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Effect of Mineral and Chemical Admixtures on the heat of hydration of Najran blended cement pastes

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ABSTRACT

Objectives: This study is to investigate the influence of mineral and chemical admixtures on the hydration of cement pastes by a replacement process of 30%. Methods: Cement pastes are made in accordance with ASTM C192M. the samples were kept at a constant room temperature of 2.5 °C until reached ambient temperature,the hydration behavior of blended cement pastes was studied at different temperatures ranging from 30 to 90°C and at constant water/solid ratios of 0.4 using an isothermal conduction calorimeter. A specific gravity 3.15, and special surface area of 410 m^2/kg.Silica fume with a specific gravity of 2.20 and special surface area of 22.500 m^2 /kg was employed. Fly ash with a surface area of 2900 cm^2/gm and specific gravity of 2.2 were measured. Findings: The results show a high intensity of the main peaks with a lower hydration temperature range between 30°C and 40°C. In contrast, at different temperatures (60°C and 90°C), it was shown the quantity of hydration decreased. The cumulative heat of hydration of blended cement paste is reduced with an increase of mineral and chemical admixtures. The activation energy Ea obtained at different temperatures of 30,40,50, 60,80 for mineral admixtures and chemical admixtures was 69.4, 57.67, 100, 87, and 66.23, respectively, and 0.65, 0.51, 0.45, 1.00, 0,90. Novelty: The performance of each material employed in cementitious composites, both chemically and minerally, admixtures affects the composites' fresh and hardened qualities. Nevertheless, very few studies have looked at the performance of mineral and chemical admixtures. As a low-cost substitute for commercial admixtures, the results obtained by both chemical and mineral admixtures give us the following completely new points in this articleThey also have a strong capability of optimizing the physical and chemical behavior of cement produced in the Najran cement industry.

Key words: Portland cement, fly ash, silica fume, CaSO4, NaSO4, Hydration kinetics, Activation energy.

1. INTRODUCTION

The accumulated heat of hydration is a special point in time to related to the total possible heat of hydration of the sample is always applied to estimate the degree of hydration.[1,2].The evolution of heat is measured immediately with isothermal calorimetry, and this procedure is the accurate measurements significance of the range of hydration than compressive strength. the thermodynamic modeling technique has been effectively applied by numerous researchers to assess chemical and mineral activity and cement hydration Activation energy E_a Calculations should be strong sufficient to distinguish between the components of different compositions and supplementary cementing materials, such as mineral and inorganic salts as an admixtures. Arrhenius' equation used to calculate activation energy E_a from isothermal calorimetric data and the Arrhenius' theory explains the hydration sensibility by temperature from the single reactions [3,4]. The thermodynamic modeling technique has been effectively applied by numerous researchers to assess chemical and mineral activity and cement hydration [5].Admixtures of mineral and inorganic salts are thought of as cement hydration accelerators because they contribute to the workability, strength, and durability of the hardened cement pastes and fresh mixtures, respectively. These properties are dependent on the enhanced properties that are frequently used.The hydration rate would rise with the addition of calcium salts like calcium chloride and calcium nitrate, which would also alter the morphology, microstructure, and composition of calcium silicate hydrates (C-S-H). Though opinions differ, the accelerating impact of calcium chloride has been linked to the increased permeability of C-S-H and the diffusion of chloride ions into the protective layer around unhydrated cement particles. By altering the mineral assemblage of hydration products and the composition, microstructure, and morphology of C-S-H, a high dosage of soluble alkali would increase cement hydration. There is still no clear explanation for why soluble alkali at low dosages (less than 0.5 M) slows down cement hydration [6]. The aim of this study , to demonstrate the benefits of mineral and chemical additives on cement paste hydration kinetics and thermodynamic stability By employing a conduction calorimeter.

2. Experimental and Methods

2.1 Materials

Cement

The heat of hydration activation energy and degree of hydration of industrial Ordinary Portland Cement (OPC I) according to ASTM C150 Type 1 [6] were tested. The samples were collected carefully from the Saudi Arabian regions. Tables 1, 2, and 3 list the mineral admixtures, cement's chemical composition, its physical characteristics. and the Blaine area of Portland cement, fly ash, and silica fume in the presence and absence of admixtures with different proportions.[7]

Mineral and chemical admixtures

Ordinary Portland cement produced by was used in Najran Saudi Arabia with a specific gravity 3.15, and special surface area of 410 m^2/kg.Silica fume with a specific gravity of 2.20 and special surface area of 22.500 m^2 /kg was employed. Fly ash with a surface area of 2900 cm^2/gm and specific gravity of 2.2 The chemical compositions of cement and silica fume are listed in Tables 1,2,and 3 Chemical admixtures $CaSO_4$ and $NaSO_4$ are available on the local market commercially in Saudi Arabia were used in the experiments.The mix proportion applied in this study is given in Table 4. The water-to-binder (the sum of cement with chemical admixtures and chemical admixtures ratio used for all mixtures is w/c (0.4) and contain by weight of binder.

Water

Drinkable tap water was used as a constituent of traditional and sustainable **cement** mixtures and for the curing of cement. The characteristics of the water were satisfied with the ASTM C1602/C1602M requirement [8].Standard Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete, ASTM C1602/C1602M; ASTM: West Conshohocken, PA, USA, 2018.

2.2 Instruments and experimental techniques

A conduction calorimeter (ZIAC, German Academy of Science) was used to calculate the heat of hydration evolution [9]. The calorimeter is made out of a 0.5-L wide-mouth vacuum jar with a cork non-reactive stopper that is held in a suitable, insulated container to protect it from extreme temperature swings. The vacuum jar was waxed over on the inside. The calorimeter had an inert fiber glass insulating layer and an insulated container. In this work, a variety of thermometer kinds were utilized (solution thermometer, and reference thermometer). Drying Oven, Mortar and Pestle, Funnel, Stirring, Storage, Sieves, Muffle Furnace, 120

mm, 1000°CAnalytical and analytical balances were the two types that were used to measure samples.

Table 1: Chemical composition Portland cement (wt%).

Items	Oxides	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	**Insoluble**	Ign.loss
wt.%	Najran cement	63.78	19.73	6.2	3.44	0.96	2.23	1.02	0.28	0.93	1.51

Table 2: Chemical composition of Mineral admixtures silica fume and Fly ash (wt%).

Items	Oxides	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	Insoluble	Ign.loss
wt.%	Fly ash	5.60	55.30	25.70	5.30	2.10	1.40	-	-	-	0.90
wt.%	Silica fume	0.30	94.3	0.09	0.10	0.43	0.10	0.048	0.058	-	1.14

Table 3: Physical properties of cement. Blaine area of different Portland cement, fly ash and silica fume

Type of cement	Surface area cm^2/g	specific gravity
Najran cement	410 m2/kg	3.15
Fly ash	2900 cm^2/gm	-
Silica fume	2774 cm^2 /gm	-

Table 4. Proportions of cement mixture with and without admixtures

Water/cement ratio	Cement (g) Control Portland cement 100%	With Mineral admixtures (silica fume, fly ash.)(g)	With Chemical admixtures (calcium sulphate and Sodium Sulphate) (g)	Water (ml)
0.4	400	-	-	200
0.4	300	10	-	200
0.4	300	-	10	200

2.2 Sampling and test specimens

Cement pastes are made in accordance with ASTM C192M [10]. Before making the paste, it was kept at a constant room temperature of 2.5 °C until the samples reached ambient temperature. Then, different weights of cement, both with and without admixtures, were combined with 200 ml of distilled water and

mechanically mixed at a water-to-cement ratio of 0.40. The mixture was then vigorously stirred for 5 minutes.. Equal amounts of the paste were placed in several plastic bottles, which were then sealed with tight stoppers and stored in a water bath at 25 °C for the duration of the test. Preparation of Partially Hydrated Sample: At the designated test time, a bottle of the partially hydrated sample was removed from storage. During the initial 15 minutes of calorimeter stirring, the plastic bottle was cracked away from the hydrated sample, and the whole cement paste was quickly ground with a mortar and pestle. The sample was then quickly passed through an 800-m (No. 20) sieve and put into a strong stoppered weighing bottle

2.4 Determining the heat of hydration

In the hydration heat test, A conduction calorimeter (ZIAC, German Academy of Science) was used.The data of hydration heat and rate were collected at different temperatures and time.Finally, the hydration kinetics model of the Portland cement with admixtures cementing material at different temperature was established and model coefficients were revised using regression analysis.A conduction calorimeter is characterized by continuous determination of heat released during hydration of cement constituents. By means of the heat released, it is possible to determine The experiment's findings include a measurement of temperature changes over time for cement pastes. The following equation establishes the equation used to compute the rate of heat evolution according to the Equation (1).

$$q_t = \frac{dq}{dt} \qquad (1)$$

where $q_{(t)}$ is the heat released by time t, t,ime in hour

$$\alpha = \frac{H_t}{H_u} \qquad (2)$$

where α is the degree of hydration at time t, H(t) equals the heat evolved from time 0 to time t (J/gram), and Hu is a total heat available for reaction (J/gram), where Hcem equals the total heat of hydration of Portland cement (J/gram) at α = 1.0.

2.5 kinetics parameters by isoconversional method

Given attention to the reaction order and temperature, the kinetics of hydration of each phase can generally be expressed as following equation:

$$\frac{d\alpha}{dt} = K(T)f(\alpha) \qquad (3)$$

Equation 4 characterizes the dehydration kinetics of cement pastes with and without admixtures . Hence, the global hydration kinetics of cement paste is considered as the combination of the kinetics of cement pastes with and with out admixtures

Arrhenius equation is used to describe the influence of temperature

$$\mathrm{K(T)} = A_0 exp - \frac{E_a}{RT} \qquad (4)$$

where A_0, the pre-exponential factor and E_a, the activation energy are referred to as the Arrhenius parameters. In this study, Friedman method was performed , and the reaction order is assumed to be one:

$$\ln\left(\frac{d\alpha}{dt}\right) = -\frac{E_a}{RT} + lnA + \ln(1-\alpha) \quad (5)$$

by plotting ln(dα/dt) against 1/T, one directly obtains the slope –Ea/R for a fixed a. And the lnA can be determined by the intercept lnA + ln(1 - α).

3. **RESULTS AND DISCUSSION**

3.1 Hydration Heat Analysis of Portland Cement–with admixtures

Figure 1 displays the hydration heat and rate curves of composite cementitious materials (blended cement, fly ash, silica fume, sodium sulfate, calcium, and sulfate). We can observe a trend where the hydration heat decreases with varying admixtures; however, the variations are not significant. Figure 1 illustrates how the inclusion of admixtures in the composite resulted in a considerable alteration in the hydration rate curves. Specifically, the hydration induction period was decreased, and the exothermic rate was enhanced during the acceleration period.Figures 1 and 2 illustrate the results, which demonstrate the values acquired during the first 12 hours. The amount of heat evolved was determined hourly at ages up to 28 hours.Figures 1 and 2 illustrate the influence of temperature on the calorimetric curves at a constant value of the water/cement ratio of 0.4. These figures explain the path of heat evolution during hydration. In the current study, the quantity of heat evolution of control cement pastes containing 30 percent of chemical admixtures ($CaSO_4$ and $NaSO_4$), fly ash, and silica fume showed less heat evolution up to 12 hours than the control cement

percent sample in the absence of admixtures. Because the cement was mixed outside of the calorimeter, it is ignored when the first peak for the initiating hydration process on the surface of C_3A phase cement particles emerges at the first step. The height of a second peak was found to be lower with cement of 30% than the control cement percent; this may be because cement with less C_3S available for increasing admixtures has a dilution effect, reducing the amount of C_3S that can be added. Additionally, the results might be a result of the alumino-silicates' sluggish interaction with calcium hydroxide (C-H), which was released from cement because of the admixtures' decreased solubility in pore solution [11]. Due to its quick reaction time, the first stage of the cement hydration process (initial hydrolysis) is frequently missed by the calorimetric test. Figures 1 and 2 depict the heat flow as a function of time during the hydration of cement in the presence and absence of the cement pastes 30 (percent) utilised in this study. Assume that:

1) Rapid heat evolution as a result of wetting the cement, alkalis dissolving, hemihydrates dissolving, C_3A hydrating, and C_3A's reaction with gypsum. During the period of induction during which the hydration products grow to a specific size, (3) As soon as the critical size is attained, nucleation and crystal formation begin. At this stage, hydration mostly affects the alite phase and accelerates over time to a maximum value. C-S-H, one of the hydration byproducts, may be nanometer-sized. 4) The deceleration phase, in which a large number of hydration products are formed and the rate of heat evolution slows as hydration duration increases. 5): The final phase, during which the reaction is diffusion-controlled and progresses at a very slow rate. Cement's hydration process heat Some high-calcium class C fly ash with self-cementitious qualities may react with water very quickly, generating excessive heat in a manner similar to how cement hydrates [12]. After 28 hours, the heat produced by dicalcium silicate and tetracalcium alumino-ferrite appears to be slightly increased by the addition of chemical admixtures calcium chloride and sodium sulphate, whereas tricalcium aluminate appears to be slightly decreased.in addition, test results indicate that the cement–admixtures binary system can short the dormant period, increase the exothermic rate during the acceleration period, decrease the second hydration heat evolution peak, and reduce the hydration rate during the deceleration period. Moreover, it is becoming clear with the different admixtures , when compared with the hydration heat evolution process of pure cement [13].

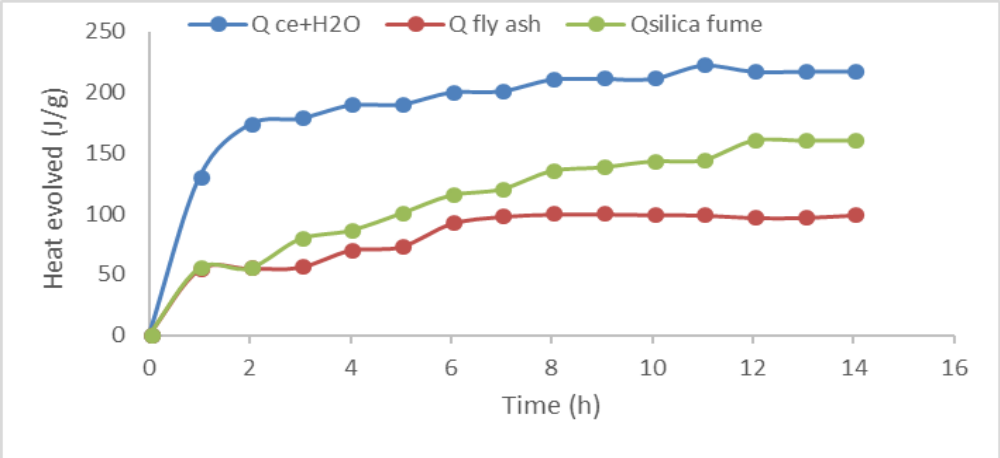

Figure 1. Heat of the composite cementitious system with mineral admixtures

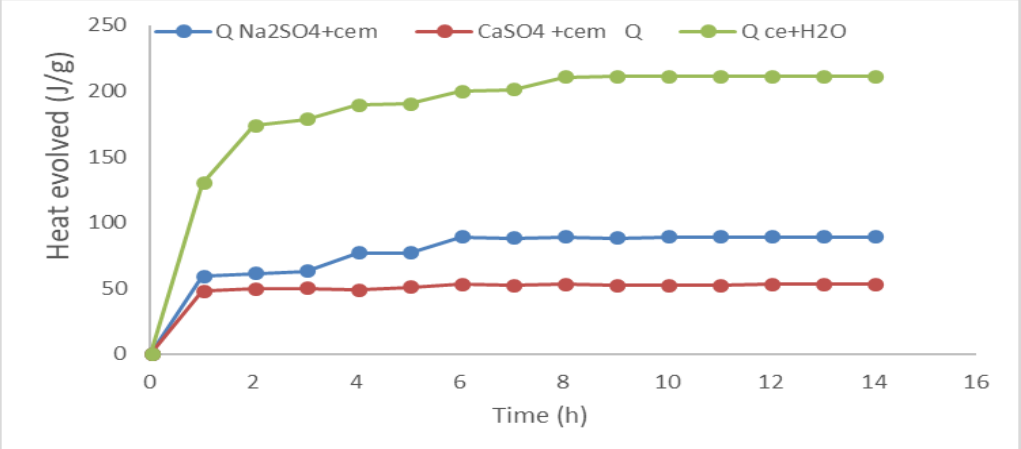

Figure 2. Heat of the composite cementitious system with chemical admixtures

3.2 Effect of Temperature on the Hydration Heat and Rate

The results from the tests of the different temperature and asmixtures are shown in Figures 3 and 4. As observed, the hydration heat was increased by increase of reaction temperature from 30 to 90^0C, but the reduction coefficient gradually became stable.It can be seen the inecrease of reaction temperatures, the induction period of hydration for all the materials was elongated, the hydration exothermic rates were high and the secondary exothermic peak was decreased and delayed. This trend becomes gradually insignificant with the rising of reaction temperature. It can be seen from Figure 4, a reduction of secondary exothermic peak of 0.4 mw/g were brought when the temperature was inecreased from 30 ◦C to 90 ◦C, while. It is known that cement pastes with out were high in heat evolved but it was reduced with cement admixtures (Figures 3 and 4), but the influence is less comparable with pure cement, especially for the higher temperature.

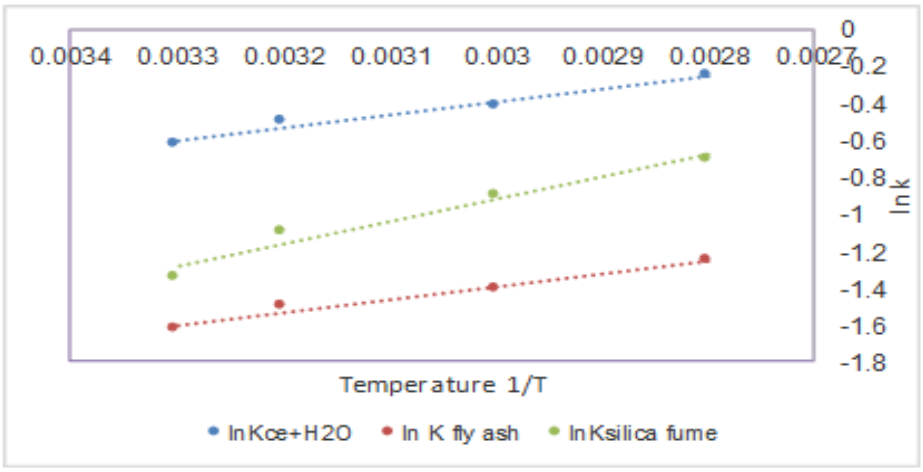

Figure3. Determination of k using linear fit for the cement samples and E_a from plot of ln (k) versus 1/T at 0.4 Chemical admixture .

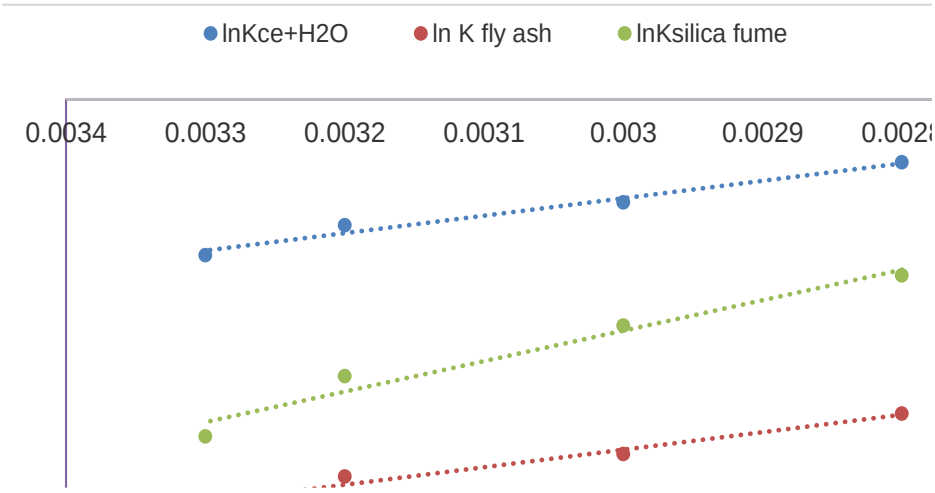

Figure 4. Determination of k using linear fit for the cement samples and E_a from plot of ln (k) versus 1/T at 0.4 Mineral admixtures .

3.3 Determination of kinetics parameters

The degree of conversion at multi-step kinetics, like the dehydration of cement samples, affects the kinetic parameters. Multiple heating rates are used in isoconversional approaches to gather data on different rates with a constant degree of conversion. As a result, multi-step processes can be identified using isoconversional approaches by comparing an Ea variation with an. Figure 5 displays the plotted ln(dα/dt) against 1/T curves of cement samples with and without admixtures at fixed hydration degrees ranging from 0.1 to 0.9. Plots of the samples' ln(dα/dt) against 1/T were found to be linear. With increasing hydration

degree, the trend of cement pastes' ln(dα/dt) against 1/T plots changes[.

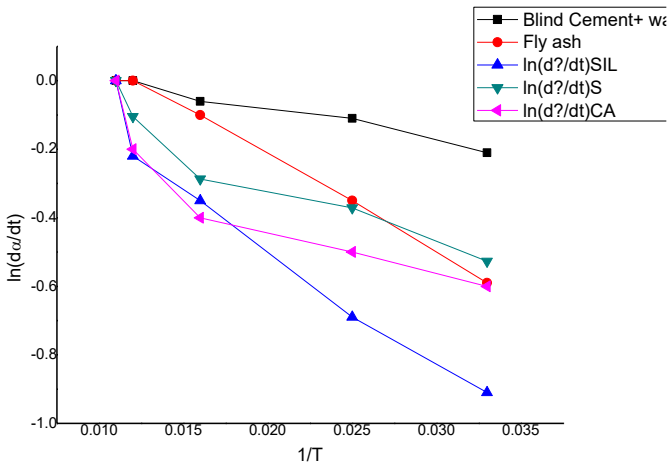

Fig 5 .Curves of ln(da /dt) against 1/T of cement pates with and without admixtures at fixed dehydration degrees range

3.4 Determination Activation energy

E_a values were determined for the cement pastes used in this experiment at various levels of hydration (α). By plotting lines of the ln(da /dt) against 1/T plots in Fig. 4, the E_a and lnA are determined and showed in Fig. 5. It is showed that the Ea and the lnA of cement samples develop with a obviously. The E_a of dehydration of cement samples are recorded in table 5. Due to C_3A, E_a for silica fume rises to a maximum of 100J/mol during the major hydration peak and then sharply falls afterward. Fly ash of Class C, sodium sulphate, and calcium sulphate mixtures behave differently in terms of activation energy; they show a modest decrease at the same degree of hydration (α). The activation energy E_a is the average excess energy that a water molecule must possess to release [14]. A variation of E_a with reaction process implies that systematic change of reactivity takes place during the progress of a reaction. The possible reasons of a variation of E_a were discussed by [15].

Table 5: Activation energy E_a ,and degree of hydration ά for control cement and blended cement samples.

Mixture	E_a J/mol	R^2	ά
Cement 100%	69.4	0.977	0.65
Fly ash 30%	57.67	0.977	0.51
Silica fume 30%	100	0.96	0.45
$Na_2SO_4$30%	87	0.98	1.00
$CaSO_4$ 30%	66.23	0.94	0,90

3.5 Thermodynamic calculation of cement samples

Thermodynamic calculations were performed for each cement samples in the present of admixtures and in absence of admixtures. The purpose of this approach was to evaluate whether the cement pastes provides a more accurate thermodynamic basis for determining admixtures reactivity as compared to cement without admixtures.Thermodynamic equations(6 and 7) were used to calculate the thermodynamics parameters and the results obtained are compiled in Table 6.

$$nkc = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \qquad (6)$$

$$\Delta G = -RTlnKc \qquad (7)$$

The calculated Gibbs energy ,enthalpy and entropy are referred to the stable modifications of the simple oxides.The estimated data of thermodynamic parameters were agree well with the dataset published by [15,16] Figures 3 and 4 show the experimental values exhibit very good agreement in the temperature range between 30 and 90 °C. Only values appear as a stable phase at different temperatures in the cement pastes with and without admixtures . This indicates from the lower total Gibbs energy and the other thermodynamic parameters of the system and is energetically favourable[17].

$$lnKc = \frac{\Delta H}{RT} - \frac{\Delta S}{R} \qquad (6)$$

$$\Delta G = -RTlnKc \qquad (7)$$

Table 6. Standard thermodynamic parameters at different temperatures.					
Type of admixture	Sample	Temp (K)	ΔG◦ [kJ/mol]	ΔH◦ [kJ/mol]	ΔS [J/K/mol]
mineral admixture	Cement without admixtures	303 313 333 353 363	1561.86 -1305.21 -1150.94 -736.00 -1877.00	-83.32	-20.28
	Cement with Silica fume	303 313 333 353 363	-3386.20 -1487.93 -2499.49 -2060.81 -4056.74	-175.33	-46.91
Inorganic or chemical admixture	Cement with Fly ash	303 313 333 353 363	-2476.47 -2088.33 -1583.01 -971.52 -999.04	-124.03	-28.711
	Cement with $CaSO_4$	303 313 333 353 363	-3537.82 -3132.50 -2360.63 -2502.41 -3632.90	-25992.82	-73.57
	Cement with Na_2SO_4	303 313 333 353 363	-2476.47 -1209.43 -1936.00 -1619.21 -999.04	-101.18	-26.99

Table 7. Comparison of heat hydration of cement pastes with admixtures

Cement pastes with and with out admixtures	Ea J/mol	ΔG◦ [kJ/mol]	ΔH◦ [kJ/mol]	Reference
Sulphate resistant neat Fly ash – Portland	--	-1570.9	-1742.4	Zajac, Maciej, et al. "Late hydration kinetics: Indications from thermodynamic analysis of pore solution data." *Cement and Concrete Research* 129 (2020): 105975.
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4.Conclusions

The aim of this study is to show the the replacement of mineral and inorganic chemical admixtures on the hydration of cement pastes because the cement hydration process is very
highly complex process that involves competing chemical reactions and physical changes over many time scales. Experimentally cement pastes had prepared using different admixtures and at absence of admixtures and using isothermal instruments used for estimation of heat of hydration. Hydration ,activation energy thermodynamics parameters were determined. The outcome show good heat hydration by using admixtures and thermodynamically stable phases of cement pastes using admixtures were shown. Because the cement hydration process is a very complex process involving competing chemical reactions and physical changes over different time scales, the goal of this study is to demonstrate the replacement of mineral and inorganic chemical admixtures in the hydration of cement pastes. Cement pastes were made experimentally with varying admixtures, without admixtures, and with isothermal devices to estimate the heat of hydration. Thermodynamic parameters for hydration and activation energy were ascertained. The results demonstrate excellent heat hydration when admixtures are used, as well as thermodynamically stable phases of cement pastes when admixtures are used. The outcome obtained from the results of the experimental investigation was as follows: At a water/solid ratio of 0.4, rising temperatures speed up the hydration reaction of cement pastes and control cement. Higher temperatures cause the primary hydration peak to develop shapelier, which indicates a shift in the hydration mechanism. Under steady water/solid ratios of 0.4, activation energy was calculated using Arrhenius' equation.It was determined that both types of admixtures speed up the hydration of calcium silicates and start the production of C-S-H under normal circumstances.
According to this study's novelty is because mineral admixtures are derived from natural sources and are safer and more thermodynamically stable in the environment, they are superior than chemical admixtures. Recomomnation for future Work is underway to incorporate new supplementary cementitious materials so cement chemistry researchers can develop new, more environmentally friendly products.

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