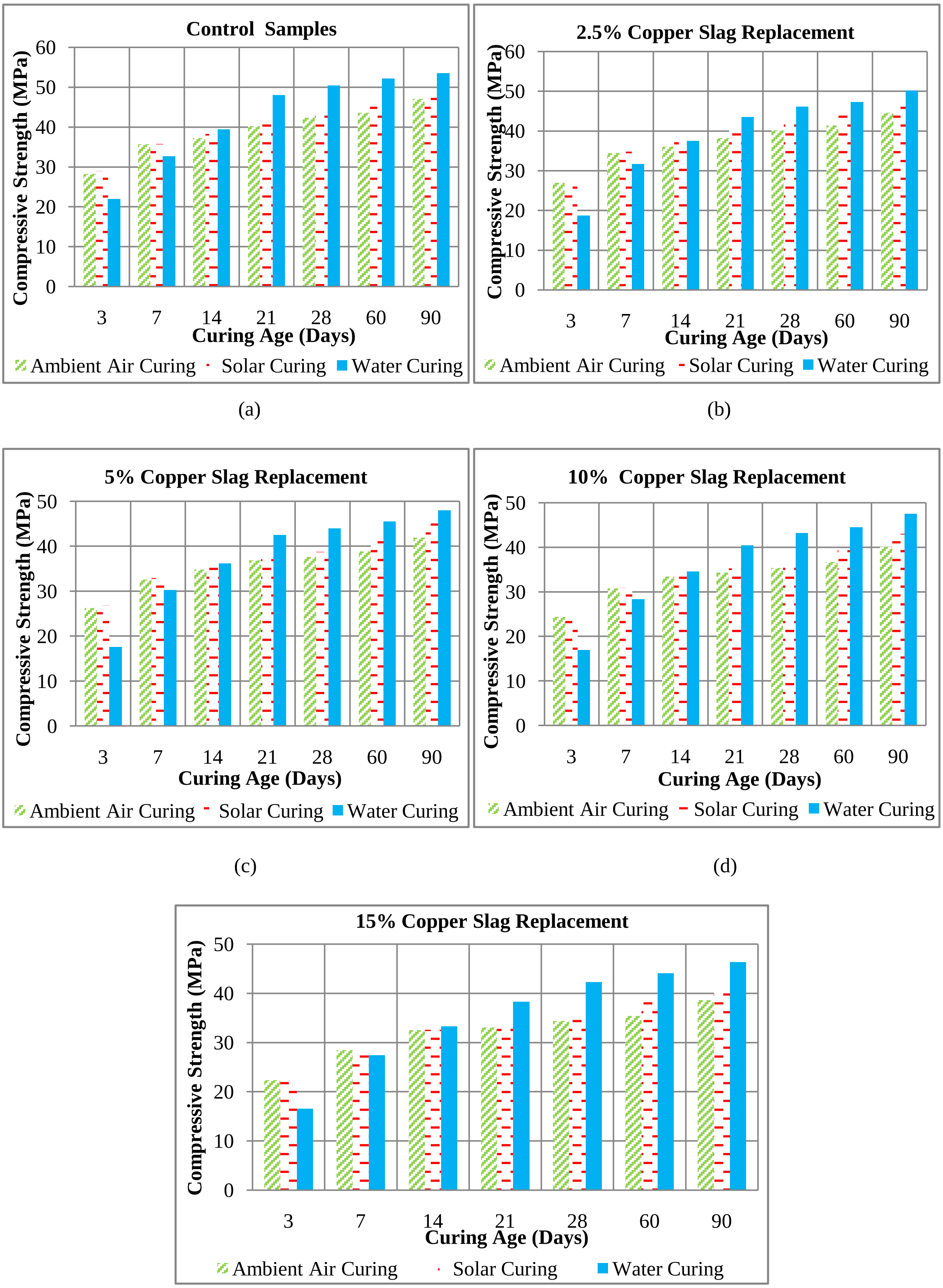
The percentage residue left on the 45μm sieve for both the copper slag and PC were 1.9 and 8.6 respectively. The trend was similar for both 90μm and 212 μm sieves. The results show that copper slag is slightly finer than Portland cement; which subsequently may lead to an increase in the surface area for pozzolanic reactivity to enhance strength development. The average size of fly ash lies between 7 and 12 μm, the above results lies between the aforementioned ranges [22].

3.3 Compressive test results

The measured compressive strength for concrete specimens up to 15% copper slag replacement with Portland cement for all the three curing methods, namely, water, solar and ambient air up to 90 days of curing is shown in Table 4. It was observed that, there is a significant decrease in the compressive strength as the copper slag content increases. The reduction trend was similar for all three curing methods namely, water, solar and ambient air up to 90 days of curing. The overall decrease in the ultimate compressive strength for copper slag concrete compared to control samples could be due the high glass content of the copper slag (99.3%). As reported in a similar study, slag concrete with high glass content in excess of 95% significantly reduces in compressive strength [23]. A low percentage of crystallization between 3–5% in mass of slag is found to be beneficial to the compressive strength development of slag concrete [24].

Table 4–Compressive strength results of copper slag concrete

	Copper Slag Replacement (%)														
	0%			**2.5%**			**5%**			**10%**			**15%**		
Curing Days	**Air**	**Solar**	**Water**	**Air**	**Solar**	**Water**	**Air**	**Solar**	**Water**	**Air**	**Solar**	**Water**	**Air**	**Solar**	**Water**
3	28.13	28.90	22.00	26.90	27.53	18.80	26.23	26.87	17.60	24.37	24.70	16.90	22.30	22.67	16.50
7	35.57	35.70	32.70	34.37	34.77	31.70	32.53	32.93	30.30	30.73	30.93	28.30	28.37	28.59	27.40
14	37.10	38.23	39.50	36.03	37.20	37.50	34.80	35.23	36.20	33.43	33.87	34.50	32.50	32.53	33.30
21	40.10	41.70	48.00	38.10	40.20	43.50	36.87	37.09	42.50	34.33	35.20	40.40	33.03	34.00	38.29
28	42.22	43.12	50.50	40.08	41.83	46.20	37.60	38.75	44.00	35.35	36.25	43.20	34.35	35.53	42.30
60	43.48	45.75	52.20	41.33	44.03	47.30	38.85	41.80	45.50	36.68	39.72	44.50	35.38	38.25	44.10
90	46.98	48.95	53.50	44.57	47.04	50.20	41.91	45.39	48.00	40.08	43.01	47.50	38.60	41.15	46.30



(a) (b)

(c) (d)

(e)

Fig. 2 Effect of curing type on the compressive strength of concrete at (a) 0% (b) 2.5% (c) 5% (d) 10% and (e) 15% copper slag replacement

3.4 Effect of temperature and humidity on the early age compressive strength

The measured compressive strength values for all three different curing methods for the copper slag concrete with a constant water-to-cement ratio are presented in *Fig 2(a)-(e).* It is observed from *Fig 2(a)* i.e. for only the control cube samples, the water cured samples yielded a lower 3-day compressive strength of 22.0MPa, followed by ambient air, 28.13MPa and solar chamber curing, 28.90MPa. This trend observed, was similar for all percentage replacement of copper slag with cement as shown in *Fig 2(a)-(e).* It is believed that, an increased in the curing temperature of average values 35°C and 25°C respectively for solar and ambient air environments, compared to water at 20°C as shown in *Fig 3*, increased the rate and extent of reaction through an increase in the heat of reaction; consequently increasing the early compressive strength development of the concrete cubes in both solar and ambient air curing methods compared to those cured in water.

Cement hydration does not improve when cured at relative humidity below 80% [25]. The solar chamber creates an average humidity of 90±1% compared to ambient air of 22±1% as shown in *Fig 4*, henceforth the overall strength development of specimens in the solar chamber performed better than that air cured.

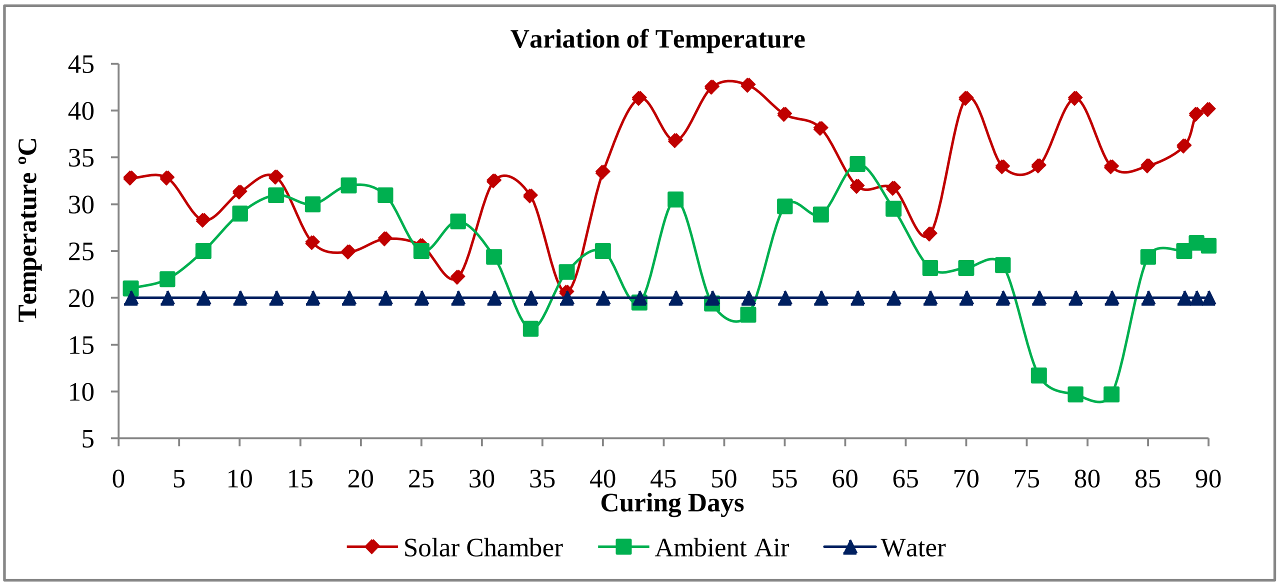


Fig. 3 – Variation of Temperature for all Curing Methods up to 90 Days of Curing

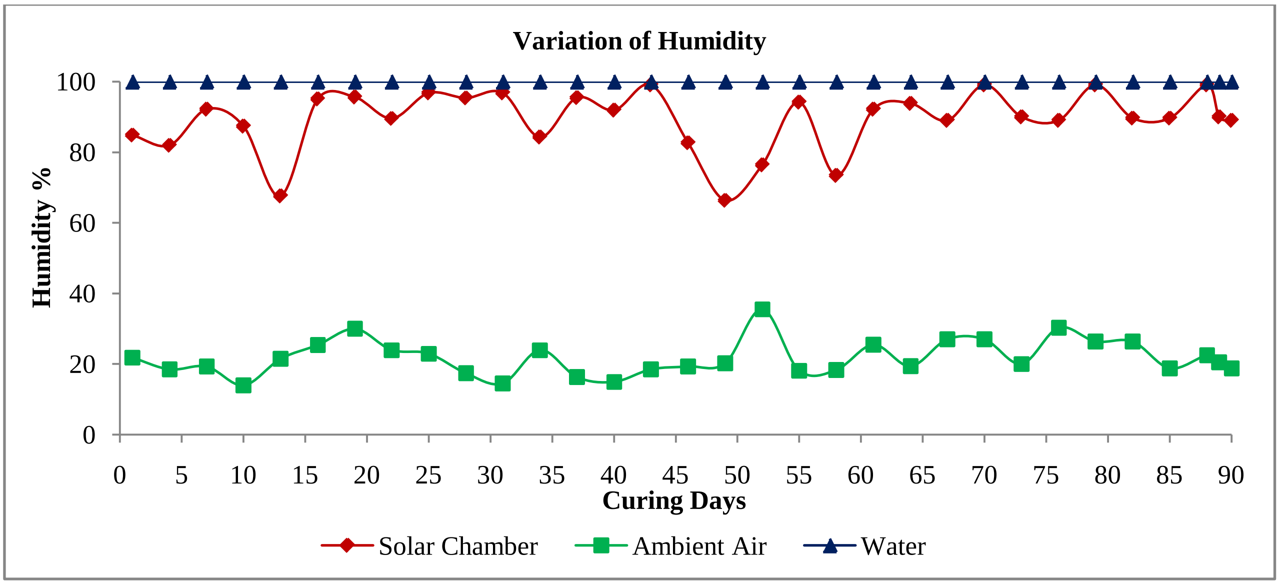


Fig. 4 –Variation of Relative humidity for all Curing Methods up to 90 Days of Curing

3.5 Effect of temperature and humidity on the late age compressive strength

There was a significant reduction in the late strength development of all concrete specimen cured in both ambient air and solar chamber, compared to water cured specimen as shown in *Fig 2(a)-(e).* The late strength development up to 90 days of curing was high for water cured specimen followed by solar and ambient air.

For the control samples up to 90 days of curing, the percentage increase in the compressive strength for water cured specimen, compared to the solar chamber and ambient air were respectively 8.5 and 12%. This trend was similar to all copper slag concrete from 2.5 to 15% replacement level. The higher late strength development for concrete samples cured in water could be attributed to water providing adequate moisture for further hydration as compared to solar chamber and ambient air. A similar research showed that at 28 days, compressive strength of cube specimens continuously wet cured were 40% higher than those cured in ambient air and attributed the higher compressive strength of the wet cured cube specimens to the improved gel/space ratio in concrete [6]. The effect of air curing causes the loss of compressive strength of high strength concrete due to self-desiccation of the concrete, therefore adequate wet curing to hardened concrete should be provided [26].

The observed reduction in the late compressive strength of the concrete samples for both solar chamber and ambient air curing methods, compared to water cured as shown in *Fig 2(a)-(e)* could also be attributed to the breakdown of the granular structure of the concrete mixture. The solar chamber creates a condition of high temperature in the surrounding concrete specimen, henceforth during the early stage of the concrete hardening process results in the formation of larger pores, which consequently increases the cumulative pore volume in the concrete interior microstructure, resulting in a negative effect on the late mechanical strength formation at later stage. This hypothesis is supported by a similar study on the effect of curing temperature on the development of hard structure of metakaolin based geopolymer [27]. The author demonstrated that, higher curing temperatures and longer curing time increases the early age of the compressive and flexural strengths of concrete as opposed to the late mechanical strength [27].

The variation of relative humidity for all curing method up to 90 days is also shown in *Fig 4.*The results demonstrated in *Fig 2(a)-(e)* signify that, a higher relative humidity leads to higher later compressive strength development for water cured samples followed by solar chamber and ambient air, presumably because moisture necessary for hydration process is supplied at a higher relative humidity consequently leading to a higher late strength for all concrete specimens.

For compressive strength development of concrete samples in relation to vapour pressure, the degree of hydration of cement is dependent on the vapour pressure within the concrete pore space [28]. Powers [28] explained that, the degree of hydration is negligible at vapour pressure below 30% of the saturation pressure and reduces when the vapour pressure is below 80% of the saturation pressure. This postulation explains the importance of saturating concrete specimen by continuously wetting with water to saturate the pores, thereby promoting cement hydration. As presented in this report, concrete cured in both ambient and solar chamber environment experienced a drop in vapour pressure of the concrete pore matrix below the saturation pressure, which consequently led to the compressive strength reduction compare to water cured specimen at later stage.

4 Conclusions

Based on the experimental outcome, observations and trends determined from the results of the above experimentations, the following conclusions were made:

- The glass content of the copper slag was approximately 99.3%, comparable to Ground Granulated Blast Furnace Slag (GGBS), with glass content between 85 and 90%. The high glassy content of the copper slag could be credited to the reduction of the overall compressive strength of the pulverized copper slag concrete compared to the control samples.

- The screen residue of the pulverised copper slag and cement passing through three sieves of sizes 45, 90 and 212 µm results shows that, the copper slag was slightly finer than Portland cement; which subsequently lead to an increase in the surface area for pozzolanic reactivity to enhance strength development.

- For the water cured samples, the compressive strength reduction of the copper slag concrete samples were more pronounced at early curing ages. The early strength reduction could be due to the slower rate of hydration, which is a typical characteristic of metallurgical slag influences on concrete. However, beyond 28 to 90 days, the strength gain of the copper slag concrete was slightly higher than for control samples. This observation could be attributed to the increased in pozzolanic and hydraulic activities.

- The water cured samples yielded a lower 3-day compressive strength followed by ambient air, and solar chamber curing. The percentage decrease in compressive strength for the 3-day curing for only the control samples for water cured specimen compared to the solar chamber and ambient air were respectively 31 and 28%. The trend observed was similar for all percentage replacement with copper slag. It is believed an increase in the curing temperature of average values 35°C and 25°C respectively for solar and ambient air curing environment compared to water at 20°C increased the rate and extent of reaction through an increase in the heat of reaction; consequently increasing the early compressive strength development of the concrete cubes in both solar and ambient air curing methods compared to those cured in water.

- For compressive strength development beyond 28 up to 90 days, there was observed a significant reduction in the late compressive strength development of all concrete specimen cured in both ambient air and solar chamber, compared to water cured specimen. For control samples up to 90 days of curing, the percentage increase in the compressive strength for water cured specimen compared, to the solar chamber and ambient air were respectively 8.5 and 12%. The late strength development was high for water cured specimen followed by solar and ambient air. This trend was similar for all pulverized copper slag concrete samples. The higher late strength development for concrete samples cured in water was attributed to water providing adequate moisture for further hydration as compared to the solar chamber and ambient air.

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