Preparation and Characterization of Low Cost Geopolymer Cement

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Abstract:

This paper presents the preparation and characterization of geopolymer cement. Sodium hydroxide with different percentage (4%, 6% and 8%) with sodium silicate (5%, 10% and 15%) of the mass of blast furnace slag are mixed to prepare the activator. The slag is mixed with the activator of different concentrations to prepare the geopolymer pastes. The physicochemical and mechanical properties of the hardened pastes cured in water at room temperature for different time intervals up to 90 days are measured.XRD of some selected samples are made. The results indicate that alkali content has significant effect on the physicochemical and mechanical properties of the hardened pastes. Compressive strength, combined water contents and bulk density increase while total porosity decrease with increasing alkali concentration.

Keywords:

Alkali activated blast furnace slag, Compressive strength, bulk density, total porosity and combined water.

I. INTRODUCTION

Construction industry contributes around 5-8 % of all global CO_2 emissions and this figure will increase in the coming decades as the developing world continues to use more and more concrete . Ordinary Portland cement is one the most pollutant products in terms of CO_2 emissions [Duxson et al. 2008]. Cement and concrete industries have implemented several mechanisms to reduce the CO_2 emissions of concrete, but they have had only a very minimal effect [Duxson et al. 2008]. The greatest carbon dioxide savings from the industry are likely to be achieved by the inclusion of supplementary cementitious materials [Tyrer et al. 2010], such as fly ash [Pedersen et al. 2008] or ground granulated blast-furnace slag [O'Rourke et al. 2009]. The manufacture of alkali-activated blast furnace slag may be considered as an alternate binder to ordinary Portland cement. Alkali-activated binders are receiving much attention because of their high strength, durability and low environmental impact [Juenger et al. 2011]. The greenhouse gas savings achievable through the use of alkali-activated concrete has been estimated around 80 % compared to Portland cement concretes [Duxson et al. 2008, Duxson et al. 2007]. Alkali-activation of ground granulated blast furnace slag (GGBS) has becomes the subject of intense research interest within the past decades. Strength development of slag cement is highly sensitive to curing conditions [Sanjayan et al. 2000].

Alkaline activation of blast furnace slag is affected due to the high concentration of OH ions in the mix [Krizan et al. 2002]. It has been reported that all alkalis and alkali compounds whose anions or anion groups can react with Ca to produce Ca compounds that are less soluble than $Ca(OH)_2$ can act as activators of slags [Shi et al. 1995, Shi et al. 1996, Caijun et al. 1989, Krizan et al. 2002]. Bakharev et al. (1999) reported that heat treatment had a significant accelerating effect on strength development of slag pastes. They further concluded that sodium silicates provided the best activation, with compressive strength of pastes, mortars and concrete exceeding that of OPC pastes of the same w/b ratio. The alkali and silicate content also plays a vital role in the development of compressive strength in pastes and concrete [Qureshi et al. 2013, & 2014].The compressive strength of the sodium silicate series increases linearly with increasing the ratio of Na_2O/SiO_2 Luukkonen et al. (2017). Alkali-activated materials (AAMs) (and geopolymers as their sub-group [Provis 2014]) are recognized as one of the alternative low-CO_2 binders [Scrivener et al. 2016] and as a way to beneficiate several kinds of industrial wastes into higher-value products [Mehta et al. 2016]. Ground granulated blast-furnace slag (GGBFS), an amorphous by-product of pig iron production, is one of the most widely studied precursors in alkali activation. It mainly consists of a $CaO—MgO—Al_2O_3—SiO_2$ system [Li et al. 2011] and forms calcium-aluminum-silicate-hydrate (C-A-S-H in cement chemist notation) gel as a result of alkali activation [Bernal et al. 2014]. The first patent regarding the alkali activation of GGBFS was published as early as 1895 and these binders have been studied extensively since then and also used in construction [Palomo et al. 2015]. The historical development of AAMs is discussed, in a recent review by Krivenko (2017) .

This paper investigates the effect of alkali concentration on engineering properties such as bulk density, total porosity, combined water and compressive strength of AAS pastes.

II. EXPERIMENTAL INVESTIGATION

2.1. Materials

2.1.1 Blast furnace slag

Blast furnace slag (GGBF) used in this investigation was obtained from Helwan Steel Company, Helwan,Egypt. The obtained blast furnace slag was then ground to size 45 microns, has a surface area fineness of about 400 to 600 m^2 /Kg, The moisture content was less than 1% and its density was 2900 kg/m^3. The angular-shaped ground slag in presence of water and an alkali activator sets like Portland cement.

The chemical composition was determined by XRF as shown in Table 1.

Table 1: Chemical composition of blast furnace slag by XRF

Oxide	CaO	MgO	Fe_2O_3	K_2O	Na_2O	Al_2O_3	SiO_2	SO_3	L.O.I[a]
Mass (%)	42.56	11.58	1.14	0.15	0.29	7.02	32.86	2.50	0.93

[a] Loss on ignition

2.1.2 Alkaline Solution

The alkaline activator liquid was the combination of liquid sodium silicate (Na_2SiO_3) and pellets of sodium hydroxide (NaOH). NaOH of 99.99 % purity was purchased from Fisher Scientific Chemical Company. Na_2SiO_3 solution of (Na_2O = 11 %, SiO_2 = 32 % and 57 % water) and bulk density of 1460 kg/m^3 was supplied by EL – Goumhouria chemical company, Cairo, Egypt. The alkaline activator liquid was used to adjust the desired composition of alkali activated GGBFS paste. The alkaline activator (AA) is prepared by mixing NaOH & Na_2SiO_3 with different ratios. The temperature of the mixture was initially quite high then left to reach to room temperature in few minutes before proceeding.

2.2 Mixing and castings of test specimens

The alkali activated GGBS paste was prepared according to the composition presented in table 2. The compositional change in GGBFS paste was obtained by adjusting the quantity of NaOH & Na_2SiO_3 solution with water. To make alkali activated GGBFS paste, the desired proportion of blast furnace slag and activating solution were first mixed together for 5 min in a Hobart mixer to get a homogeneous paste. The mixing procedure is similar to that described by Qureshi and Ghosh (2013). Geopolymer is obtained by the addition of activator solution to each dry mix. After mixing the water consistency, initial and final setting time of the geopolymer pastes is determined by standard Vicat apparatus according to AATM C 191 (2013).

Table 2: Mix composition of the alkaline activator.

MIX	**S1**	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**	**S8**	**S9**
Na_2SiO_3 %	**5**			**10**			**15**		
NaOH %	**4**	**6**	**8**	**4**	**6**	**8**	**4**	**6**	**8**

2.3 Curing conditions of test specimens

The paste is placed in stainless-steel mold 1-inch cubic, then the mold was kept in humidifier (100 % R.H) at room temperature for the first 24 hrs. After this period the cubes are removed from the mold and then kept in water in fully immersed condition at room temperature until the day of testing 2, 7, 28, 56 and 90 days.

2.4 Test Procedure

The hardened cubes of the different mixes have been tested via determination of compressive strength, bulk density, total porosity and combined water content. The formed hydration products were determined by XRD analysis.

The alkali activated GGBFS paste specimens are tested for compressive strength using 20 ton capacity digital compressive testing machine with a loading rate of 200 kg/min. The Compressive Strength test is carried out according to ASTM C-109 (2013). Three specimens of each series at each age are crushed and the average strength of three specimens is reported as the compressive strength.

Bulk density and total porosity are determined according to Archimedes principle with the water as immersion medium.

III. RESULTS AND DISCUSSION

3.1 Influence of Alkali content on Compressive strength

The relationship between alkali content and compressive strength is shown in Fig. 1. An increase in compressive strength is observed with increase in alkali content. This may be due to excess Na^+ 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 [Qureshi et al. 2013]. The optimum alkali content was observed to be 15.0 % of Na_2SiO_3 and 8 % NaOH content. 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 [Bernal et al. 2014]. The Compressive Strength test is carried out according to ASTM C109 M (2016).

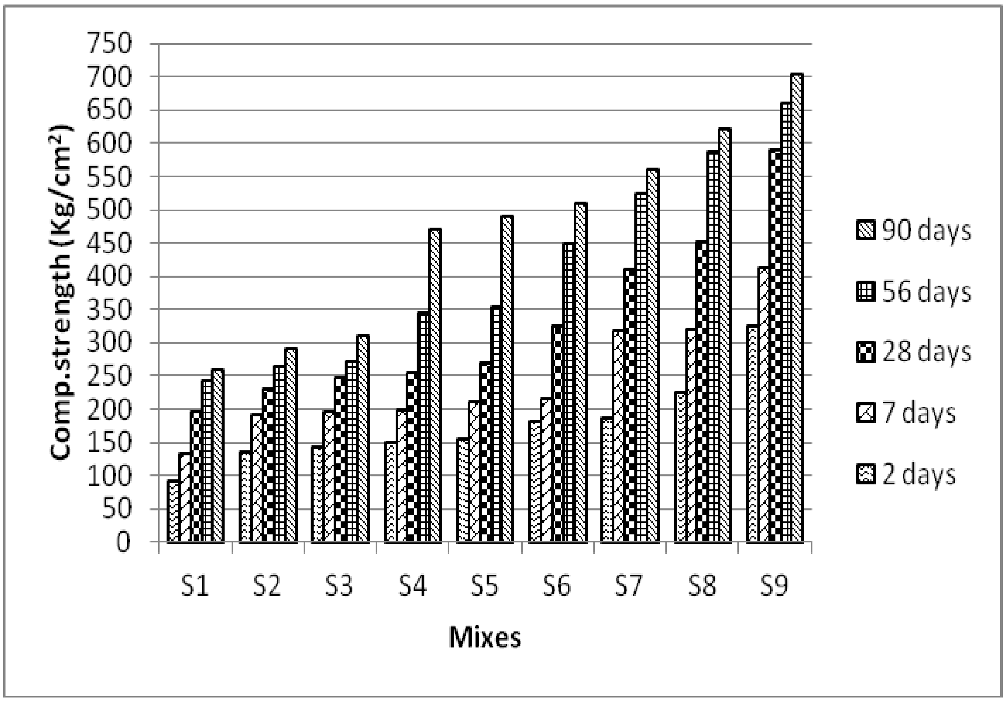

Fig. 1 Relation between alkali content and compressive strength for various curing time.

3.2 Bulk Density

The relationship between alkali content and bulk density is illustrated in Fig. 2. The increase in bulk density was observed with increase in alkali content until 15.0 % of Na_2SiO_3 and 8 % NaOH content. It is clear that the formed hydration products fill a part of open pores, therefore the bulk density increase.

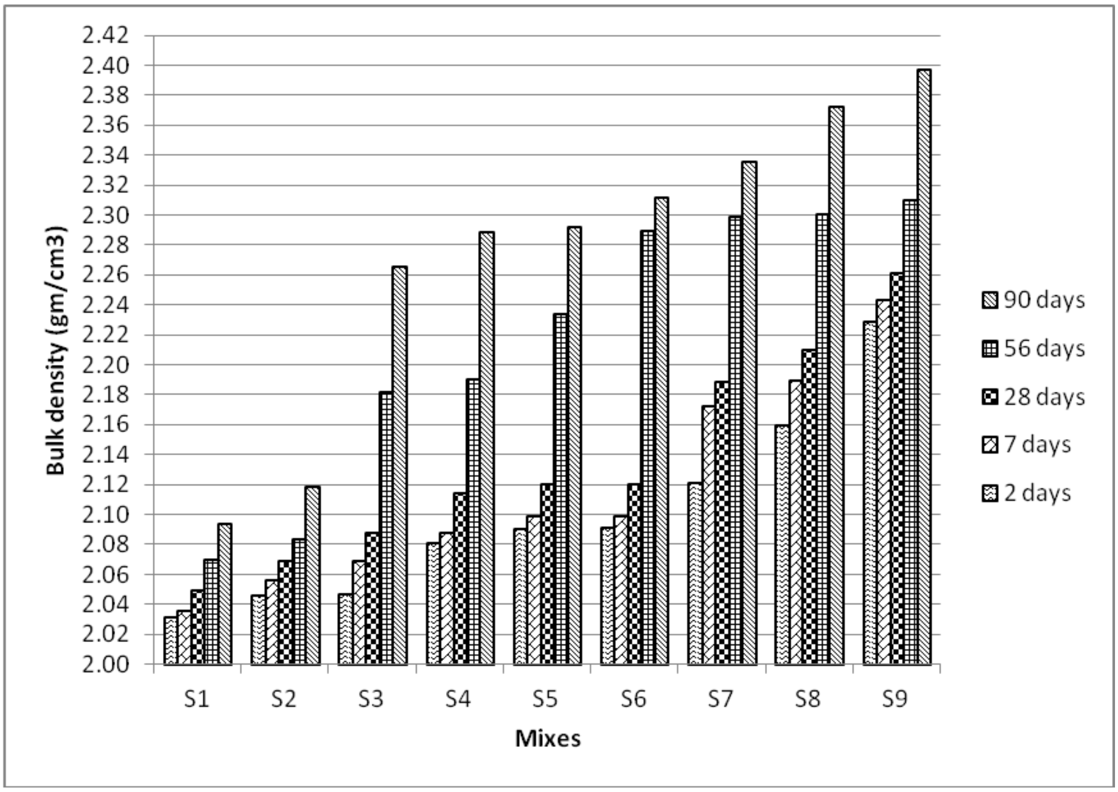

Fig. 2 Relation between alkali content and bulk density for various curing time.

3.3 Porosity

The porosity of the prepared geopolymer hardened pastes decrease with increase in alkali concentration as shown in Fig. 3.

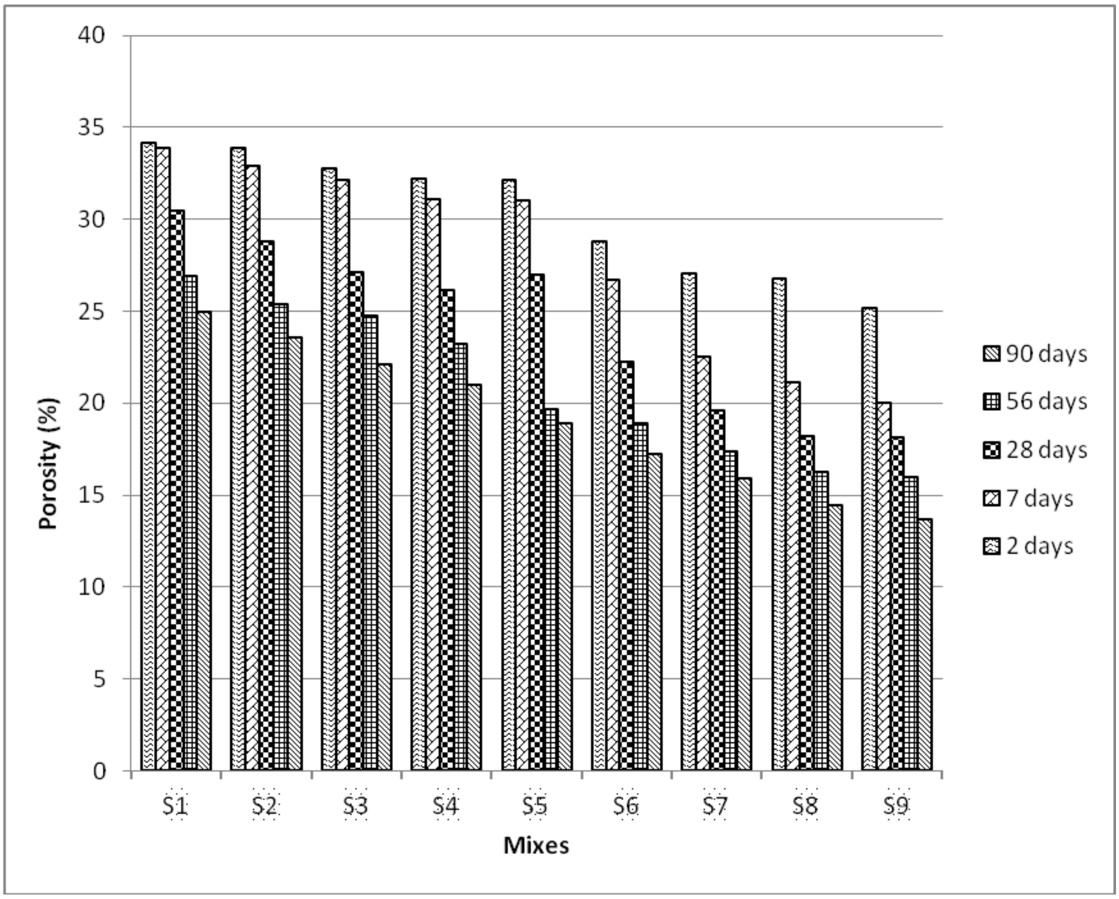

Fig. 3 Relation between alkali content and porosity for various curing time.

3.4 Combined Water (Wn. %)

The relationship between alkali content and combined water contents is presented in Fig.4. The combined water contents depend on the amount and type of the hydration products. It is clear that the chemically combined water contents increase with curing time for all mixes. As the hydration time increases more hydration products formed and precipitated in the available open pores, and then the chemically combined water contents increase with curing time.

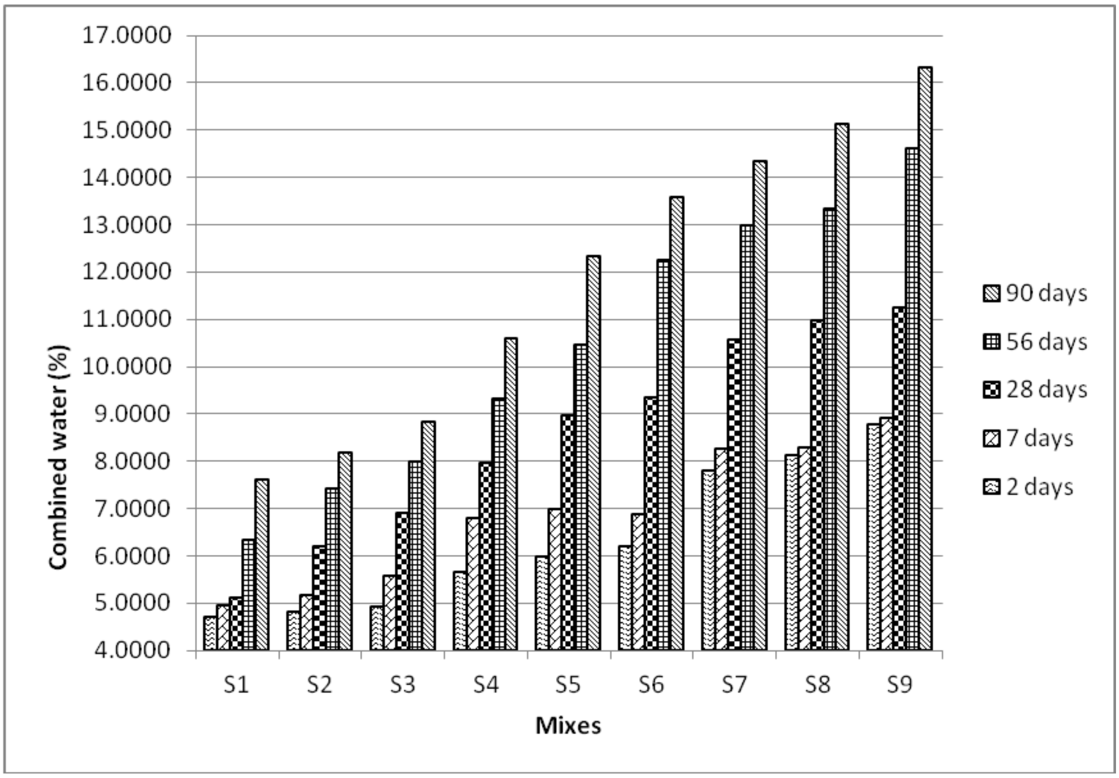

Fig. 4 Relation between alkali content and combined water for various curing time.

3.5 X-ray diffraction

In the X-ray diffraction (XRD) patterns of alkali activated slag, the hump peak can be observed in the range of 25-35°, 2Ə changes with curing time 7, 28 and 90 days suggesting that poor crystalline CSH gel produced in the pastes. Geopolymer gel and CSH gel could be formed after setting and hardening. In alkali activated slag, the presence of the strong peak at d = 3.08 A° is attributed to CSH phases as shown in Figs. 5,6,7,8,9,10,11,12 and 13.

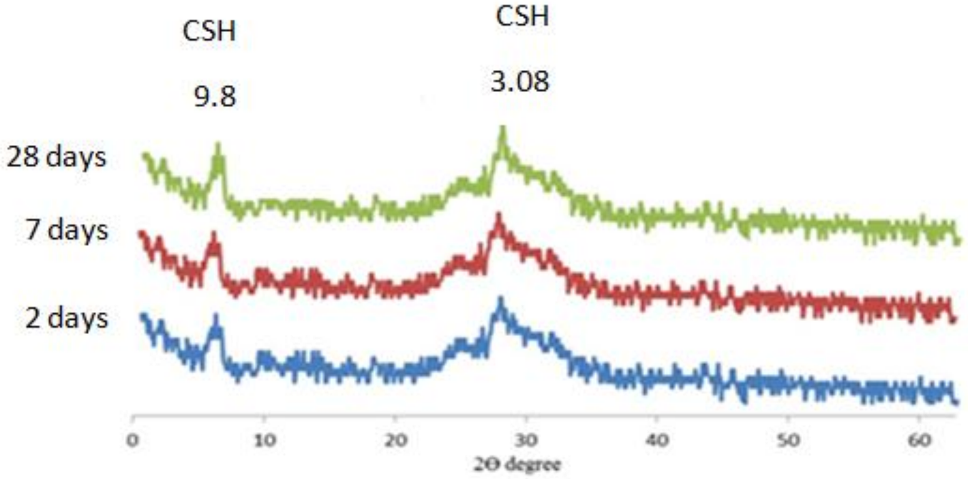

Fig.5 X-ray diffraction patterns for mix (S1)

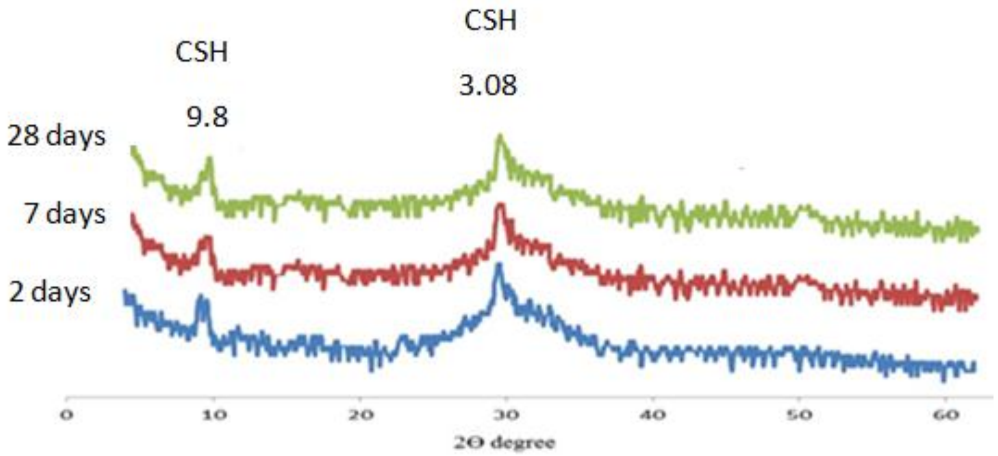

Fig.6 X-ray diffraction patterns for mix (S2)

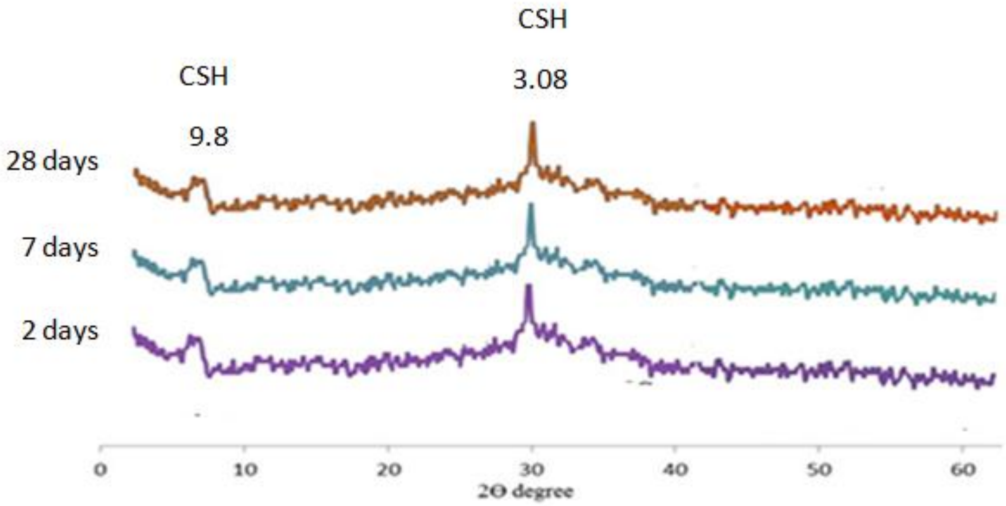

Fig.7 X-ray diffraction patterns for mix (S3)

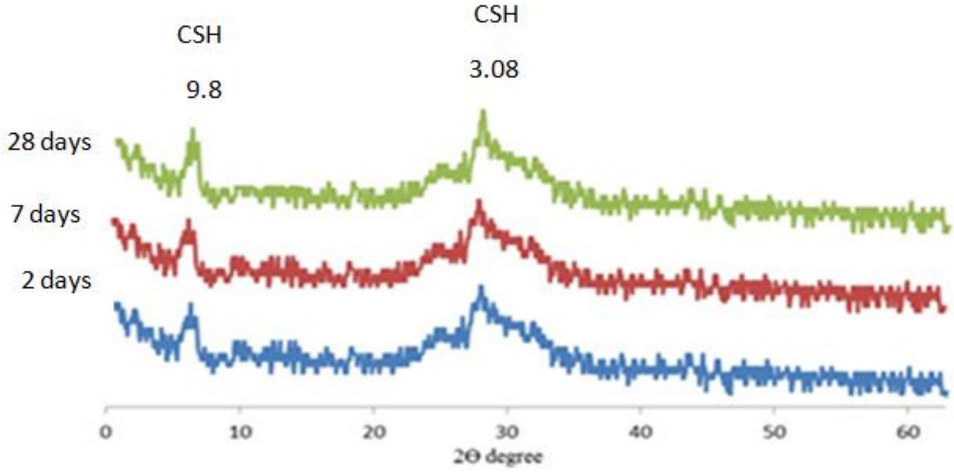

Fig.8 X-ray diffraction patterns for mix (S4)

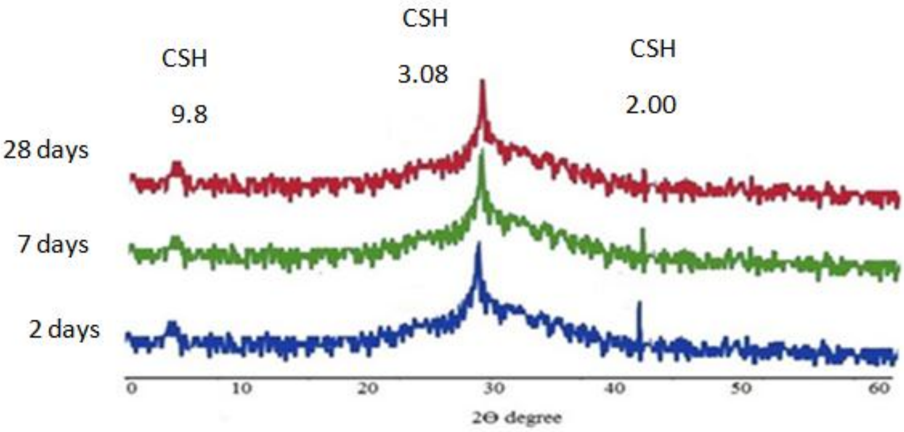

Fig.9 X-ray diffraction patterns for mix (S5)

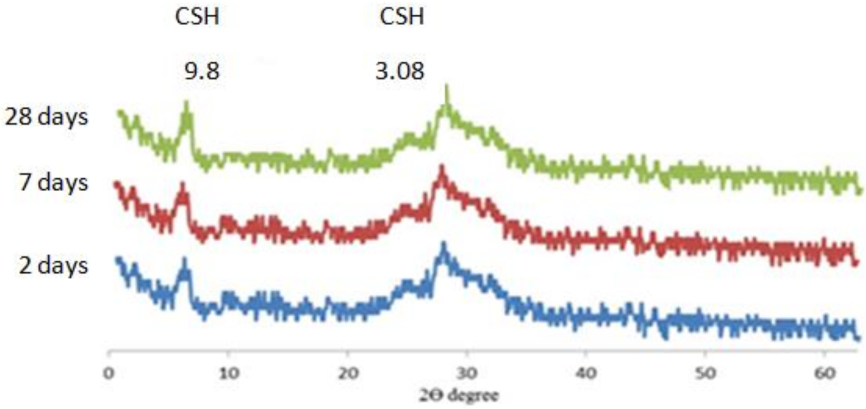

Fig.10 X-ray diffraction patterns for mix (S6)

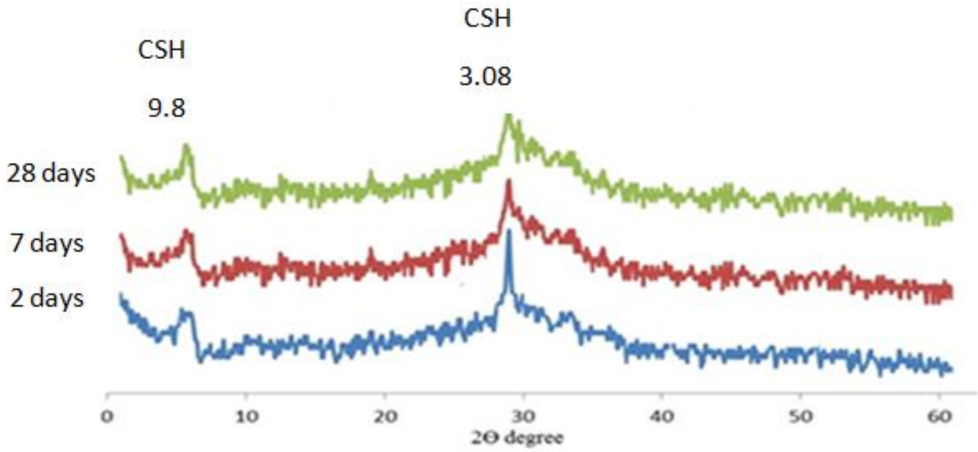

Fig.11 X-ray diffraction patterns for mix (S7)

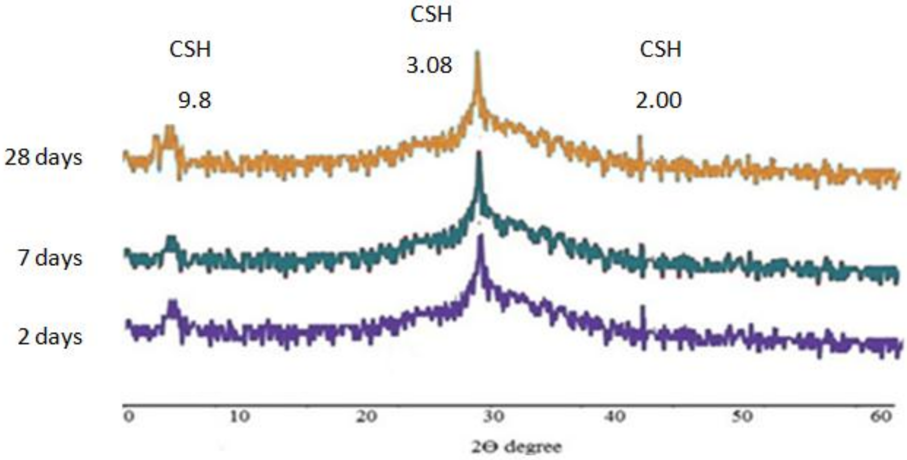

Fig.12 X-ray diffraction patterns for mix (S8)

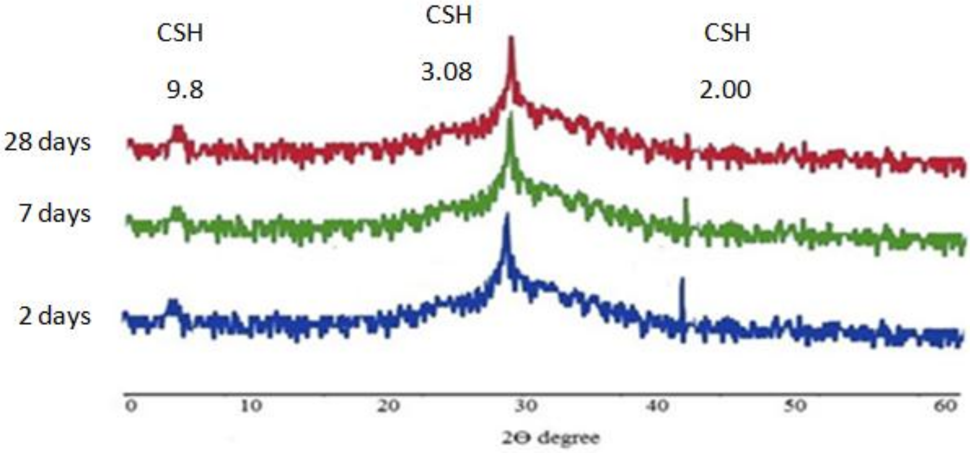

Fig.13 X-ray diffraction patterns for mix (S9)

IV. CONCLUSIONS

The effect of curing time on engineering properties such as compressive strength Bulk density, porosity and X-ray diffraction of alkali activated GGBS paste has been investigated. Based on experimental study, the following conclusions are drawn.

1- The geopolymer can be an alternative to cement in all applications. It can reach similar or higher strength and durability, without heat activation.

2- The characteristic strength of geopolymer shouldn’t be considered at 28 days like concrete. Geopolymer gain considerable strength up to 90 days.

3- The maximum compressive strength of 705 kg/cm^2 was achieved for water cured specimens for alkali content (sodium hydroxide content 8%) and sodium Silicate (15%), having lowest porosity of 13.6611 % and having highest bulk density and combined water of 2.3966, 17.3211 % respectively.

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دراسة الخواص الطبيعية والكيميائية والميكانيكية لعجائن الخبث المنشطة قلوياً

أشرف رزق جاد السيد[١] ، عصام عبد العزيز كيشار[١] ، دعاء عبد المنعم أحمد[١] ، محمد كحيل محمد فايز[٢]

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تم استخدام بعض المواد من البوزولانا الصناعية مثل حبيبات خبث الفرن العالي لتحضير أسمنت جيوبوليمري. باضافة هيدروكسيد الصوديوم وسيليكات الصوديوم كمواد منشطة للتفاعل حيث تم اضافتهم علي ماء الخلط وتم عمل تسعة خلطات بنسب مختلفة من المنشط وتم دراسة خواص كل خليط بعد عملية التأدرت في الماء بعد فترات زمنية تصل الي ٩٠ يوم وذلك بقياس مقاومة الضغط الميكانيكي، الكثافة، المسامية، الماء المتحد كيميائياً ودراسة التركيب الدقيق بإستخدام حيود الاشعة السينية. من هذه الدراسة اتضح أن الخليط المحتوي علي الخبث والمنشط بتركيز (٨ % هيدروكسيد صوديوم +١٥% سيليكات صوديوم) أنسب الخلطات من حيث جميع الخصائص التي تم دراستها.