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Research article

Investigating the expansion characteristics of geopolymer cement samples in a water bath and compared with the expansion of ASTM Class-G cement

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ABSTRACT

In selecting the binder composition for oil well application, its stability is an important design parameter. This paper presents the results of an experimental study conducted for comparing the linear expansion characteristics of geopolymer cement with the traditionally used ASTM Class G cement system. The expansion test was done in a water bath at 60 °C subjected to different curing intervals. The linear expansion of a cement system defines as the dimensional changes occur in the system, which is sometimes required to avoid the cement shrinkage during the hydration phase. In the case when the desired level of expansion is not achieved in the system, then the commercially available expandable materials are added in the class G cement system that enables the system to expand to the desired level. Shrinkage in the cementing system causes the formation of a microannulus or induces a gap that may allow the migration of fluid, hence the integrity of the system could be lost. This experimental study has revealed that the geopolymer cement tends to expand 0.15%–0.2% without the addition of any admixture, whereas the ASTM Class G cement has shown a lower value of linear expansion, which was obtained less than 0.1% after 18 days of curing. In the case of Class G cement, the addition of expandable material helped to increase the expansion; in the case of a geopolymer system, the additive has further accelerated the expansion.

1. Introduction

It is essential that the hydrocarbon production from a well should be safe, economical, and environmentally compatible. Therefore, flawless cementation of the installed casing strings is vital to prevent fluid flow through annuli to other features. The satisfactory performance of annulus is achieved by providing an impermeable bond between the casing strings and the surrounding rock. It is only possible if the cementing system possesses adequate compressive and shear bond strength by the time of the final plugging of the well.

Managing the uncontrolled migration of fluid and gas to the surfaces is a big challenge to the global oil and gas industry for a long time. Liquid and gas can migrate through the void spaces present either inside the cement sheath itself or through gap occurs between the casing and cementing or casing and the formation [1]. Once the fluid and gas catch the path to flow, it moves up to the surface and accumulates below the wellhead where it can build-up the pressure known as sustain casing pressure (SCP) [2]. SCP represents the potential risk of losing the hydrocarbon reserves and polluting the aquifer and sea [3]. The problem of SCP occurs in all types of wells, such as shallow gas wells, heavy oil producers, and also deep gas wells. All the problematic wells suffer from SCP issues require work-over jobs, replacing corroded tubing, and also cement remedial work. Injecting of cement is performed in the cracked formation for sealing the leak; it uses the dehydration process, which requires injecting of cement slurry into the problem area [11]. However, the success rate in the earlier attempts was relatively low and involved several squeeze job before a successful seal obtained; the cost of work-over job went up as high as USD100,000 per well [4]. It has become a known fact that the volumetric shrinkage of the cement during hydration contributes substantially to the existing problems of the well cementing system. Such shrinkage results may result in an increase in the porosity and permeability of the hardened cement slurry or the formation of micro-annuli by contraction of the external dimensions of the cement or both of them. The resulting flow channels may offer mobilization possibilities within the annulus, especially for gas. If the cement has an ability to expand after it gets harden slightly, that provides an opportunity for sealing the small gap or "micro-annulus" and improving the primary cementing results [5,8,9]. Expansion admixtures are usually added into the cement blend to produce an expandable element in cement, which can be measured in the form of linear expansion [12].

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Portland cement class-G is generally used for for-well cementing, for which a necessary requirement is that it should be very low permeable. When the Portland cement is used in large volume such as for oil-well cementing, drying shrinkage may be a point of major concern for the integrity of the system. The drying shrinkage, as well as the brittle nature of Portland cement, increases the risk of cracking and hence the permeability of the system may be increased. Ultimately this type of deficiency of the system may cause the sealing failure. Therefore, to induce the ability of expansion in the system by adding some admixtures is considered one of the solutions for avoiding the sealing of the oil-well tubing system.

Conventional geopolymer binders or, in other words, alkali-activated materials (AAM) are considered as a potential alternative to ordinary Portland cement (OPC). It is important to note that AAM offers an opportunity of converting a variety of waste streams into valuable byproducts [13]. Existing research in the Australian database focusing on conventional geopolymer concrete mixes showed that such types of concretes have a potential for a 44–64% reduction in greenhouse gas emissions. However, in the latest research on geopolymer binders, the focus is shifting towards the one-part of geopolymers. Whereas, conventional geopolymers are considered as potential alternatives to OPC for limiting CO_2 emissions [14]. Nour et al. (2018) synthesized a geopolymer binder by activating blast furnace steel slag (GGBFS) using two parts consisting of NaOH and Na_2SiO_3 [15]. However, some serious concerns with the use of the alkali activation process are highlighted, which are corrosive, viscous, and, as such, difficult to handle and not user-friendly. Therefore, the current development of the one-part geopolymer binders is expected to bring more significant potentials than the conventional geopolymers. It is anticipated that the one-part geopolymers would be more beneficial for cast-in-situ applications. Because one-part geopolymers are based on dry mixing process that consists of a solid aluminosilicate precursor, a solid alkali source, and possible admixtures to which water is added, similar to the preparation of OPC concrete [14]. In some studies focusing on one part of geopolymer binders, researchers have used rice husk ash and red mud as the precursors and synthesized them using sodium aluminate solution [16, 17].

Because the geopolymer systems (particularly one part) are receiving much interest by researchers as the futuristic binding materials in many disciplines, which may result in a low carbon footprint, with growing concerns for achieving sustainability in every discipline, therefore materials causing high emission to the environment and consume a massive amount of natural resources are being criticized. In view of this, futuristic binding materials will be designed in such a way that they can reduce the emission level and consume a low amount of natural resources. Portland Cement is considered one of the unsustainable materials. Therefore, researchers are focusing on alternative materials for the future, such as geopolymers.

Therefore, oil and gas cementing experts are also focusing on exploring the potential of geopolymer system in enhancing the excellent integrity and durability. In some earlier research on geopolymer cementing (GPC) system, it has been explored that GPC exhibits encouraging results on rheological properties and compressive strength, which are referred to in this paper. Therefore, the principal aim of this study was to assess the ability of geopolymer cement to expand in water. The expansive properties were compared with the expansive properties of Class-G cement. In the case of Class G cement, a suitable expandable material was added into the cement blend at a different concentration, and the ability of it to expand was evaluated. The expansion test was conducted according to the procedure stated by the API RP 10B-5/ISO 10426–5:2003 Recommended Practice on Determination of Shrinkage and Expansion of Well Cement Formulation at Atmospheric Pressure [6]. The linear expansion of cement is calculated using Eq. (1)

Where; L_f is the final length in (mm), and L_I is the initial length (mm), and0.358 is the constant calculated considering an inner diameter of 88.9mm for the outer expansion ring. The annular expansion cell or mold method measures the linear bulk expansion or cement. The percentage of linear bulk expansion indicates the changes in the external volume or dimensions of a cement sample. The magnitude of expansion depends on the amount of expandable material used, type of cement, slurry design, and also curing condition.

2. Experimental program

2.1. Materials and properties

2.1.1. Fly ash

Low calcium fly ash, type F, was used as a primary binder material or precursor, which was acquired from Manjung power station, Malaysia. Table 1 shows the chemical composition of the supplied material that was obtained using the XRF technique.

2.1.2. Alkaline solution

Sodium hydroxide (8 Molar), NaOH, was used as an alkaline activator together with Sodium silicate, Na_2SiO_3. In this study, the Na_2SiO_3/NaOH ratio of 2 was used.

2.1.3. Expandable material

Two types of expandable materials were used in this study; Magnesium Oxide (MgO) based and Elastomeric based. Cement slurry blended with MgO based are labeled as E, and those with elastomeric based are labeled as R.

2.1.4. Curing medium

Tap water and oil were used to cure cement at 60 °C for the selected test duration.

2.2. Apparatus

2.2.1. Expansion cell

Figure 1 shows the image of an expansion cell used to measure the linear expansion of cement according to the requirements of API RP10B-5.

2.2.2. Water bath

In this experimental program,a water bath was used to cure the cement with water or oil at 60 °Ctemperature and atmospheric pressure.

2.3. Sample preparation

2.3.1. Mixing

Geopolymer cement slurry was prepared by mixing the solid blend (fly ash) with an alkaline activator, whereas Class G cement was mixed with distilled water to prepare the slurry. Table 2 shows the slurry design

Table 1. Oxide composition of fly ash (determined using X-Ray Fluorescence Method).

Element	Weight (%)
SiO_2	46.47
Al_2O_3	25.95
TiO_2	1.16
Fe_2O+	8.31
CaO	6.88
MgO	4.95
Na_2O	1.72
K_2O	2.11
SO_3	0.63
Cl	<0.1
Moisture	0.11
Loss of ignition	1.61

Figure 1. Expansion cell as per API RP 10B5.

Table 2. Blend formulations.

Designation	FA Content [Cement] (%)	Alkaline [water] (%)	Expandable Material (%)	Density (kg/m^3)
GP15	60	40	0	1798
G15	[60]	[40]	0	1798
GP13	40	60	0	1642
G13	[60]	[40]	0	1642
GP13E1	40	60	5	1642
G13E1	[60]	[40]	5	1642
GP15E1	60	40	5	1798
GP15E2	60	40	8	1798
G15E1	[70]	[30]	5	1798
GP15R1	60	40	5	1798
GP15R2	60	40	10	1798
GP15R3	60	40	25	1798
GP15R4	60	40	30	1798

NOTE: Expandable material is measured by weight of fly ash (FA) content.

used in this study. The cement slurries were mixed at 4000 rpm, and 12,000 rpm is conforming to the requirements of API RP10B-2 [7].

2.4. Cement slurry conditioning

After mixing, the cement slurry was immediately poured into the slurry cup of an atmospheric consistometer and heated to 60 °C for 30 min according to the requirements of API RP10B-2 and API RPB10-5 requirement.

2.5. Compressive strength test

When the compressive load on a cement sample subjected to a uni-axial testing mode crushes or breaks-down, the load is referred to as the ultimate load. The stress value at the ultimate load is called the unconfined compressive strength (UCS). It is defined as the ability of the cement slurry to resist compression. For this experimental program, the testing method followed ASTM D 7012-04.

2.6. Curing at atmospheric pressure

After conditioning for 30 min, the cement slurry was immediately poured into the expansion cells through the large fill hole provided at the outer portion of the ring mold. The mold was filled when the cement slurry passed the small hole. The expansion cell was appropriately labeled according to the designation defined in Table 2; the initial measurement of expansion was taken by measuring the distance (mm) between two steel balls and recorded as L_i (initial length).

After initial measurement, the expansion cells were placed into the water bath, which was preheated at 60 °C, they were cured by the number of days as specified. After completion of curing days, the expansion cell was removed from the water bath, and the measurement of cement expansion was taken immediately (within 5 min) by measuring the distance of two steel balls that was recorded as L_f (final length). The linear expansion was calculated according to Eq. (1) as below:

$$\text{Linear expansion } (\%) = (L_f - L_i) \times 0.358 \quad (1)$$

3. Results and discussion

3.1. Compressive strength test results

Table 3 shows the unconfined compressive strength, UCS test results of all slurry samples prepared at the different density, and containing different expandable materials. The control mix, GP15 possessing the density of 15 lb/gal showed the highest UCS at all ages. GP15 achieved a strength of 725 psi after 24 h curing, and after 14 days, the strength was enhanced to 2.86 times, whereas at 60 days, it was increased to 3.54 times. The slurry samples possessing a density of 13.7 lb/gal, the strength was reduced to about 45% after 24 h with respect to the 1-day strength of GP15. The addition of E type expandable material caused a reduction in strength up to 18% as compared to the 1-day strength of GP15 samples. Whereas R type expandable material caused positive effects in the gaining of compressive strength, which resulted in an increase of 20% in the one day strength when the content was used as 5% and 10%. With a 30% addition of R type mixture, it caused a reduction of 52% in the one day strength.

3.2. Expansion of paste

The expansion tests on the slurry samples were conducted at 60 °C temperature and atmospheric pressure for a curing period ranging from 1 day up to 18–20 days and, in some cases, up to 40 days. This section

Table 3. Unconfined compressive strength test results.

Mix Type	FA (%)	EM (%)	Density (kg/m^3)	Compressive Strength (MPa)			
				Age (Days)			
				1	14	30	60
GP15	60	0	1798	5.0	14.3	16.3	17.7
GP13	40	0	1642	2.8	6.2	7.8	11.0
GP13E1	40	5	1642	2.7	5.3	6.8	7.8
GP15E1	60	5	1798	4.8	7.1	8.0	10.2
GP15E2	60	8	1798	4.1	6.2	7.2	9.6
GP15R1	60	5	1798	6.0	11.1	14.3	15.0
GP15R2	60	10	1798	6.0	9.2	10.2	10.5
GP15R3	60	25	1798	5.1	8.4	8.5	10.3
GP15R4	60	30	1798	2.4	7.5	8.6	10.3

discusses the experimental results of the measurement of the linear expansion of geopolymer samples bearing different density and compared the results with the linear expansion measurements of the Class-G samples. The results are plotted in the graphical for different densities. Figure 2 shows the linear expansion results of samples bearing a density value of 15 lb/Gal (1798 kg/m^3); the samples did not include any additive in the mix to accelerate the expansion process.

Geopolymer cement shows some expansion on the first day of curing, which is around 0.05%, while G cement showed very little expansion (0.02%). After 18 days curing at 60 °C, the percentage of linear expansion obtained by 15 Ib/gal (1798 kg/m^3), Geopolymer cement attained 0.15% expansion as compared to the Class-G cement that expanded up to 0.06%, which the expansion that geopolymer achieved after one day of curing. Test results show that a geopolymer cement system has the ability to expand by itself when exposing to water without the addition of any kind of expandable material. The geopolymer binders acted like an expansive cement, which usually shows higher drying shrinkage, but with the exposure to water, it expands. For comparing the results of geopolymer and Class-G cement, it is clear that since an early age (after one day curing period), geopolymer samples showed higher expansion than the Class-G samples. The geopolymer samples expanded almost 2.5 times higher than the Class-G samples almost at all curing ages. Such results indicated that a geopolymer has a better potential of expansion than the Class-G cement that would be an added value of geopolymer binders from an application point of view [10].

Similarly, the linear expansion test was conducted on low density (13.7 lb/Gal) samples under the same curing conditions and curing age. In the preparation of these slurry mixes again, no chemical admixture was added. Both the GP13 and G13 samples were cured until 18 days of curing, and measurements were made with an interval of one day. Such results are shown in Figure 3 below, geopolymer and Class-G cement samples exhibited the same behavior as that was discussed with 15lb/Gal density samples. Geopolymer cement samples showed almost 0.1% linear expansion after 24 h of curing and reach 0.21% of linear expansion after

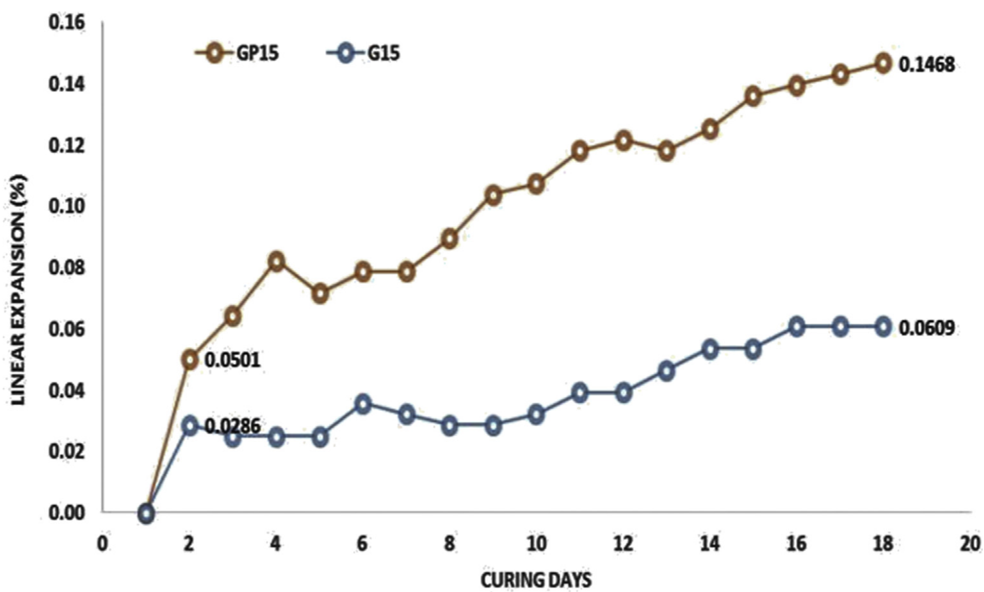

Figure 2. Measured Linear Expansion (%) vs. curing age (days) of 15 Ib/Gal Geopolymer and Class G Cement samples cured at 60 °C.

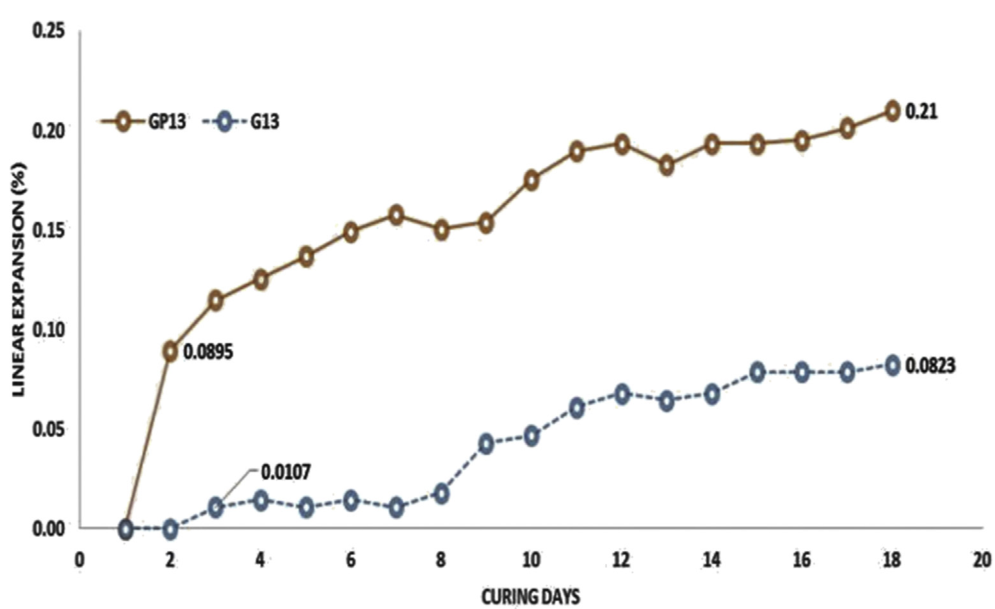

Figure 3. Measured Linear Expansion (%) vs. curing age (days) of 13.7 Ib/Gal Geopolymer Cement (GP13) and G Cement (G13) Cured at 60 °C.

18 days of curing. G cement system expands very slow on the first ten days of curing, which is around 0.04% and reaches 0.08% after 19 days. The value of Geopolymer cement expansion is almost double the value of expansion provided by G cement.

In the second phase of the experimental program, commercially available admixture called expandable material was added into both types of cement slurry; Class-G and geopolymer, the samples were cured at the same conditions, i.e., 60 °C up to the desired age of curing. The purpose of adding was to investigate the effect of admixture on accelerating the expansion process. Figure 4 shows the results of 13.7 ppg samples containing 5% dosage of the expandable material.

The results showed that the addition of a dosage of 5% by weight of Fly Ash (BWOFA) of expandable material increased the value of linear expansion of both the groups of cement slurry, GP, and Class-G. It can be noticed that after 24 h of curing, Class-G cement showed a bit higher expansion (0.11%) than the 13GP samples that achieved 0.09% expansion. From this observation, it can be concluded that the Class-G cement tends to exhibit significantly better initial (early age, i.e., one day) expansion with the addition of expanding material compared as compared to that shown without any addition of the expanding material. Although at an early age (two days of curing), Class-G cement showed better results than the GP samples, however, after the next interval (4 days) until 18 days of curing GP samples showed much better expansion than the Class-G samples. At the age of 18 days of curing, the expansion of the geopolymer was achieved as 0.38% as compared to that of Class-G cement that only achieved 0.24%. After curing to another five days (total 23 days of curing), expansion in GP samples increased to 0.4%, which was the final observation of this group. Referring to the results presented in Figures 3 and 4, it can be generalized that geopolymer binders are compatible and capable with the addition of the same family of expandable materials that are commercially available for use in the Class-G cement. The measured linear expansion of the geopolymer cement system (density of 13.7 Ib/gal) was obtained as 0.21% (without any dosage of admixture), which was increased to 0.38% with the addition of 5% expandable material after 18 days curing at 60 °C.

Similarly, Figure 5 presents the comparison of results of the linear expansion of samples made of the possing slurry density of 15 Ib/gal. In this case, the expandable material dosage was used as 5% and 8% by weight of fly ash content. In this case, when a dosage of 5% of expandable material was, 15 ppgGP samples linearly expanded to 0.15% just after 24 h curing at 60 °C, similarly, when a dosage of 8% was mixed in Class-G cement samples that cured for 24 h at 60 °C, it achieved only 0.018% linear expansion. However, Class-G cement experienced a gradual increase in achieving linear expansion with the extended number of days of curing, after 18 days of curing such samples achieved 0.12%, which is lower than that the 15GP samples achieved only after 24 h.

When a high dosage of expandable material, i.e., 8% was added to 15ppg GP samples, the achievement of linear expansion of such samples increased the higher value that was obtained as 0.18% after 18 days curing at 60 °C, which was approximately 29% higher than that was obtained with a dosage of 5% expandable material. However, the final percentage of linear expansion with and without the expandable material is almost similar indicated that the MgO based expansion material helps on early expansion development but provides a little effect on improving the Geopolymer cement expansion at this concentration. It was observed that in Geopolymer cement, a higher percentage of linear expansion obtained on the first few days of curing and start to reach its plateau although cement exposed to water for a more extended curing period.

Linear expansion tests were also performed GP and Class-G cement samples using another type (elastomer-based) of expandable material. For this type of material, dosage concentration is stated in Table 2. For this set of samples, the same curing condition was maintained; up to 40 days of curing in a water bath at 60 °C and atmospheric pressure. Figure 6 shows the values of linear expansion measured at different curing days for different samples.

In this set of slurry samples, only a geopolymer binder was used that contained four different dosages 5%, 10%, 25%, and 30% of the expandable material. When a dosage of 10, 25, and 30% was used, it can be observed that the samples underwent a maximum amount of linear expansion at an early age (3 days of curing). After that, there was very little expansion achieved, as shown in Figure 6. On another note, the dosage concentration has a significant effect on the linear expansion of the samples. With a 5% dosage at the age of 3 days, the samples achieved 0.0304% expansion, whereas, with a 10% dosage of expandable material, the expansion was measured as 0.2882%, which about nine times that obtained with 5% dosage. Similarly, with 25% or 30% dosage, after three days curing, the expansion was obtained as 0.766%, which was further increased to about 1% after 40 days curing. On another note, it is observed that when the dosage was increased from 25% to 30%, there wasn't any noticeable increase in the linear expansion of the samples obtained.

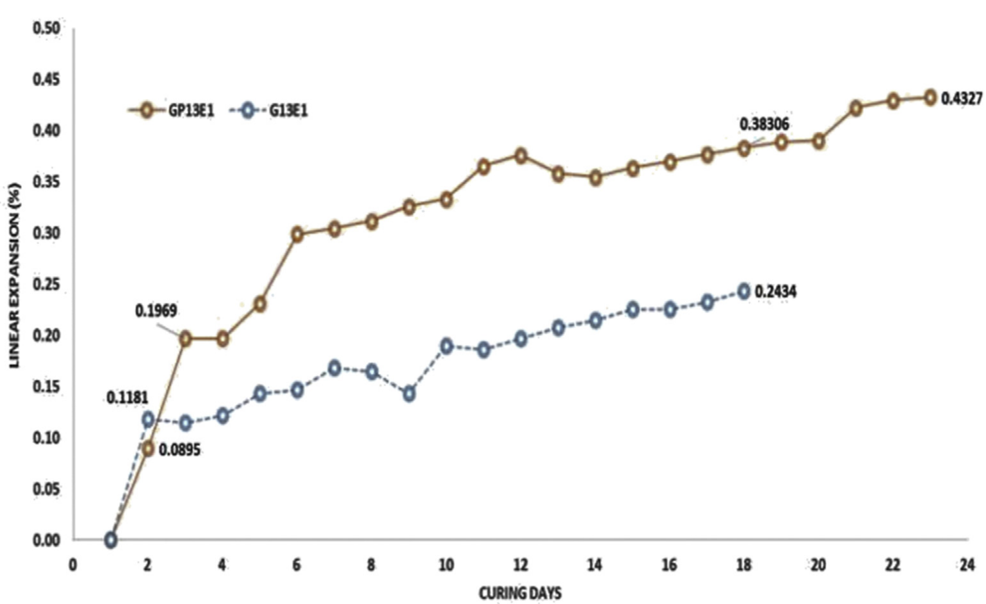

Figure 4. Measured Linear Expansion (%) vs. curing age (days) of 13.7 Ib/Gal Geopolymer Cement (GP13E1) and G Cement (G13E1) Cured at 60 °C with 5% Expandable Material.

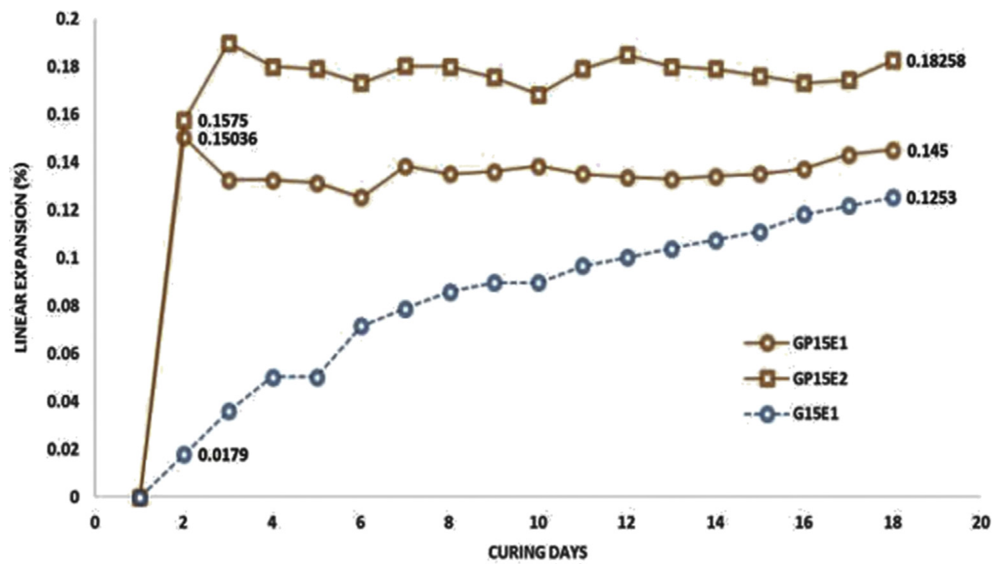

Figure 5. Measured Linear Expansion (%) vs. curing age (days) of 15 Ib/gal Class G Cement and Geopolymer Cement with Addition of 5% and 8% BWOFA of Expandable Material Cured at 60 °C.

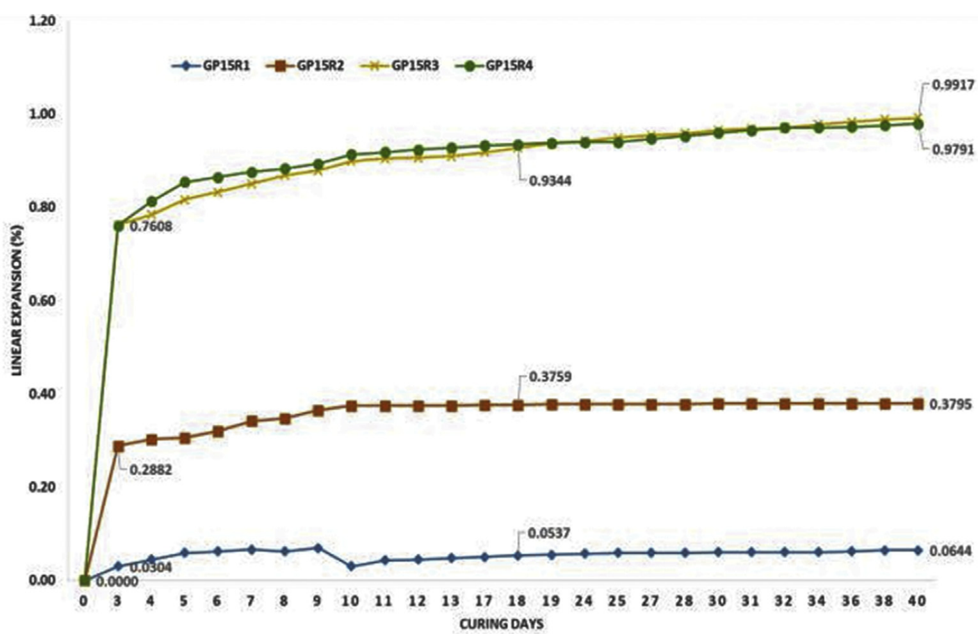

Figure 6. Measured Linear Expansion (%) vs. curing age (days)of 15 Ib/Gal Geopolymer Cement Blended with Different Percentage of Expandable Material Cured at 60 °C.

By results of the linear expansion of GP samples with different dosages of elastomer-based material, it can be generalized that such type of admixture shows better performance in enhancing the linear expansion of cement when the dosage concentrations kept more than 10%. However, a 25% dosage may be classified as the upper threshold value because when a dosage of 30% was added, a minimal increase in the linear expansion was observed concerning that measured with 25% dosage samples.

4. Conclusion

- Based on results and discussion of the two sets of cement slurry, geopolymer, and Class-G, it is concluded that geopolymer cement has a reasonable tendency to expand without the mixing of an expansive material.
- It was also concluded that the maximum amount of linear expansion of samples (both GP and Class-G) was obtained after early days of curing (i.e., three days) after that minimum increment is observed (up to 18 or 40 days of curing), the plot shape is observed as a plateau.
- The addition of MgO based expandable material help on early expansion development in Geopolymer cement, but it was not very helpful to improve the overall expansion (at the end of curing age),-however, higher than 8% may give better expansion. The elastomer-based material improved both on overall expansion and also early expansion development.
- When the density of the slurry was maintained at 15 lb/gal, it exhibited the highest UCS at all ages. 5% addition of R type expandable material content is found as the optimum addition that caused an increase of 28% in the one dau UCS of the control mix

Declarations

Author contribution statement

Siti Humairah Bt Abd Rahman: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Sonny Irawan: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

Nasir Shafiq: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Raja Rajeswary: Performed the experiments; Analyzed and interpreted the data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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