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Effect of Curing Conditions on the Compressive Strength and Microstructure of Alkali-activated GGBS paste

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ABSTRACT: *This paper presents the effect of curing method on the strength development of alkali- activated blast furnace slag paste. In this study, alkali activation was done using a combination of potassium hydroxide and sodium silicate. The test parameters include the curing methods (water curing at 27^0C, heat curing at 50^0C and controlled curing with relative humidity 50%, 70% and 90 % at 27^0 C), alkali content with 6.41 %, 8.41 %, 10.41% and 12.41 % of the mass of GGBS. The compressive strength results showed that there is an increase in compressive strength with the increase in age of water curing and controlled curing specimens. A comparison of hot cured specimens, the increase in compressive strength with age was less. Further heat curing has shown to adversely affect compressive strength and to create internal micro cracking as well as surface cracks. The higher compressive strengths were obtained from water cured specimens.*

Keywords: *Alkali content, blast furnace slag, compressive strength, curing conditions, microstructure, scanning electron microscopy (SEM).*

I. INTRODUCTION

The production of concrete is used by some economists as a measure of a country's economic strength. Some countries already have a tax on carbon dioxide generation. Portland cement production has been already second only to automobile as the major generator of CO_2 greenhouse gas worldwide concrete production. For every ton of cement produced generates nearly one tone of carbon dioxide. Among the greenhouse gases, CO_2 contributes about 65% of global warming. Hence, to preserve the environment, we are in need of new sustainable and environmentally friendly composites to replace conventional Portland cement. Ground granulated blast furnace slag is a by- product from manufacturing of iron, is a molten material which appears above the pig iron at the bottom of the blast furnace. The main component of slag, melilite is a solid solution of ($2CaO.Al_2O_3.SiO_2$) ($2CaO.MgO.2SiO_2$), consisting 40% by weight of CaO. The production of 1 ton of iron results in the by-product of 0.6 tons of iron slag and generates 0.19 tons of chemical- CO_2 [1].

The term 'Curing' stands for procedures devoted to hydration reaction of GGBS, consisting of control of time, temperature, and humidity conditions. The properties of alkali activated GGBS are dependent on the hydration and cement and time. The strength development of alkali activated slag cement concrete cured in water bath, sealed and exposed to air conditions was studied earlier [2]. They have reported that when the concrete is cured in water, compressive strength of concrete keeps increasing until the end of the testing period of 365 days. However, if the specimens are cured in sealed conditions, the strength stopped increasing at about 90 days and the strength of the exposed specimens was found to be 54% and 41% less than bath and sealed specimen strengths, respectively, at 365 days. The early age strength development (up to 28 days) of GGBS blended concrete had found to be increasing with curing temperature, but the effect on later strength is very less [3-5]. It was found that the effects of curing method for strength development are found only after the age of 7 days [5]. For improved durability room temperature curing is the best, but steam curing is preferable to autoclaving curing, if rapid strength development is required [6]. Some studies suggested internal curing is an effective method for improving performance of low W/C – low permeability concrete because they require additional water to hydrate the cementitious materials [7]. Curing conditions had a significant effect on the mechanical behavior in the hardened state of alkali activated slag paste compared to ordinary Portland cement [8]. In comparing lab-cured cylinders to in-situ strength, it is commonly known that in-situ strength is lower than strength determined from lab-cured cylinders under standard conditions. A factor of 0.85 is typically assumed, although more recent work indicates the factor may be as low as 0.7 [9]. The pore size distribution is strongly influenced by the curing temperature; higher temperature increases the volume of mesopores [10]. If the potential of concrete with regards to strength and durability is to be fully utilized, it is most essential to be cured adequately. As reported by researchers [11], the higher strengths could be obtained using lower binder contents for cement and cement-slag mortars provided the specimens were cured in water. It was reported that the increase in level of cement and slag makes the mortars more sensitive to air curing conditions. The curing becomes even more important if the concrete contains supplementary cement materials such as fly ash or ground granulated blast-furnace slag or silica fume, and is subjected to hot and dry environments immediately after casting [12]. The fineness of GGBS also plays a significant role in developing the compressive strength of Alkali-activated GGBS paste [13].

To obtain the required durability, strength and high performance during the life cycle of the structure, curing is crucial. Many previous researchers studied the behavior of particular slag at either room temperature or at elevated temperature, but very few studies compare in- depth the activation of GGBS paste with different curing conditions. This paper emphasizes the effect of curing method on the compressive strength development of alkali activated blast furnace slag paste.

Table 1: Compressive strength and workability of AABFS paste with different curing conditions (% of mass added with respect to the total mass of slag)

S.No.	% (K_2O + Na_2O)	% SiO_2	Mix ID	Compressive Strength (MPa)		
				3 d	7d	28 d
1	6.41	8	WC	17.5	29.50	41.5
2	6.41	8	OC50	16.92	18.2	21.13
3	6.41	8	RH50	14.5	21.9	27
4	6.41	8	RH70	15	23.5	35.1
5	6.41	8	RH90	16.5	28.53	40.5
6	8.41	8	WC	25.5	36.3	47.4
7	8.41	8	OC50	25.88	27.5	32.6
8	8.41	8	RH50	18.95	26.5	33.5
9	8.41	8	RH70	22.6	32.03	39.3
10	8.41	8	RH90	24.8	36.69	44.1
11	10.41	8	WC	29.50	38.30	50.2
12	10.41	8	OC50	30.8	32	39.12
13	10.41	8	RH50	22.95	33.55	43.33
14	10.41	8	RH70	24.5	37.4	46.5
15	10.41	8	RH90	29.5	39.2	47.25
16	12.41	8	WC	23.4	35.2	44.7
17	12.41	8	OC50	26.68	28.4	34.15
18	12.41	8	RH50	20.38	28.6	38.11
19	12.41	8	RH70	22.9	33.43	42.4
20	12.41	8	RH90	24.5	35.4	44.4

II. EXPERIMENTAL DETAILS

2.1 Materials

In the present study, ground granulated blast furnace slag (GGBS) was used as a source material for the synthesis of alkali activated blast furnace slag paste (AAGGBS). GGBS was obtained from the Tata Metaliks Ltd. Kharagpur, India. The slag obtained was ground to fineness less than 45 microns. The chemical composition was determined by X-Ray fluorescence spectrometry (XRF) analysis and in terms of the main oxides was (in weight %) SiO_2 (32.50), CaO (33.50), Al_2O_3 (18.50), MgO (8.0), Fe_2O_3 (0.40), S (0.5) and MnO (0.55).

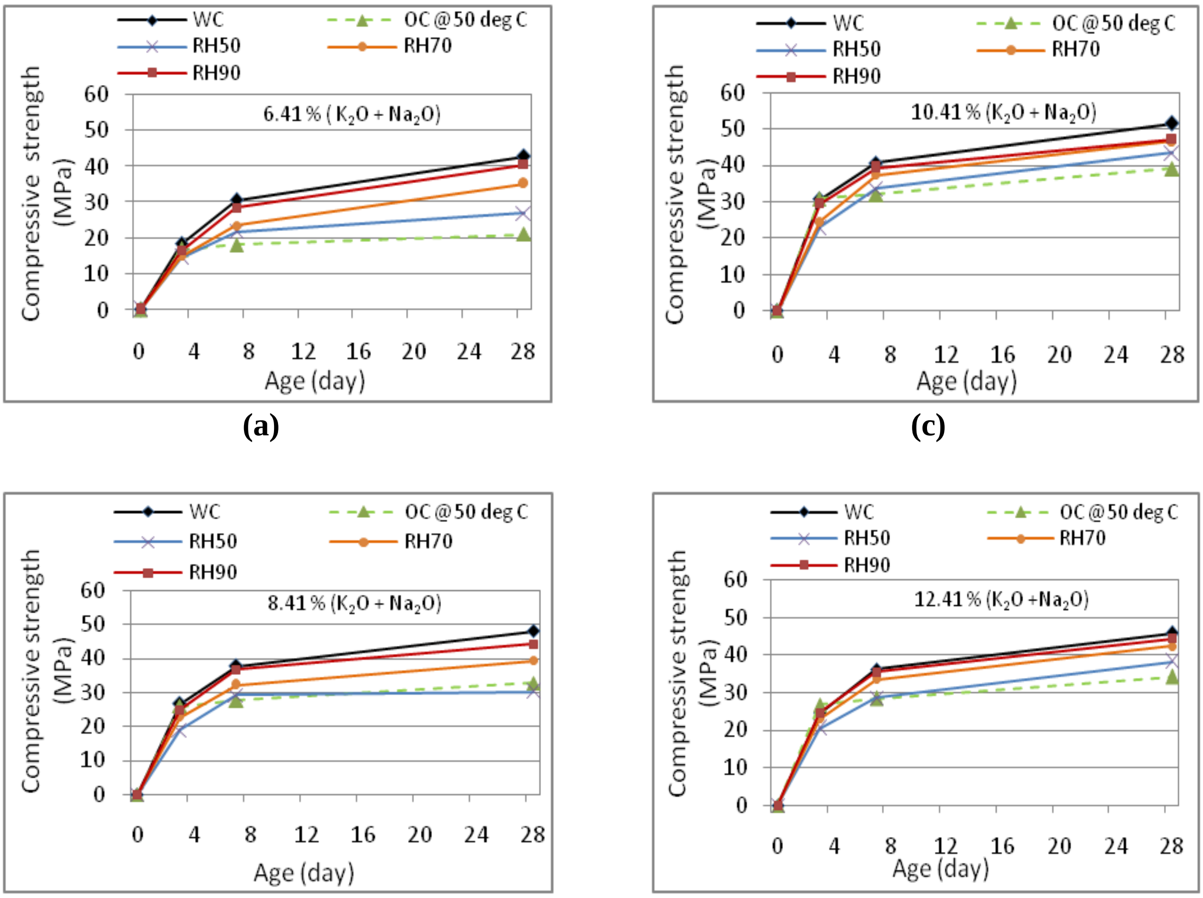
For the alkaline activator, a combination of potassium hydroxide and sodium silicate solution was used. Potassium hydroxide in pellet forms with 84% purity, supplied by Merck India Ltd. (K_2O =83. 93% and 16.07% water) and sodium silicate solution (Na_2O = 8%, SiO_2 = 26.5% and 65.50% water) with silicate modulus ~3. 3 and bulk density of 1410 kg/m^3 was supplied by Loba Chemie Ltd. India, were used to adjust the desired composition of alkali activated GGBS paste. The activator solution was prepared at least one day prior to its use. Potable water was used in all mixes for casting and in curing of the specimens.

2.2 Design of Mix Proportion

In this experimental work, the alkali activated GGBS paste was prepared according to the composition shown in Table 1. The compositional change in GGBS mix was obtained by adjusting the quantity of potassium hydroxide (KOH), sodium silicate solution and water.

2.3 Mixing and casting of test specimens

Mixing procedure was carried out in two stages. Initially, GGBS (in dry condition) were hand mixed in a container for about 5 minutes along with the desired proportion of alkaline solution and then the mixture is transferred in Hobart mixer and mixed for another five minutes to get homogeneous paste. The fresh mix was placed in steel moulds of size 50 mm x 50 mm with two layers of fresh paste and each layer is compacted using a rod of 16 mm diameter. Then the moulds with fresh mix were vibrated for two minutes on vibrating table to remove any entrapped air. The workability of fresh paste was assessed using mini flow table test as per ASTM C 1437-07 [14] with a modification; the table was raised and dropped 15 times in about 15 seconds. The mini flow table apparatus was used as per ASTM C 230/C 230M-08 [15]. The workability of the mix was determined by measuring the diameter of paste flow on a flow table in two perpendicular directions after 15 drops in 15 seconds and the average value is considered as a flow diameter. The percentage increase in flow diameter with respect to the initial diameter of mould is considered as flow value. The flow percentage was found to be 41 %, 55 %, 65 % and 75 % for 6.41 %, 8.41 %, 10.41 % and 12.41 % alkali content respectively [16].



(a)

(c)

(b)

(d)

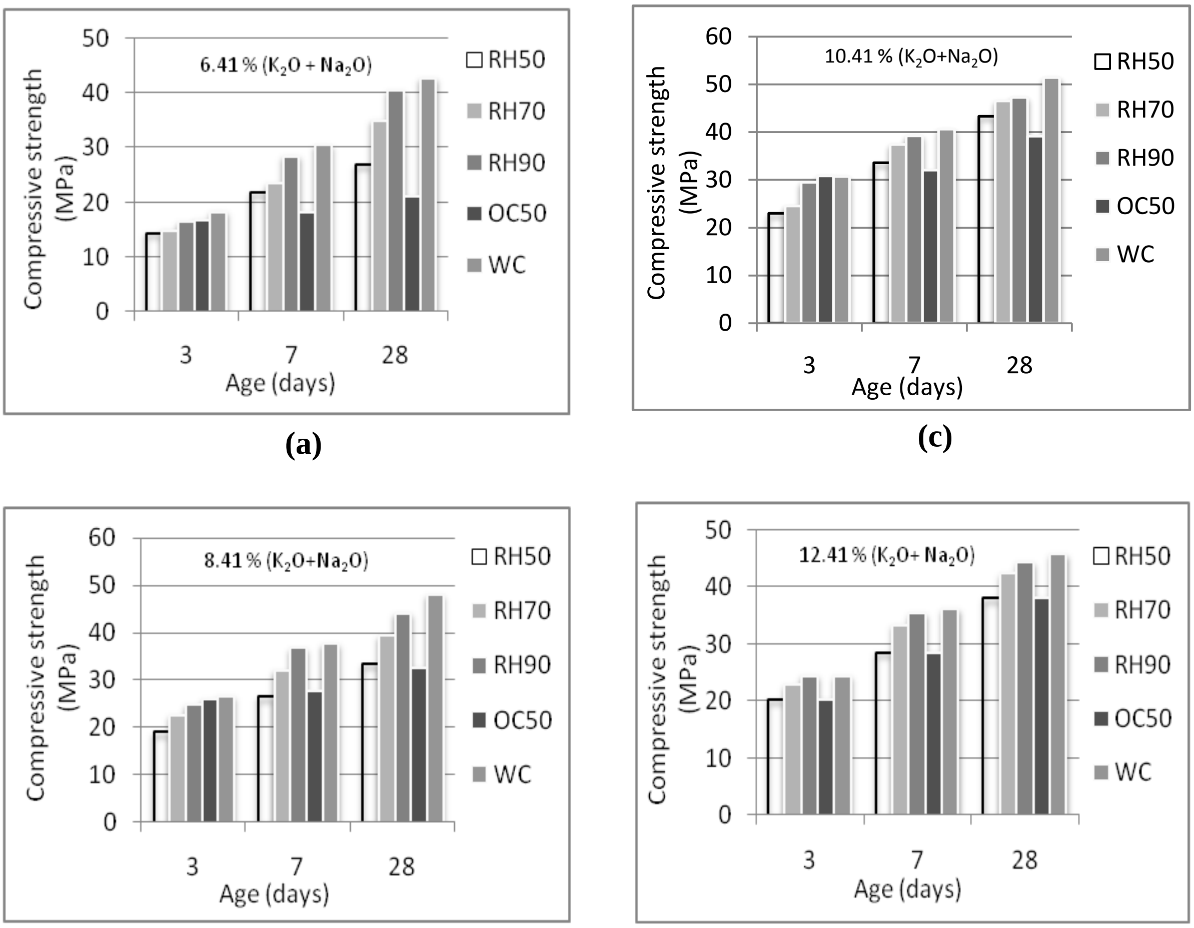
Figure 1: The effect of various curing conditions on compressive strength for different alkali content (K_2O+ Na_2O) (a) 6.41 % (b) 8.41 % (c) 10.41 % and (d) 12.41 %.

2.4 Curing conditions of test specimens

The test specimens were cured in water curing , controlled curing with 50 % , 70 % and 90 % relative humidity and dry oven curing at 50^0 C designated as 'WC', 'RH50', 'RH70', 'RH90' and 'OC50' respectively. For water curing, the specimens after casting were left at room temperature for 24 hours, then de-molded and kept in water in fully immersed condition at room temperature until testing was done. For controlled curing, the specimens after casting were left at room temperature for 24 hours, then de-molded and kept in humidity chamber at 50 % RH, 70 % RH and 90 % RH. For heat curing, the specimens after casting with two hour delay were kept in the oven at 50^0 C for 24 hours. The test specimens then removed from the molds and left to air dry in the room temperature conditions until tested for direct compression at a specified age.

2.5 Test Methods

The alkali-activated GGBS (AAGGBS) paste specimens (50mm x 50mm x 50mm) were tested for compressive strength using 20 ton capacity digital compressive testing machine with a loading rate of 20 MPa/min. The compressive strength tests were conducted at the age of 3, 7 and 28 days. Three specimens of each series at each age were crushed in a digital compression testing machine in accordance with ASTM C-109-02 [17] and the average strength of three specimens is reported as the compressive strength.



(a)

(c)

(b)

(d)

Figure 2: Variation of compressive strength with age for various curing conditions for different alkali content (K_2O+ Na_2O) (a) 6.41 % (b) 8.41 % (c) 10.41 % and (d) 12.41 %.

III. RESULTS AND DISCUSSION

3.1 Influence of curing conditions on compressive strength

Table 1 presents synthesis parameters and the compressive strength of AAGGBS paste specimens produced at alkali content (K_2O +Na_2O) varied from 6.41 % to 12.41 %. Water to slag ratio and silica content was kept constant to 0.32 and 8 % respectively. Variations of compressive strength of the specimens cured in water (WC) at room temperature (27^0 C), controlled curing with 50 % (RH50), 70 % (RH70) and 90 % (RH90) relative humidity and dry oven curing at 50^0 C (OC50) are shown in Fig. 1 and 2. It was observed that the compressive strength increases with increase in alkali content from 6.41 % to 10.41 % for all types of curing methods and reaches maximum 50.20 MPa for alkali content of 10.41 % for water cured specimens. The compressive strength results showed that, there was an increase in strength with increase in age for all types of curing methods. The percentage increase in 28 days strength over 3 days strength was found to be 67.55 %, 27.01 %, 88.88 %, 89.79 % and 60.16 % for WC, OC50, RH50, RH70 and RH90 respectively for the optimum 8 % (K_2O +Na_2O). Comparisons of hot cured samples the increase in compressive strength with age was less. The hydration of cement can take place only when the vapor pressure in the capillaries is sufficiently high, about 0.8 of saturation pressure [18,19]. Therefore, early drying of concrete may stop the cement hydration before the pores are blocked by hydration products and thus a more continuous pore structure may be formed. The comparison of compressive strength of various curing methods was given in Table 1. The results revealed that the 28 days strength of water cured specimens were 31.49 %, 18.71 %, 10.62 %, and 8.86 % higher than that of OC50, RH50, RH70 and RH90 respectively for 10.41 % alkali content.

3.2 Influence of alkali content on compressive strength

The relationship between alkali content and compressive strength is presented in Fig. 3.The increase in compressive strength was observed with increase in alkali content until 10.41 % alkali content. Further increase in alkali content reduces the compressive strength. This trend was observed for all types of curing method. This may be due to excess K^+ ions in the framework, the slag grains might not completely participated in the reaction process forming C-S-H gel and the degree of reaction of the system might remain moderate. In the present study, the optimum alkali content was observed to be 10.41 % with a SiO_2/ (K_2O+Na_2O) ratio of 0.76 for all types of curing methods. The optimum silicate modulus phenomenon to the formation of "primary C-S-H" and /or polymerization of silicate anions in the water glass, which promotes the hydration of slag and the formation of less porous structure [20]. The alkali metal cations in the original materials or added alkali metal hydroxides are considered important due to their catalytic role.

3.3 A microstructure study by scanning electron microscopy (SEM)

Microstructural images of alkali activated hardened paste specimens at 28 days were obtained from optimum alkali content of 10.41% and SiO_2 content of 8 %. Scanning Electron Microscoy (SEM) was used to record micrographs using a JEOL JSM 6360 scanning electron microscope. Fig. 4 shows the microstructure of AAGGBS hardened paste specimens having optimum alkali content of 10.41% and SiO_2 with 8 % for various curing conditions (RH50, RH70, RH90, OC50 and WC) .The formation of C-S-H in the AAGGBS matrix has a significant effect on the development of compressive strength. The matrix of water cured specimen was different from other type of specimens as the microstructure was very dense with fewer unreacted slag grains and voids, hence responsible for higher compressive strength. Microstructure of oven cured (50^0 C) specimens shown the micro cracks as clearly observed from Fig. 4 (d).

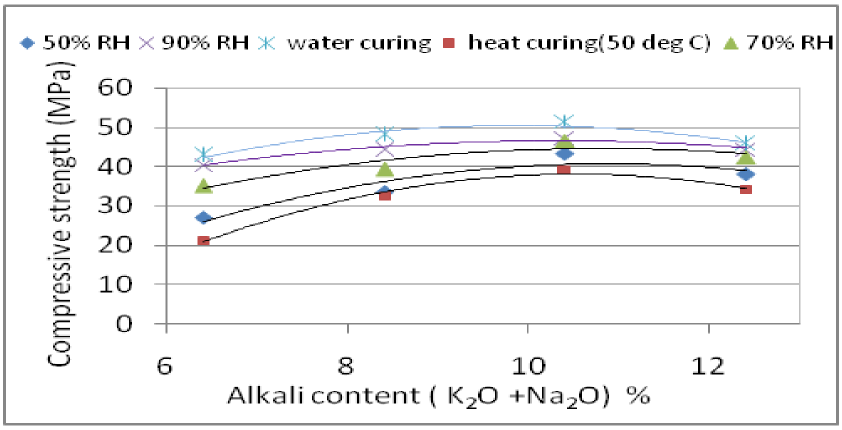


Figure 3: The Effect of alkali content on compressive strength under different curing conditions.

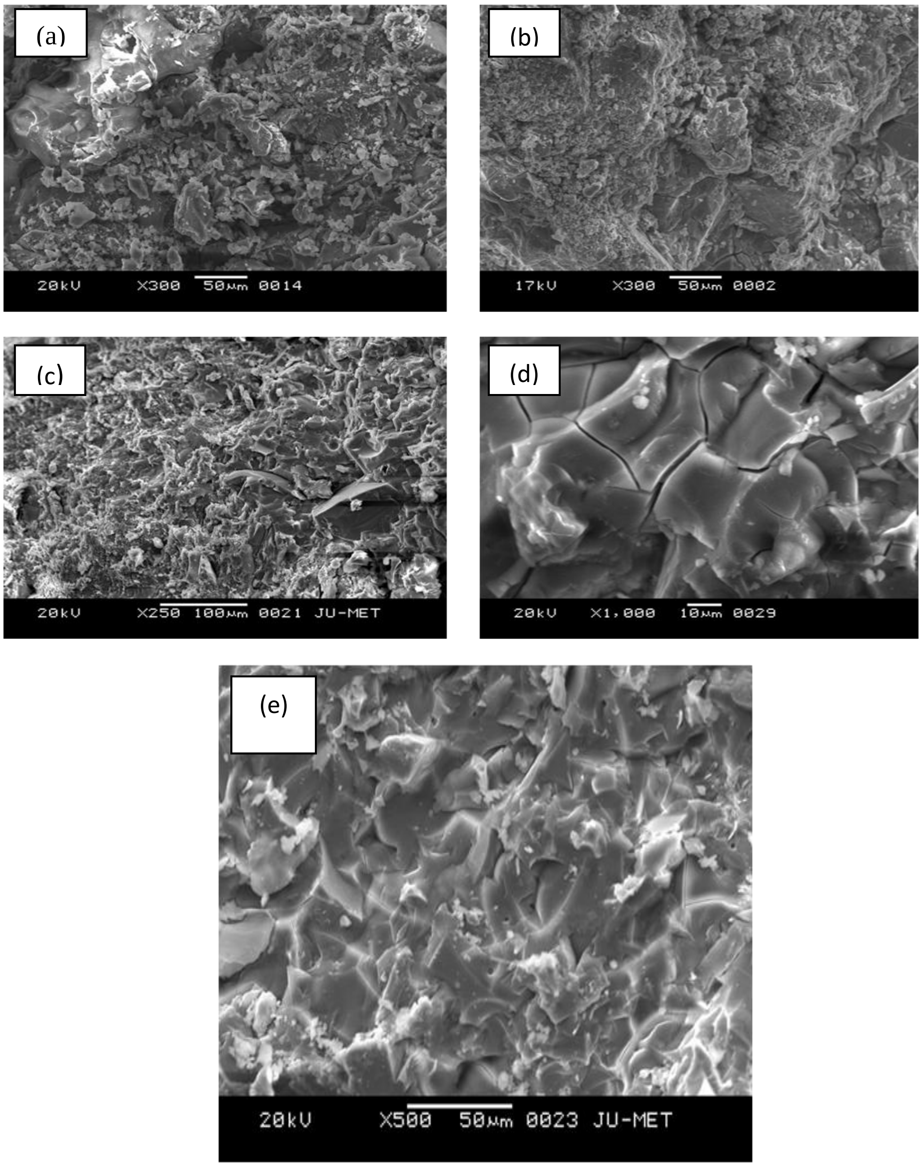


Figure 4: SEM micrograph of alkali activated GGBS with different curing conditions with 10.41% alkali content and 8% SiO_2 (a) RH50, (b) RH70, (c) RH90, (d) OC50 and (e) WC.

IV. CONCLUSION

Based on the experimental investigation, the following conclusions are drawn.

- The higher strength was obtained from water cured specimens for all types of alkali contents. The optimum alkali content ($K_2O + Na_2O$) was found to be 10.41 % with a $SiO_2/(K_2O+Na_2O)$ ratio of 0 .76. The maximum strength of 50.20 MPa was obtained from water cured sample having 10.41 % alkali content. (Fig.1c, 2c and Fig.3).
- The oven curing is more sensitive for alkali activated slag. The micro cracks were observed on the surface of the specimens as observed in the SEM image (Fig. 4d).
- Curing conditions had a significant effect on the mechanical behavior in the hardened state of alkali activated slag paste. The compressive strength of the alkali activated blast furnace slag paste can be controlled by judiciously choosing the curing conditions.

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