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Effect of Sodium Gluconate on the Fluidity and Early Hydration of Cement Pastes Along with Polycarboxylate Ether

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Abstract: The main objective of this paper is to provide a better understanding of the effect of sodium gluconate (SG) along with Polycarboxylate Ether (PCE) on ordinary Portland cement (OPC) hydration behavior. The Pastes performances of ordinary portland cement, including mechanical strength, fluidity, the heat of hydration, Fourier Transform Infrared Spectroscopy (FT-IR), and thermal analysis (TGA-DTA) are studied. The dispersion behaviour of Cement paste in the PCE with varying percentages of retarder such as Sodium Gluconate SG is presented in this paper. The correlation was aimed at quantifying the impact of the varying percentage of SG & constant percentage of PCE in the Cement paste. The instrument flow cone, Compressive testing machine & Isothermal Calorimeter were employed in the present study for evaluating the behavior of the Cement paste, which is found to show systematic changes in fluidity, compressive strength (CST), and Heat of hydration successfully. The workability & compressive strength of Cement paste improved by the addition of SG in the PCE up to certain a percentage.

Keywords: Cement paste; fluidity; Isothermal Calorimeter; Polycarboxylate ether (PCE); Retarder.

I. INTRODUCTION

All over the world for construction purposes concrete is widely used. The cement paste is the main ingredient of the concrete which causes it to harden. For new generation concrete, the properties such as workability and strength are required. High strength and high-performance concrete can be achieved by reducing the water-cement ratio (w/c) [1-3]. The Workability of concrete has a direct impact on the reduction of the water-cement ratio.

The desired workability of concrete at a low water-cement ratio is achieved by using PCE because it helps with the dispersion of Cement particles. The working mechanism of PCE in concrete is to disperse the flocculated cement grains and to release entrapped water by creating a repulsive force between them [4-6]. The Cement content major five compounds are Tricalcium Silicate, Dicalcium Silicate, Aluminate, Ferrite & Calcium Sulphate hemi or dihydrate [7-9,18]. Cement hydration is the exothermic reaction that takes place in five steps (Young 1985) such as (a) Rapid Initial Process, (b) Induction Period, (c) Acceleration period, (d) Retardation period & (e) Long-term reactions. In the Isothermal calorimeter, we can study the b, c & d steps [10-11].

The primary objective of this study is to understand the behavior of high-strength cement paste (HSCP) at constant PCE dosages with varying the percentage of SG because in the current market PCE is used along with SG for retention purposes. If the percentage of SG is more in PCE then it will affect fresh as well as harden properties. The addition of PCE to concrete delays cement hydration due to the steric hindrance mechanism, also it shifts the main hydration peak of tricalcium silicate [12-13]. According to Santos et al. when SG is dissolved in the water it forms a Gluconate ion, which forms complexes with Ca^{2+} ions present in the Cement causing delayed setting of cement hydration [14]. The SG as a retarder is used along with PCE for workability with retention. If the percentage of SG is more than 0.1% then it has a negative effect on the dispersion ability of PCE [15-17]. Shen et al. [19] proposed that when superplasticizer is added along with SG improves dispersion and the principle peak of hydration shifted to the right side and there is a lower in the hydration peak. Tan et al. [20] suggest that in the SG-PCE system when the dosage of SG is less than 0.10% then more dispersion and dosages of SG is more than 0.10% resulting a negative impact on the dispersion due to the competitive adsorption effect. Ma et al. [21] proposed that SG improves cement fluidity and improves 3- and 28-day compressive strength. SG less than 0.03% promotes ettringite (Aft) formation at an early age and inhibits Aft formation when the dosage was more than 0.05%. Fourier transform infrared spectroscopy and thermal analysis are commonly used to investigate the formed hydrated products in the cement paste [22-25].

The previous studies discussed the fluidity and heat of hydration of cement paste with PCE system or SG system only. The combine effect of different dosages of SG on the PCE is less investigated. This paper gives better understanding of the effect SG system, PCE system and PCE-SG system on the Cement hydration. The effect of PCE-SG system on fluidity and mechanical strength is studied. The effect of PCE-SG system on cement hydration behaviour are investigated by the means of Isothermal Calorimeter, FTIR and TGA-DTA. Only early cement hydration i.e., 24 hours data studied in the FTIR & TGA-DTA for the better understanding.

II. EXPERIMENTAL

A. Materials

1) Cement

The Cement used was ordinary Portland cement (like ASTM – Type-I) confirming IS 12269 (OPC 53). Physical and chemical composition is described in Table 1 and Table 2 respectively. The Physical properties of Cement such as density and specific surface are measured as per IS 4031-Part 11 and IS 4031-Part 2 respectively. The main chemical composition of cement was determined by X-ray fluorescence (XRF).

2) Polycarboxylate Ether Pure Polymer (PCE)

Polycarboxylate Ether (commercially available) High range water reducer (HRWR) is used. The physical properties of PCE were evaluated using state of the art instrumentation available at the Research and Development Centre of Master Builders Solutions India Pvt. Ltd., Navi Mumbai. These properties are presented in Table 3.

3) Sodium Gluconate

It is a solid crystalline powder with 99% purity which is available commercially. The molecular formula is $C_6H_{11}NaO_7$, and the structural formula is given in Figure 3.

TABLE 1

Physical properties of the OPC used in the study

Compound	Density (g/cm³)	Specific Surface (M²/kg)
OPC	3.15	328

TABLE 2

Chemical compositions of the OPC used (wt. %)

Compound	SiO_2	$A1_2O_3$	Fe_2O_3	CaO	MgO	SO_3	Na_2O
OPC	20.39	5.64	5.18	61.30	1.07	2.61	0.25

TABLE 3

Properties of super-plasticizer used.

Type of super-plasticizer	PCE
Aspect	Pale yellow
Relative density at 25°C	1.10 to 1.12 at 25°C
Solids Content	50.0 to 52.0
pH	>6
Solubility in Water	Readily Soluble in Water

TABLE 4
Mix proportions for laboratory mortar trials

Mix. No.	OPC	PCE	w/c	SG	Flow mm
UoM	%	%	%	%	mm
B 01	100% OPC	0.00	0.29	0.0	60
P 01	100% OPC	0.11	0.29	0.0	140
SG 01	100% OPC	0.00	0.29	0.25	81
SG 02	100% OPC	0.00	0.29	0.125	65
SG 03	100% OPC	0.00	0.29	0.1	68
SG 04	100% OPC	0.00	0.29	0.05	65
SG 05	100% OPC	0.00	0.29	0.025	60
SG 06	100% OPC	0.00	0.29	0.0125	62
SG 07	100% OPC	0.00	0.29	0.00625	55

B. Experimental Methods

1) Physical and Rheological Properties

The fluidity test was conducted by using a flow cone test for all 16 different combinations of cement paste samples to find the fluidity behavior using PCE & SG. at Room temperature 25^0C. & at a constant water-cement ratio (w/c) of 0.29.

The flow cone is a simple device for measuring workability from flow spread. The flow cone is 60 mm in height with Top and bottom diameters being 36 and 60 mm respectively as shown in Figure 1. The CTM machine is used to measure compressive strength is having capacity of 1000 KN. The sample for investigating the performance of compressive strength was prepared using specimens the size of 50 mm x 50 mm x 50 mm. In this study, cement paste was prepared with a combination of different percentage dosages of SG (Table 4 & 5) by cement mass and at a constant 0.11% PCE by cement mass. Mixing was immediately started after the addition of water and continued for 90 seconds. Paste scaling on the vessel walls was scrapped off, and mixing was continued for another 90 seconds, then the cement paste is poured into the cup and lifted for fluidity test & is made to spread for 30 sec and fluidity is measured.

TABLE 5
Mix proportions for laboratory mortar trials

Mix. No.	OPC	% PCE	w/c	% SG	Flow mm
UoM	Gm		%		
B 01	100% OPC	0.00	0.29	0.0	60
P 01	100% OPC	0.11	0.29	0.0	140
PSG 01	100% OPC	0.11	0.29	0.25	142
PSG 02	100% OPC	0.11	0.29	0.125	190
PSG 03	100% OPC	0.11	0.29	0.1	205
PSG 04	100% OPC	0.11	0.29	0.05	213
PSG 05	100% OPC	0.11	0.29	0.025	148
PSG 06	100% OPC	0.11	0.29	0.0125	140
PSG 07	100% OPC	0.11	0.29	0.00625	135

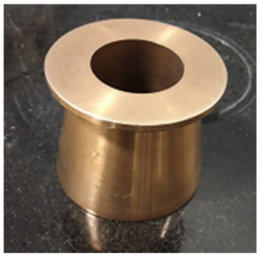
Figure 1: Flow Cone


Figure 2: Hobart Mixer

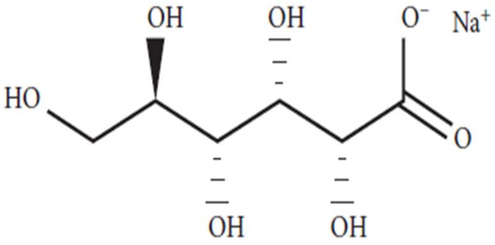

Figure 3: Structural formula of SG.

2) Hydration Heat Flow

The TAM AIR instrument is used to carry out thermodynamic experiments such as cement and concrete hydration. The Heat of hydration of Cement can be studied in a non-destructive manner using a minuscule quantity of cement. The sample is poured into an ampoule that is in contact with a heat sensor, which in turn is connected to a heat sink. A temperature gradient is developed across the sensor when the heat is produced or consumed by the process. This generates a voltage that is proportional to the heat flow. Each sample has a standard reference that is in contact with a parallel heat flow sensor. This is done to ensure that any deviations from standard conditions will influence both sample and reference sensor equally. This facilitates an accurate determination of the heat exclusively associated with the sample. The heat of hydration is measured at 24 $\pm 1^0$C & same is recorded in the computer up to 72 hrs.

3) FT-IR

Infrared spectroscopy reflects changes in the vibrational energy of the molecule, and qualitative analysis of the substances in the cement pastes can be performed according to the position and shape of the absorption peak. The FTIR spectra were recorded by using taken a Perkin - Elmer 1710 spectrophotometer. The analysis was carried out in the frequency of range of 500 – 4000 cm^{-1} with 2.0 cm^{-1} resolution.

4) Thermal Analysis

The thermal analysis by TGA was performed using a Rigaku Thermo Plus TG/DTA 8120 machine. The sample was heated from 20 ^{0}C to 900 ^{0}C with a heating rate of 10 ^{0}C/min. The Nitrogen gas was continuously supplied throughout the test.

III. RESULTS AND DISCUSSION

A. Fluidity

The influence of PCE on the fluidity of cement pastes with and without the addition of SG are discussed in this part, and the results are given in Figure 4 and 5. The fluidity test of cement paste carried out at seven different percentages of SG keeping constant dosage of PCE. The 100% OPC mix was used at 0.29 constant w/c for this study. From figures 4 and 5, it has been observed that, fluidity parameter measure with flow cone indicated the effect of SG on the dispersion of Cement paste. From Figure 5, it seems that the dispersion of cement paste is reduced at 0.25% of SG with 0.11% PCE. When fluidity is measured by keeping a constant percentage dosage of PCE (0.11%) with varying percentages of SG such as 0.125%, 0.1% & 0.05% dosages of SG then fluidity increases respectively. The fluidity of cement paste reduced at 0.025%, 0.0125% & 0.00625% dosages of SG at 0.11% of PCE respectively. From Figures 4 and 5, it seems that when the PCE is added along with the SG then it helped to improve the fluidity of cement paste. The PCE(0.11%)-SG(0.05%) i.e., PSG 04 system has maximum fluidity.

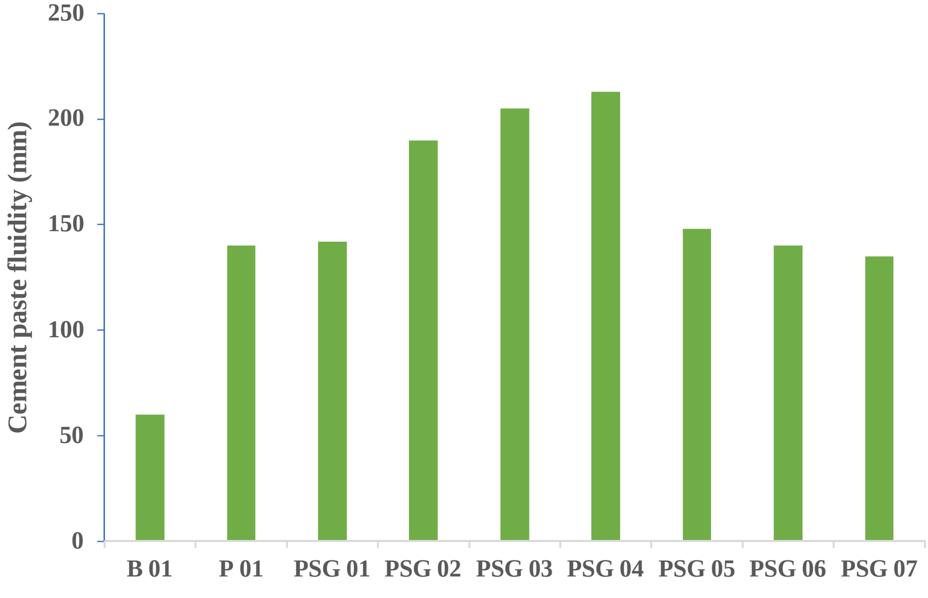

Figure 4: Influence of PCE with the addition SG on cement paste fluidity.

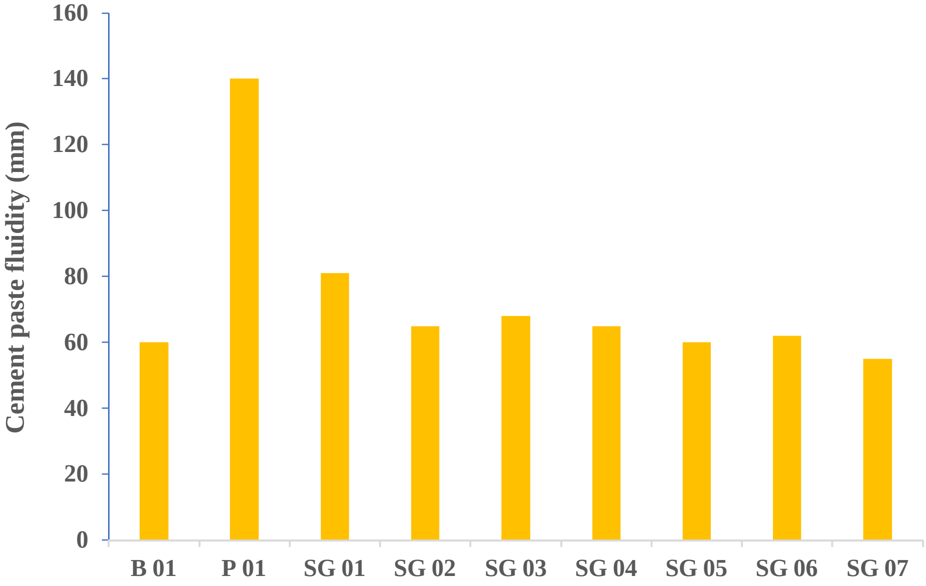

Figure 5: Influence of SG without addition of PCE on the cement paste fluidity.

B. Compressive Strength

The PSG04 system i.e., SG(0.05%) added to PCE(0.11%) has given more fluidity as compared to all samples, hence B01, P01, PSG04, and SG04 systems are further considered for the 01, 03, 07, & 28 days. The compressive strength for the above-mentioned samples is given in Figure 6. It was found from Figure 4 that, the B01, P01, PSG04 and SG04 samples have obtained 01-day CST is 19.61 MPa, 21.22 MPa, 18.52 MPa, and 17.11 MPa respectively. The 01-day CST of PCE added with 0.11% (P01) dosage is highest but at 0.05% dosage of SG (SG04) given lowest CST even less than Blank sample. When PCE(0.11%) is added along with 0.05% of SG (PSG04) given more 01-day CST than SG04, that means optimum dosage SG when added in the PCE gives better 01-day CST. The 03-day CST obtained for the samples B01, P01, PSG04, and SG04 is 38.68 MPa, 43.34 MPa, 42.56 MPa, and 32.64 MPa respectively (Figure 06). The 03 days CST of the P01 and PSG 04 sample is more than the blank sample, but for SG04 sample the 03-days CST even less than a blank sample. The 07-days CST obtained for the samples B01, P01, PSG04 and SG04 is 54.04 MPa, 64.56 MPa, 68.51 MPa, and 56.92 MPa respectively. Also 28-days CST for B01, P01, PSG04 and SG04 is 82.05 MPa, 89.98 MPa, 91.96 MPa, and 87.23 MPa respectively (Figure 06). From Fig. 6 it seems that PSG 04 system gives better lateral CST such as 07 & 28 days as compere to B01, P01, & SG 04.

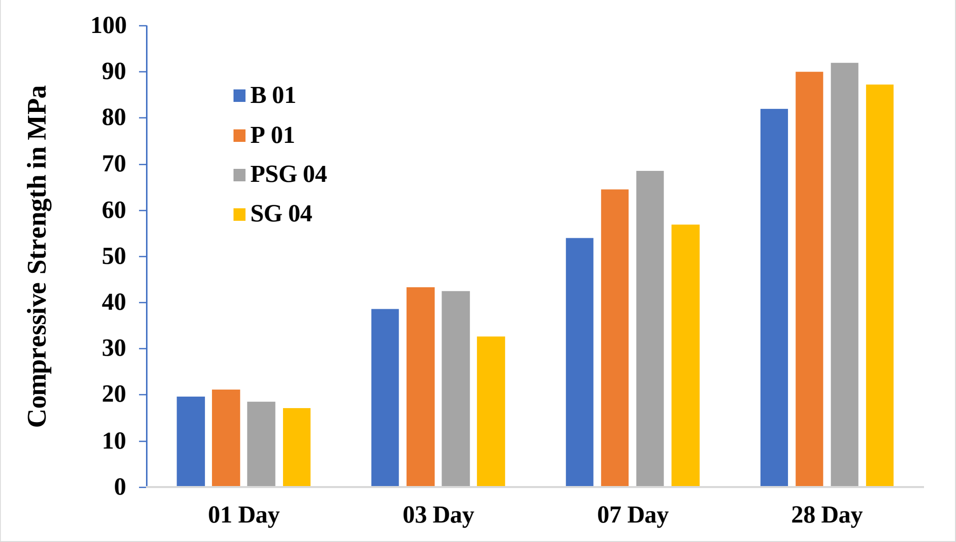

Figure 6: Compressive strength at different percentage of SG with 0.11% PCE.

C. Hydration Heat Analysis

1) Effect of PCE-SG and SG system on the Heat of Hydration of cement paste:

The curves for hydration heat evolution rate of cement paste for B01, P01, PSG04 and SG04 samples are shown in Figure 7. From Figure 7, it is obtained that for B01, P01, PSG04 and SG04 samples the maximum heat flow is 0.00225 W/g, 0.00233 W/g, 0.00208 W/g and 0.00172 W/g. The P01 produced 3.62% of more heat flow as compere to B01. The maximum heat evolution peak occurrence time of sample P 01, PSG 04 and SG 04 are delayed by 1.50, 5.92 and 8.41 hrs. respectively as compere to blank sample (B 01). In addition, the evolution rate peak values of samples PSG 04 and SG 04 are 7.71% and 23.61% lower than the blank samples respectively. the value of heat evolution rate peal of sample P 01 is more than that of blank sample. The heat evolved by P 01 sample is 3.62% more as well as its main principle heat curve is shifted to right side as compere to blank sample.

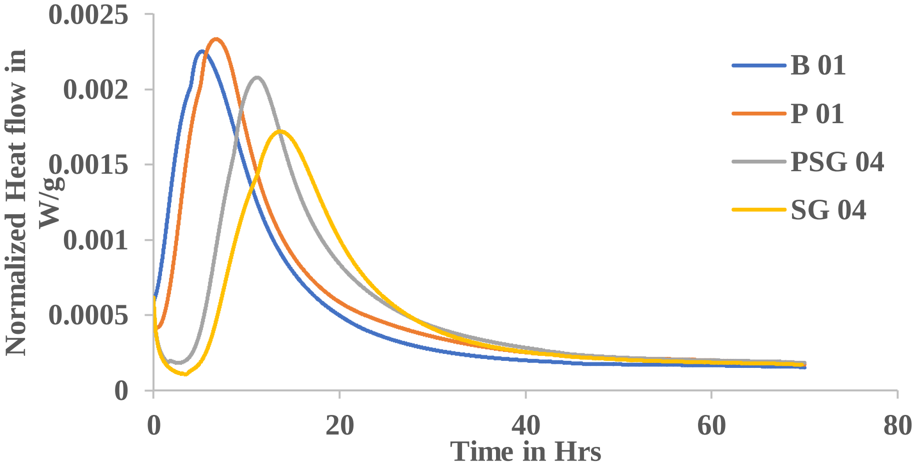

Figure 7: Normalized Heat flow curves of cement pastes.

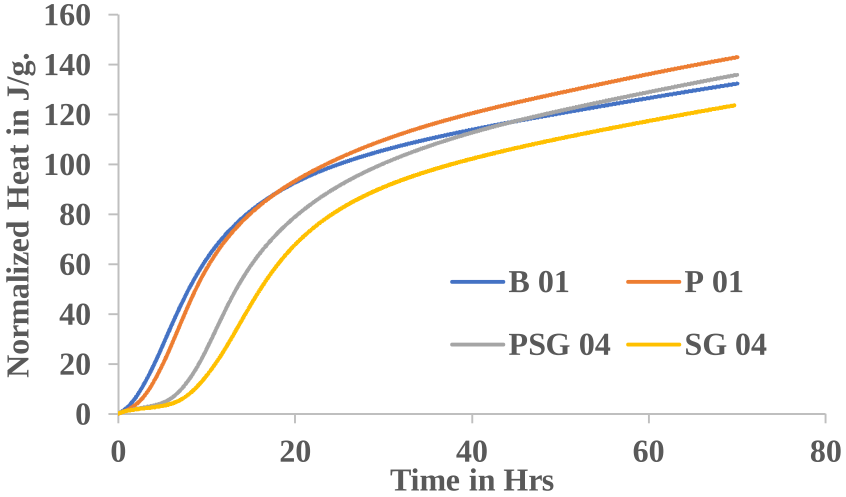

Figure 8: Normalized Heat curves of cement pastes.

From Figure 8, cumulative heat at 24 hours produced for B01, P01, PSG04 and SG04 is 98.91 W/g, 100.96 W/g, 89.42 W/g and 79.66 W/g respectively. The cumulative heat for the sample P01 is increased by 2.07% as compere to B01 sample. The cumulative heat for PSG04 and SG04 is reduced by 9.59% and 19.46% as compere to B01 sample. From Figure 8, cumulative heat at 70 hours produced for B01, P01, PSG04 and SG04 is 132.45 W/g, 143.04 W/g, 136.00 W/g and 123.73 W/g respectively. The samples PSG 04 and SG 04, the cumulative heat is reduced 2.68 and 6.58% respectively as compere to blank sample (B 01) at 70 hrs. The hydration cumulative heat for P 01 is 7.99% more than that of blank at 70 hrs.

2) Significance Or Relationship Between Compressive Strength And Cumullative Heat

The Statistical modelling is carried out using multiple linear regression to establish a relation between the 01- & 03-days CST versus cumulative heat. The result indicates that both 1- and 3-days CST is a unique function of the cumulative heat.

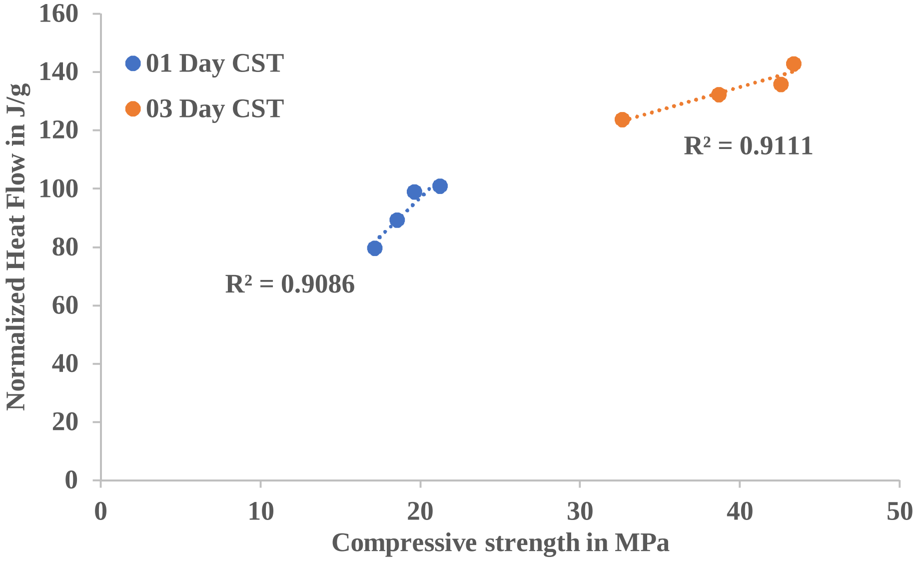

Figure 9: Relation between Compressive strength and Normalized Heat Flow J/g.

D. FTIR Investigation

The FTIR spectra were recorded for 24 hrs. The peak reflects the formation of new species. There are dips in bands corresponding to loss of alite [27] at 800-1000 cm^{-1} and gypsum [22, 28,29] at 1100-1200 cm^{-1}, as well as loss of free water, that is, the bands at 1650 cm^{-1} and 3000-3600 cm^{-1}. The peaks appear at 900-1200 cm^{-1} and 1350 cm^{-1} due to combined Si-O stretching and Si-OH bending modes characteristic of calcium silicate hydrate, a sharp resonance at 3650 cm^{-1} corresponding to the O-H stretch mode in Portlandite, $Ca(OH)_2$.

Compared with B01, after the addition of only SG i.e., SG04 Sample & PCE along with SG i.e., PSG04 has less wave number, but only addition of PCE i.e., P01 has almost the same wave number for the Si-O-Si. This may be the reason for the decrease in the compressive strength in the samples PSG04 and SG04. The absorption peak of $Ca(OH)_2$ is caused by the stretching vibration of O-H(3650 cm^{-1}). It can be clearly seen that the absorption peak of $Ca(OH)_2$ decreases for the samples PSG04 and SG04.

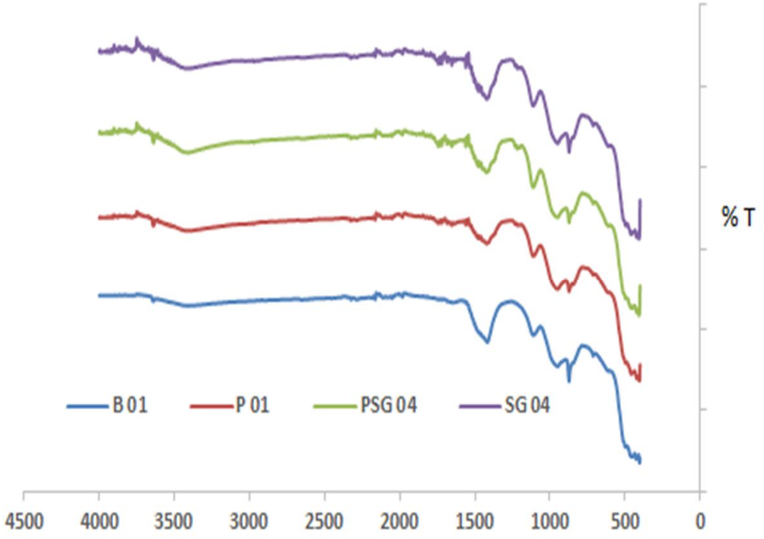

Figure 10: FTIR Spectra after 24 hours cement paste hydration.

E. Thermo-Gravimetric Analysis (TGA)

TGA testing was conducted for cement paste samples (B01, P01, PSG04 & SG04) at the age of 24 hours. The small pieces of samples were grounded to a 75-micron and then the formed powders passed through 75-micron sieve and collected to stop the hydration reaction. The powders the soaked entirely in the acetone and then dried using an air pump. The process of soaking and drying was repeated three times to ensure complete termination of hydration before being subjected to TGA.

Table 06. Weight loss (%) of the pastes in the range 50-120 ^{0}C and 50-550 ^{0}C.

Sample	24 hours	
	50-120 ^{0}C	50-550 ^{0}C
B01	4.18	8.31
P01	4.11	9.30
PSG04	1.49	5.77
SG04	3.66	8.37

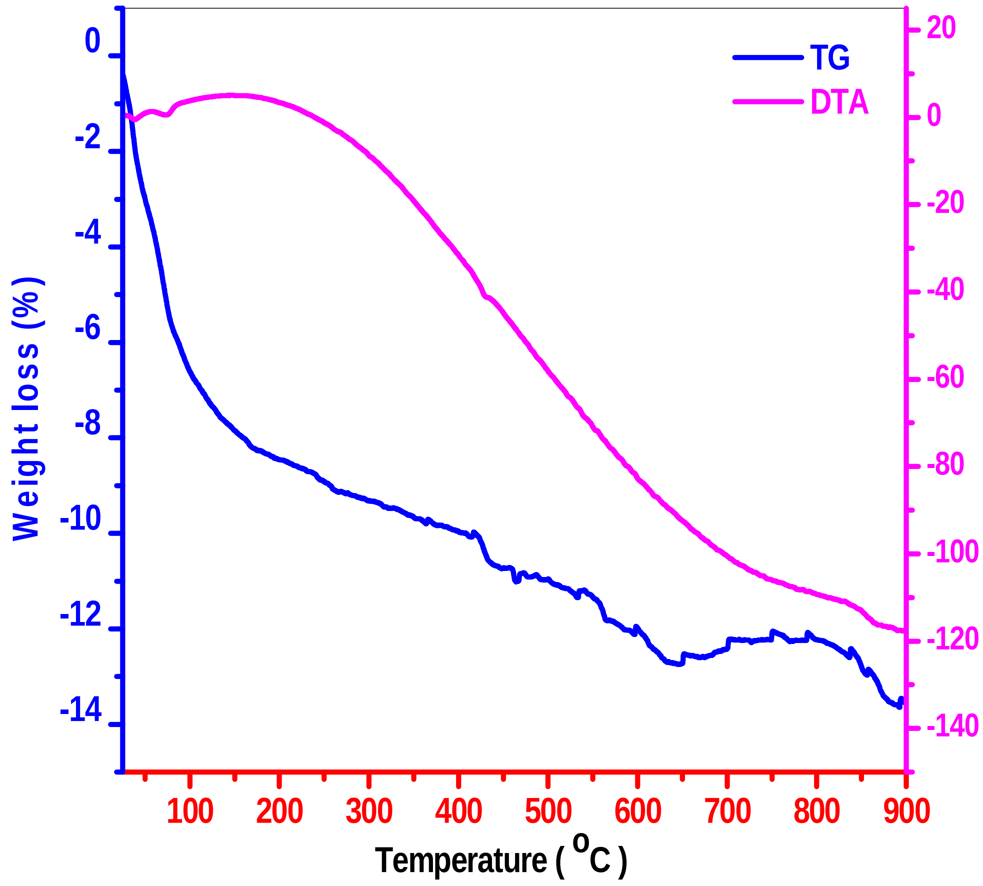

Figure 11: TG-DTA curve for cement paste sample B01 at 24 hours.

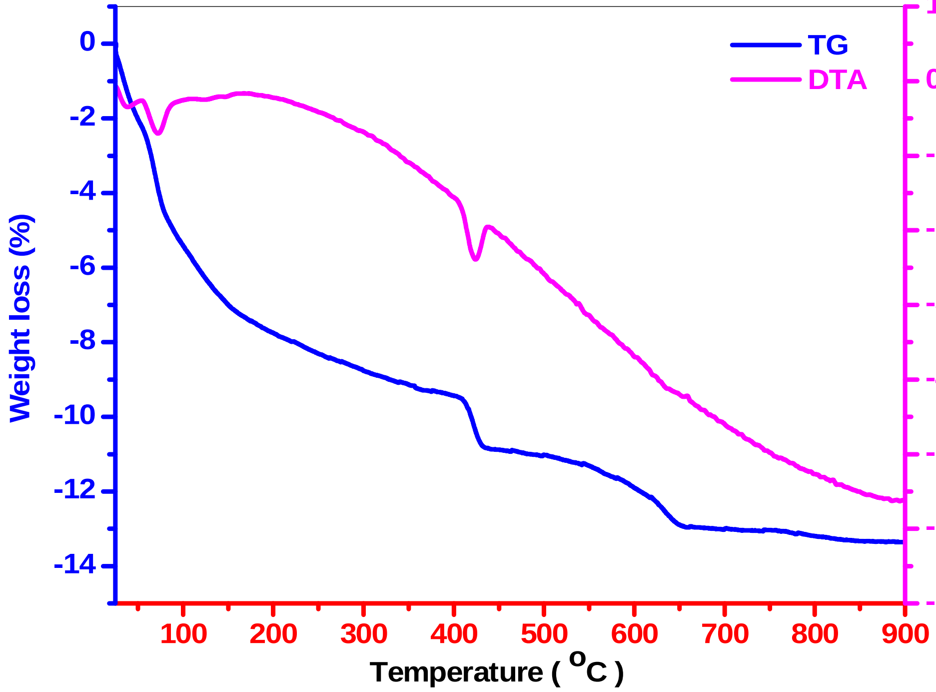

Figure 12: TG-DTA curve for cement paste sample P01 at 24 hours.

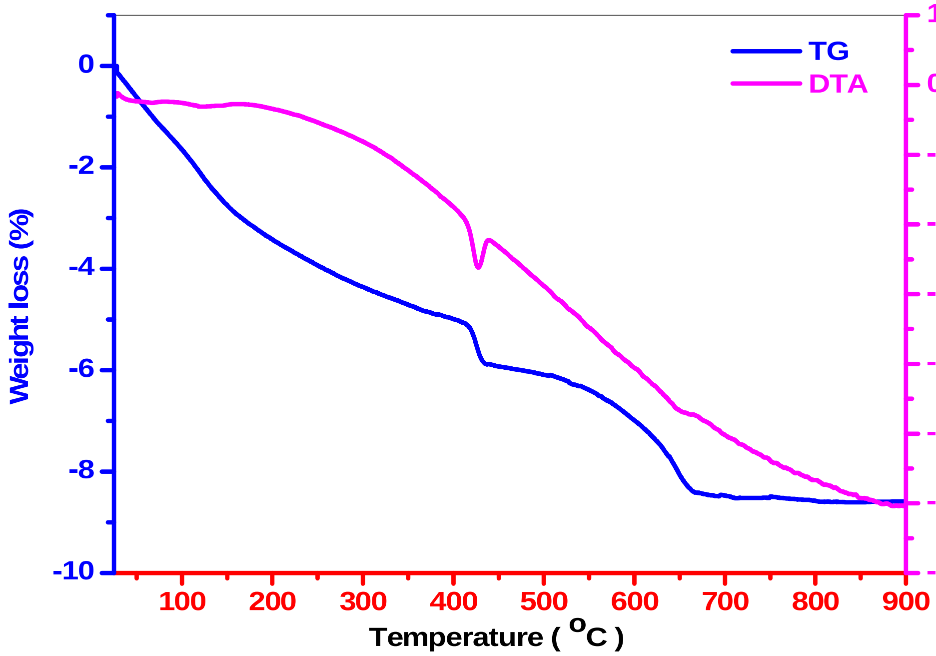

Figure 13: TG-DTA curve for cement paste sample PSG04 at 24 hours.

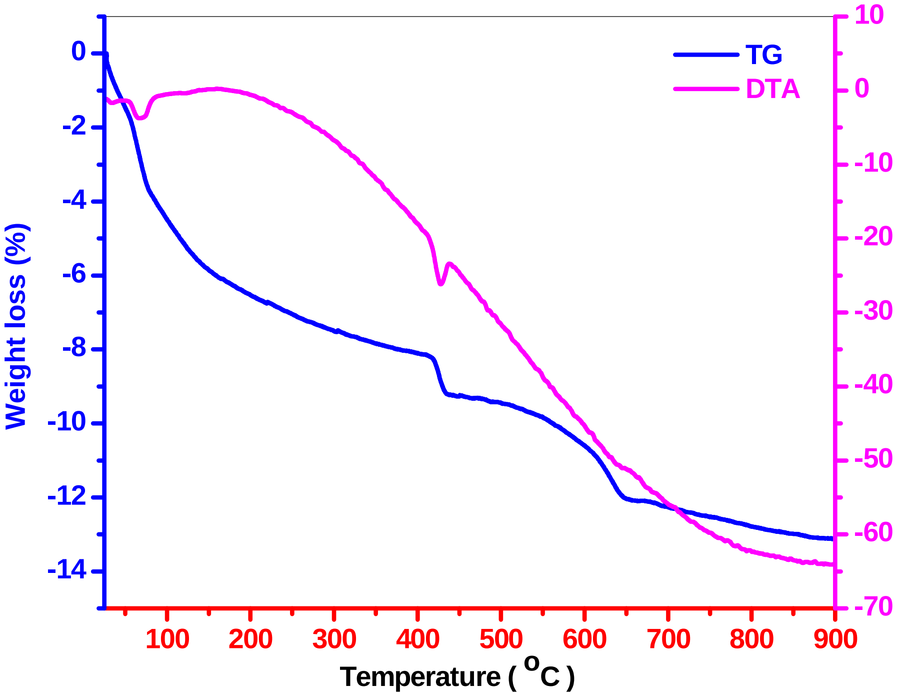

Figure 14: TG-DTA curve for cement paste sample SG04 at 24 hours.

The TG/DTA curves of cement paste hydrated at 24 hrs for the samples B01, P01, PSG04, and SG04 are plotted in Figures 11, 12, 13, and 14. The amount of ettringite (AFt) is defined as a mass loss at temperature ranging from 50 to 120 ^{0}C, and the amount of chemically bound water is defined as a mass loss at a temperature ranging from 50 to 550 ^{0}C [26]. The mass losses at 50 to 120 ^{0}C and 50 to 550 ^{0}C for all samples are mentioned the Table 6, and the results show that the amount of Aft at 24 hours for the PSG04 and SG 04 is less than P01 and B01.

IV. CONCLUSIONS

In this study, the fluidity, compressive strength, and hydration process of cement paste with w/c of 0.29 with and without the addition of Sodium gluconate in the PCE were investigated through a series of experiments. According to the results and discussion, we can derive the following conclusions.

1) SG when added to PCE at a higher percentage, shown a negative impact on the cement paste fluidity i.e., fluidity is reduced. When the optimum percentage of SG (0.05%) is added to the PCE then the fluidity of cement paste is improved.
2) Better early CST i.e., 01-day observed when only PCE(0.11%) is added to the cement paste, but when SG or SG added is added along with PCE in the cement paste then 01-day CST is reduced. The reduction in the 01-day CST is due to inhibition of hydration of Tricalcium silicate (C_3S).
3) The delay in the cement hydration due to the formation of low AFt for the PSG04 and SG 04 is observed in TGA-DTA analysis when it is measured for the hydrated paste at 01 day. The absorbance of the PSG04 and SG04 sample is at low wavenumber which indicated delayed hydration.
4) The lateral CST such as 07 and 28 days for the P01, PSG04, and SG04 samples are more than the B01 sample. The maximum CST for the 07 and 28 days was observed for the PCE-SG system. Therefore, it shows that optimum dosages of Sodium gluconate in the PCE improved lateral CST.
5) The heat evolution rate of cement paste with P01 sample (PCE0.11%) is more as compared to PSG04, SG04, and B01, this indicates that when only Sodium gluconate or it is added in the combination of PCE then more cement hydration retardation effect is observed.

6) The heat flow peak shifted from the left side to the right side in order of SG04>PSG04>P01>B01 is observed i.e., SG04 has more retardation effect.
7) The order of cumulative heat production at 24 hours is P01>B01>PSG04>SG04 but at 70 hours the order changed to P01>PSG04>B01>SG04, which indicates that at 24 hours the retardation effect of PSG04 is more, but which is reduced at 70 hours.
8) It is possible to co-relate the fundamental relationship between compressive strength (01 and 03-days CST) and produced cumulative heat (24 & 72 hrs.) with the help of statistical modelling.

V. ACKNOWLEDGEMENTS

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VI. CONFLICT-OF-INTEREST STATEMENT

The authors have no conflicts of interest to declare. All co-authors have seen and agree with the contents of the manuscript, and there is no financial interest to report.

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