Research Article

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Activation energy of lime cement containing pozzolanic materials

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Abstract: The impact of locally pozzolanic materials on the activation energy of lime cement, with Grade 32.5, was assessed in the fresh and hardened phases of paste and mortar, respectively. The activation energy is used to represent the relationship between the strength gain of concrete over time and temperature sensitivity. Two proportions of mixed pozzolanic materials, metakaolin (MK) and silica fume (SF), were used as a replacement for cement weight. The first was 5% (MK) and 7% (SF); while the second was 10% (MK) and 15% (SF) as a high replacement level. The activation energy of cement was calculated, as per the ASTM C1074 approach, at 5, 20, and 35°C temperatures. The results showed that the rate of strength development (*k*-value) increased in mixes containing pozzolanic material at all temperatures. The activation energy (E_a) increased in all mixes containing pozzolana but decreased with a high replacement level of pozzolana, because of the filler effect during the hydration progress due to the high surface area of these materials, resulting in loss of strength. The results concluded that using the strength development rate (*k*-value), calculated from ASTM C1074, to estimate the concrete strength at any age is valuable and it is very close to the actual values.

Keywords: activation energy, pozzolanic materials, strength development, temperature

1 Introduction

The ASTM C1074 standard adopts two approaches to estimate concrete strength in engineering projects. The first is concrete maturity determining compressive strength based on time and temperature measurements. The second, which is more accurate, uses Eq. (A1.1) as designated in ASTM C1074 depending on time and heat energy. The specification recommends additional studies to assess the impact of additives, such as pozzolanic materials, on concrete performance during early and later ages. The use of pozzolanic materials (SCMs) as partial replacement to lime cement concrete improves concrete durability and reduces carbon dioxide emission within cement manufacture [1,2]. The hydration kinetics of the cementitious system, presented in the setting time and strength development rates, depends significantly on the compounds of the cementitious system and their physical and chemical properties. The rate of cementitious hydration is also affected by the activation of these materials along with curing temperatures that impacts significantly in the rate of hydration [3]. Consequently, understanding the effect of temperature sensitivity and apparent activation energy of the cementitious mixes containing different pozzolanic materials such as silica fume (SF) and metakaolin (MK) in different percentages is necessary to identify the time-dependent behavior of the cementitious mixes. The activation energy is considered the minimum energy needed for the reaction to occur. The temperature has an impact on this energy; a higher temperature produces higher energy. Various methods have been utilized to predict the activation energy of cementitious materials [4–7]. These methods are presented by setting time [8,9], heat evolution [5,6], and strength development ASTM C1074 [4]. The method used to determine the activation energy depends on the function of maturity, whether it is for setting time, strength gain, or the rate of hydration [8]. In 2012, Siddiqui and Riding examined the impact of temperature sensitivity on the strength gain and activation energy of cement paste and mortar. The study compared the activation energy values resulting from various approaches, such as setting time, mortar strength, chemical shrinkage, and isothermal calorimetry tests. The study concludes that the activation, resulting from isothermal calorimetry, of cement paste and mortar was almost the same; this means that the aggregate has little impact on the activation energy. The results imply

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the possibility of using the activation energy of cement paste to concrete [10]. In 2021, Yogiraj and Wang studied the effect of the use of different nanoparticles including powdered $n\mathrm{SiO_2}$, $n\mathrm{Al_2O_3}$, $n\mathrm{CaCO_3}$, and $n\mathrm{SiO_2}$ on the hydration and activation energy of cement paste. The study used different percentages of replacement of cement with nanoparticles under various temperatures. The results implied that cement hydration increases with high nanoparticle replacement, while it decreases with the increase in testing temperature. On the other hand, the activation energy increases with low nanoparticle replacement, while it decreases with high replacement of nanoparticles. This finding may aid in selecting the optimum dosage of nanoparticles for optimizing the activation energy [3]. In 2021, Al-Amoudi *et al.* inspected the advantages of using hydrated lime in concrete to activate natural pozzolan materials. The study focused on the use of nanopozzolans as a partial replacement of cement to improve concrete durability. The study determined the optimum replacement of nanopozzolan and lime in concrete based on the performance of various trial mixes. The results concluded the use of lime with nanoparticles may improve concrete strength, chloride diffusion, corrosion, and strength loss for the specimens' exposure to sulfate attack [11]. In 2023, Khalil and Dawood examined the impact of several grades of cement (32.5, 42.5, and 52.5), specifically those containing SF, MK, and limestone powder, on the mechanical qualities of high-performance cement mortar. Two percentages of pozzolanic material substitutions (25 and 30%) on compressive, flexural, and tensile strengths demonstrated the substantial influence of the cement grades on the effectiveness of high-performance mortar. Furthermore, the study established the optimum replacement of each pozzolanic ingredient incorporated in the mortar mix for every cement grade to achieve optimal performance [12]. In 2024, Li *et al.* evaluated the effect of SF content on the hydration rate and activation energy at low curing temperatures using the isothermal calorimetric method. The results indicated that the increase of SF content exhibited increasing in the intensity of the hydration rate, while low temperature caused lower and delay of the heat flow peak. In addition, the high SF content reduced the effect of temperature sensitivity on the hydration process [13].

All previous researches have provided only limited information regarding the maturity and apparent activation energy of mixes including varying percentages of mixed pozzolanic materials, which were cured under non-standard temperature conditions. Thus, this study focused on determining the activation energy for the cement paste and mortar, using a modified version of ASTM C1074 [4], that was later used to estimate the development of compressive strength in concrete at different ages at standard curing temperatures. All mixes contained limestone cement incorporating two different pozzolanic materials, namely SF and MK, at varying replacement ratios. The mixes were then subjected to different temperature conditions throughout the curing process. This study is mainly focused on the ASTM C1074 recommendations for using pozzolanic materials that have cement properties and are environmentally friendly. The study evaluated the combined effect of SF and MK, in different replacements (12 and 25%), on the rate of hydration and concrete maturity. The evaluation can be achieved by tracking the behavior of concrete based on (k-value and E_a) for mortar and cement paste. The study is additionally validated the effectiveness of Eq. (A1.1) as designated in ASTM C1074 with the presence of pozzolanic materials in assessing the behavior of concrete based on the compressive strength and comparing it with the actual strength resulting from the experimental study.

2 Materials and methods

2.1 Constituents of all mixes

2.1.1 Cement

Ordinary Portland cement was used for all the mixes in Grade of 32.5, and mixed with 10% limestone powder, which was newly made in Sulaimani, Iraq. The cement was in accordance with the specifications of CEN-EN 197-1 [14]. Tables 1 and 2 display the chemical and physical properties, respectively, of the cement obtained from Almas Factory, which is locally accessible. All tests were conducted in the Construction Materials Testing Laboratory, Civil Engineering Department, University of Mosul, in accordance with the Iraqi Standard Specification IQS: 5/2019 [15].

2.1.2 Limestone

Limestone powder used to produce lime cement (10% replacement of cement) was derived from the crushing process at Badoosh Cement Factory and ground to a fineness of 3,500 cm^2/g. The activity index was 80% and determined in accordance with the ASTM C618 [16]. Table 1 provides the chemical properties of Limestone.

Table 1: The chemical properties of cementitious materials

Chemical composition %	Lime cement		Limestone	SF	MK
	Test results	IQS 5/2019 limitations			
CaO	62.8		—		—
SiO_2	19.8		6.9	—	51.3
AL_2O_3	5.6		1.7	98.5	40.2
Fe_2O_3	3.5		0.98	—	0.45
SO_3	2.6	≤2.8%	0.42	—	0.16
MgO	3.4	≤5%	2.5	—	0.3
Insoluble residue	2.8	≥1.5%	—	—	—
Loss of ignition	0.9	≤4%	—	—	1.3
Total	98.8		—	—	91.9
Free CaO	1.4		—	—	—
$CaCO_3$			84	—	—
Total	98.8		96.5	98.5	91.9
Main compounds					
C_2S	19.7		—		—
C_3S	49.2		—		—
C_3A	8.8		—		—
C_4AF%	10.8		—		—

2.1.3 MK

MK is a type of clay called MK Clay that is readily available locally. It is utilized as a pozzolanic material of Type N, as defined in ASTM C618 [16]. The activity index was 108% and determined in accordance with ASTM C618 [16]. Tables 1 and 2 provide a comprehensive overview of the physical and chemical properties, respectively, of MK.

2.1.4 Micro SF

Micro SF known as SF was utilized. The activity index was 117% and determined in accordance with the ASTM C1240 [17]. Table 1 contains a list of chemical properties of micro SF.

2.1.5 Fine aggregate

The river sand was utilized as the fine aggregate, sourced from the Kanhash area in Mosul, Iraq. The highest size of the aggregate is 2.36 mm. The specific gravity of the sand is 2.67, with a water absorption rate of 1.7%, as determined using the ASTM C128 [18]. The density of the material was 1,670 kg/m^3, as determined using the ASTM C29/C29M [19], and a fineness modulus of 2.85. The sand grading meets the specified restrictions outlined in ASTM C33 [20]. Furthermore, the sand utilized in all the mixes was in the saturated surface dry state.

2.1.6 Water

Tap water is used for both the mixing and typical curing procedures.

2.2 Research approach

2.2.1 Setting time measurement of cement paste

To create cement paste, combine lime cement with SF and MK in three varying proportions: 0, 12, or 25%. The total amount of cementitious materials, including cement, lime, MK, and SF, was measured to be 500 g. Several researchers [3,10] have accepted the use of the setting time approach to determine the progress of hydration. The present study employed a Vicat Needle Test, as per the guidelines specified in ASTM C191 [21], to determine the precise initial and final setting time of various cement pastes, as shown in Figure 1(a). The Vicat Needle Test was performed on cement pastes with varying proportions of pozzolanic ingredients to

Table 2: The physical properties of cementitious materials

Physical properties	Lime cement		Limestone	SF	MK
	Test results	IQS 5/2019 limitations			
Setting time, initial (min.)	135		—	—	—
Setting time, final (min.)	270		—	—	—
Pozzolanic activity index %	—		—	117	108
Water requirements with control (g)	106		—	115	114
Autoclave %	0.23	≤0.8	—	—	0.08
Specific gravity	3.15		2.1	2.3	2.63
Blaine fineness (cm^2/g)	3308	≥10.0	3,500	200,000	7,800
Color	Grey		white	Grey	white
Compressive strength (N/mm^2)					
2 days	21.3	≥10 N/mm^2			
28 days	39.5	≥32.5 N/mm^2			

Figure 1: (a) Vicat needle measurement for initial and final setting time. (b) Cement paste specimens cured at 5°C. (c) Mortar specimens sealed and cured at 5°C. (d) Compressive strength test of mortar specimens.

determine the standard consistency of each paste. The specimens were subjected to testing and cured at various temperatures, specifically 5, 20, and 35°C, as shown in Figure 1(b). The initial setting time refers to the duration needed for the paste to acquire its ability to resist penetration, while the final setting time is the duration required for the paste to lose its plasticity and transition into the hardened stage, at which point it is safe to remove the form.

2.2.2 Mortar strength

To create cement paste, combined lime cement with the mortar cube compressive strength test, as outlined in ASTM C1074 [4], was employed to determine the apparent activation energy (E_a) for various mortar mixes. The mixing ratio for all mixes was 1:2.75 with a w/cm ratio of 0.48. Nine specimens were cast for each combination in cubes measuring 70.1 mm × 70.1 mm × 70.1 mm. After demolding, each three specimens were sealed to prevent evaporation and stored at three different temperatures: 5, 20, and 35°C, as shown in Figure 1(c). The compressive strength test, following ASTM C109 [22], was conducted on the specimens at 1, 2, and 4 days to determine the rate of hydration, as shown in Figure 1(d).

2.2.3 Concrete strength

A total of 45 concrete specimens, with a proportion of 1:1.44:2.1 with a w/cm ratio of 0.48, were cast in standard dimensions of cube 150 mm × 150 mm × 150 mm and then subjected to a curing process under sealed conditions at a temperature of 20°C. The specimens were tested to a compressive strength according to the BS EN 12390-4 [23]. The specimens were tested at 1, 3, 7, 28, and 56 days to determine the actual strength. This actual strength was later compared to the predicted strength obtained from the activation energy approach. Table 3 provides a comprehensive list of mix proportions for all mixes.

3 Experimental results and discussion

The combined effect of pozzolanic materials and curing temperature on the activation energy of various cement pastes, mortar, and concrete mixes has been examined and resulted in the following.

Table 3: Mix proportions of all mixes

Mix type	Weight of materials (kg/m^3)						
	Cement	MK	SF	Limestone	Fine. agg.	Coarse agg.	Water
*P(control)	450	0	0	50	0	0	145
P(5%MK + 7%SF)	410	21	26	45	0	0	145
P(10%MK + 15%SF)	363	42	55	40	0	0	145
**M(control)	450	0	0	50	1,375	0	240
M(5%MK + 7%SF)	396	35	25	44	1,375	0	240
M(10%MK + 15%SF)	338	75	50	37	1,375	0	240
***C(control)	450	0	0	45	720	1,050	240
C(5%MK + 7%SF)	396	35	25	44	720	1,050	240
C(10%MK + 15%SF)	338	75	50	37	720	1,050	240

*P: cement paste.
**M: mortar.
***C: concrete.

3.1 Activation energy in cement paste

The apparent activation energy (E_a) of cement pastes was determined using a method prescribed by ASTM C1074. This method includes measuring the final setting time of three mortar specimens cured in three temperatures (5, 20, and 35°C) and then determining compressive strength with ages of 1, 3, 7, 28, and 56 days for the specimens. To compute the relationship between the rate of strength development (k-value) *versus* the curing temperature, the slope and the intercept of the fitting line of obtained strength results for each curing temperature were determined. Finally, the natural logarithm of the k-value was plotted against of reciprocal temperature; the negative of the slope of the best fitting line through the three points (Q) was multiplied by the gas constant (R) to determine the activation energy E_a, according to Eq. (1) ASTM C1074 [4]. The specimens' setting time at temperature conditions (5, 20, and 35°C) are displayed in Table 4. Subsequently, the natural logarithm of the reciprocal of the initial and final setting time (in hours) was graphed against the reciprocal of the temperatures (in Kelvin), as illustrated in Figures 2 and 3, respectively. This was done to determine the Q value, which is then used to calculate E_a using Eq. (1) and documented in Table 4.

$$E_a = R \times Q, \tag{1}$$

where E_a = apparent activation energy (J/mol), R = gas constant = (8.314 J/mol/k), and Q = slope of the fitting line to the natural logarithmic [1/setting time (h)] *versus* $1/T$ (Kelvin).

At early hours, utilizing pozzolanic materials as a replacement for cement in the mixes exhibited a notable enhancement in cement hydration, which was amplified at elevated temperatures. The rate of strength development (k-value) increased in mixes containing pozzolanic material at all temperatures; however, the activation energy (E_a) decreased as the percentage of pozzolanic materials replacement increased. The reason for this might be due to the high specific surface area of these materials, which makes them suitable as fillers rather than activation materials, as stated in previous studies [3,24]. The activation

Table 4: Setting time of cement pastes under different temperatures and activation energy (E_a) of pastes

Mix type	Curing temp.					
	Initial setting time (min)			Final setting time (min)		
	5°C	20°C	35°C	5°C	20°C	35°C
P(control)	165	135	90	390	270	195
P(5% MK + 7% SF)	150	120	75	330	210	150
P(10% MK + 15% SF)	120	105	75	345	240	180
Mix type	**Rate of strength development (k-value) %, (1/day)**					
P(control)	36	45	67	15	22	31
P(5% MK + 7% SF)	40	50	80	18	29	40
P(10% MK + 15% SF)	50	57	80	17	25	33
Mix type	**Activation energy E_a (J/mol)**			**Activation energy E_a (J/mol)**		
	Initial setting			**Final setting**		
P(control)	14,375			16,468		
P(5% MK + 7% SF)	16,350			18,759		
P(10% MK + 15% SF)	11,185			15,471		

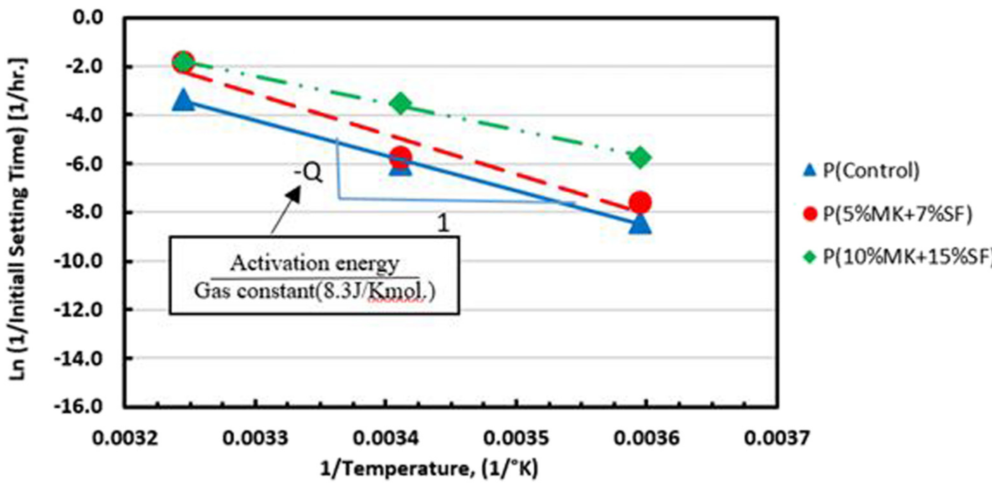

Figure 2: Natural logarithm of reciprocal initial setting time *versus* reciprocal temperature.

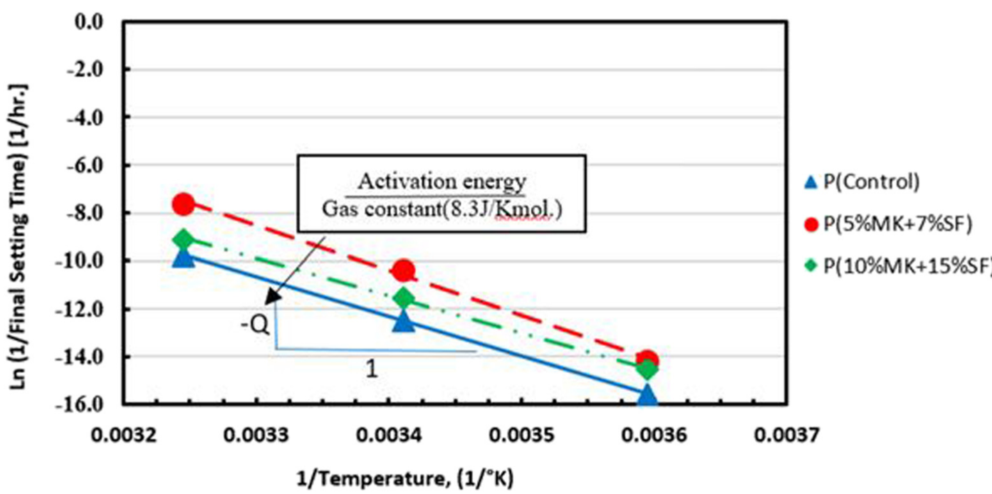

Figure 3: Natural logarithm of reciprocal final setting time *versus* reciprocal temperature.

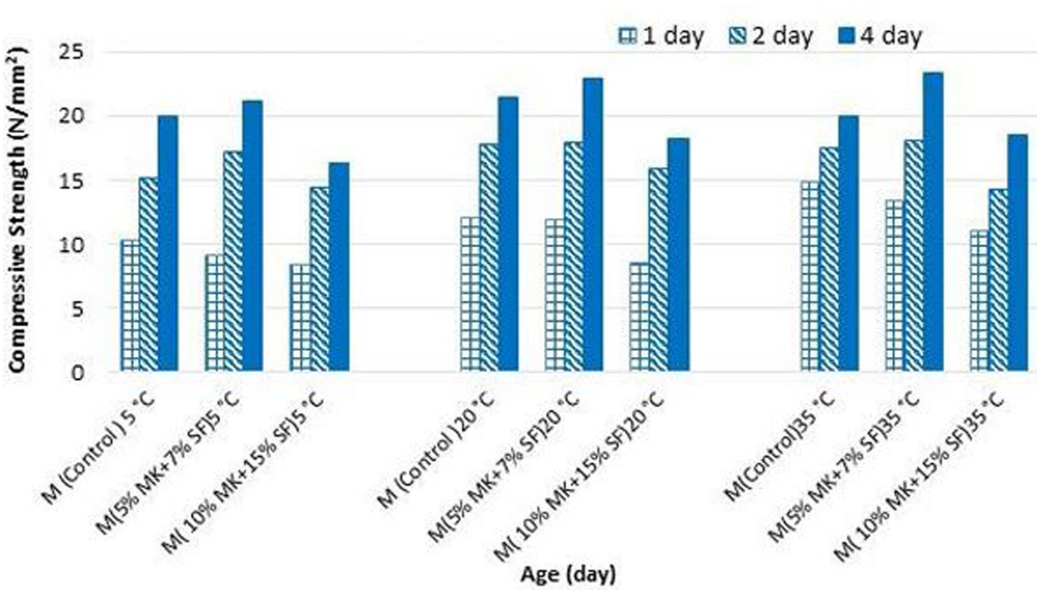

Figure 4: Compressive strength of mortar.

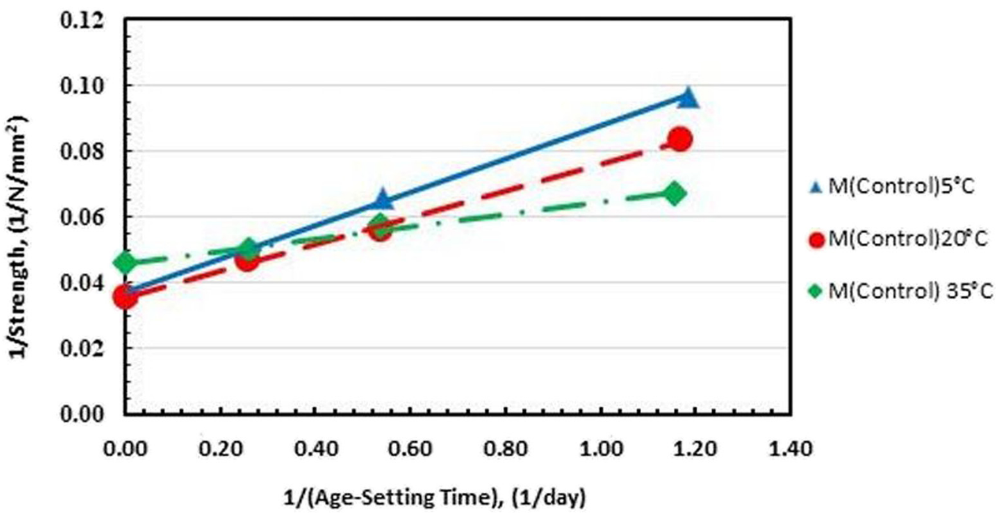

Figure 5: Reciprocal strength *versus* reciprocal age beyond the time of final setting of the M(control) mixes.

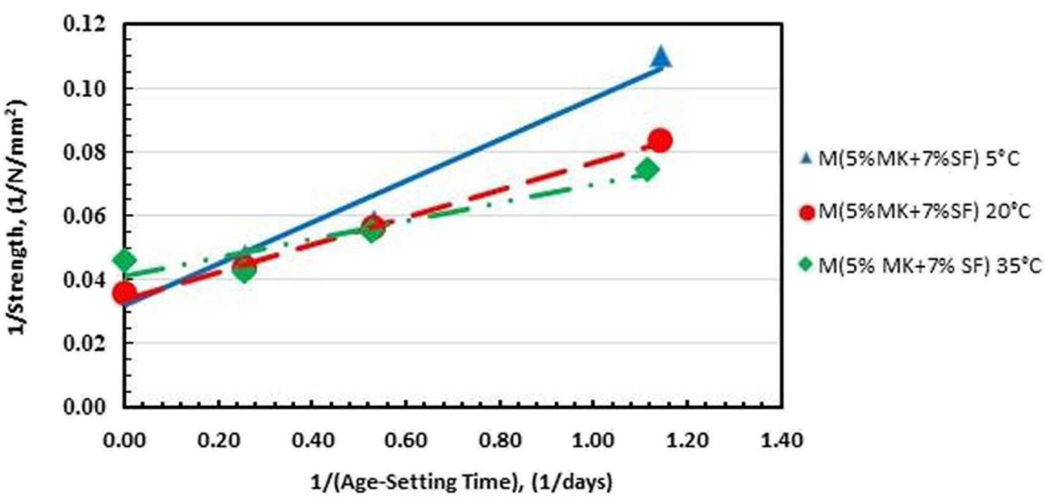

Figure 6: Reciprocal strength *versus* reciprocal age beyond time of final setting of the M(5%MK + 7%SF) mixes.

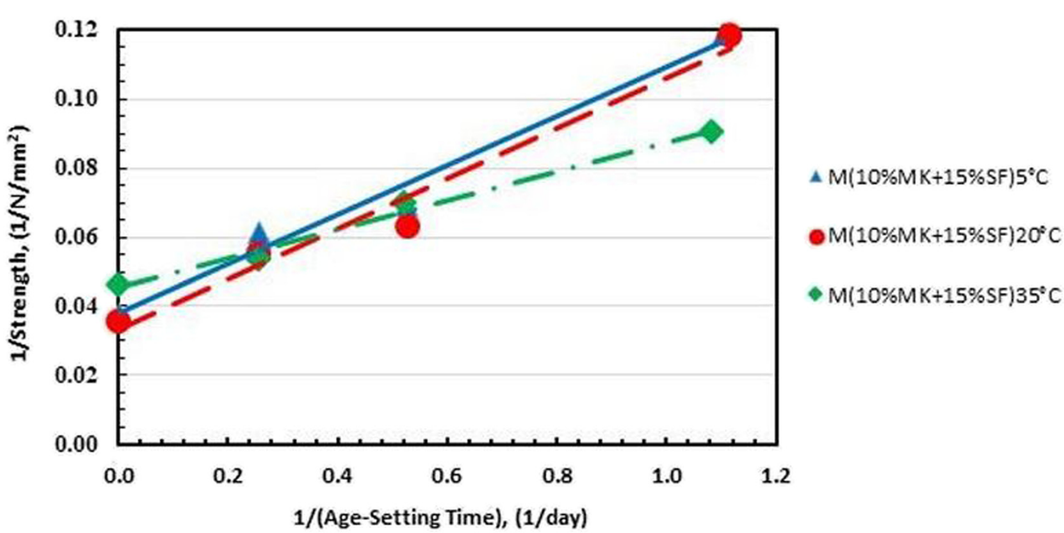

Figure 7: Reciprocal strength *versus* reciprocal age beyond time of final setting of M(10%MK + 15%SF) mixes.

energy at the final setting time in all mixes was higher than the activation energy at the initial setting time. The activation energy for the mixes containing 12% pozzolanic materials was the highest in both initial and final setting times, while the activation energy for the mixes containing 25% pozzolanic materials was the least in both initial and final setting times, for the reason explained previously [3].

3.2 Activation energy in mortar

The compressive strength values of mortar mixes at various ages and curing different temperatures are displaced in Figure 4. The strength of 12% pozzolanic mortar exhibited slightly higher strength than other mortar, especially at later ages. The strength of all mixes increased with increasing temperature. A rise in temperature leads to a corresponding increase in the number of molecules that have sufficient kinetic energy to start the reaction. During cement hydration, several simultaneous reactions take place, and the temperature has an influence on all of these reactions [25]. The calculation of the activation energy of mortar mixes relies on the increase in strength observed over time in the specimens. The correlation between the reciprocal of strength and age of each specimen was determined at three temperatures, 5, 20, and 35°C, and presented in Figures 5–7 for three different mixes: M(control), M(5% MK + 7%SF), and M(10%MK + 15%SF), respectively. The study utilized the setting time of each paste in the calculation as the setting time of the corresponding mortar mix, since the aggregate does not impact the activation energy [10]. The strength development rate (*k*-value) of the mortar mixes was estimated, following ASTM C1074, by dividing the intercept value of the best-fitting line through the data of the reciprocal strength axis, as shown in Figures 4–6, by the slope of that line, for each temperature. This is listed in Table 5 and illustrated in Figure 8. The relationship between the logarithmic *k*-value and the reciprocal of temperature in Kelvin is illustrated in Figure 9. From the last figure, the negative slope (Q) of the fitting line passing through the three *k*-values of each mix was multiplied by the gas constant (R) to determine the activation energy (E_a), according to Eq. (1) ASTM C1074 [4], and documented in Table 5.

The results showed that the rate of strength development (*k*-value) increased with increasing the replacement level of pozzolanic materials in mixes at all temperatures. On the other hand, the activation energy (E_a) decreased with increasing the replacement level of pozzolanic materials because of the high specific surface area of these materials, which makes them suitable as fillers rather than activation materials, as mentioned in [3].

Table 5: Compressive strength of mortar for different temperatures and ages with strength development rate (*k*-values) and activation energy (E_a)

Compressive strength (N/mm²)			
	Age		
Mix	**1 day**	**2 days**	**3 days**
Curing temp.	**5°C**		
M(control)	10.33	15.16	20.01
M(5% MK + 7% SF)	9.08	17.13	21.14
M(10% MK + 15% SF)	8.43	14.47	16.37
Curing temp.	**20°C**		
M(control)	12	17.8	21.38
M(5% MK + 7% SF)	11.96	17.9	22.93
M(10% MK + 15% SF)	8.47	15.86	18.19
Curing temp.	**35°C**		
M(control)	14.83	17.42	19.96
M(5% MK + 7% SF)	13.38	18.13	23.41
M(10% MK + 15% SF)	11.07	14.29	18.55

Curing temp.	**Rate of strength development (*k*-value) % (1/day)**		
Mix	**5°C**	**20°C**	**35°C**
M(control)	75	97	155
M(5% MK + 7% SF)	34	70	126
M(10% MK + 15% SF)	55	75	150

Mix	**Activation energy (E_a) (J/mol)**
M(control)	18,441
M(5% MK + 7% SF)	31,446
M(10% MK + 15% SF)	23,800

3.3 Utilizing the activation energy to estimate the concrete strength

Three distinct concrete mixes were subjected to compressive strength tests after being cured at 20°C for various durations of 1, 3, 7, 28, and 56 days. The results of these tests are depicted in Figure 9. During the initial ages, up to 7 days, the strength of the control specimens was about equivalent to the strength of the specimens containing 5% MK and 7% SF. However, after 28 days, the strength of the latter mix increased slightly to reach approximately

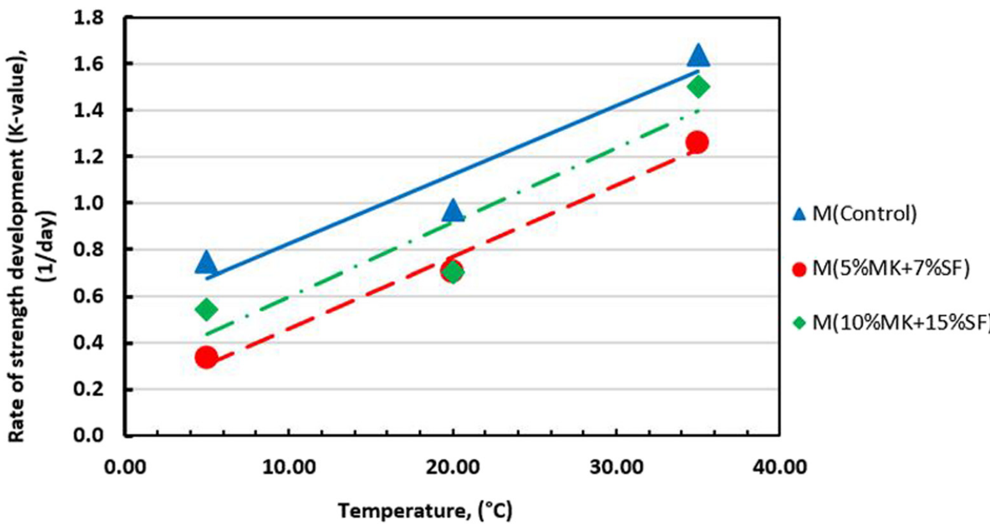

Figure 8: Rate of strength development (*k*-value) *versus* curing temperature.

8% at 56 days. Conversely, all specimens containing 10% MK and 15% SF had the lowest strength among all ages. The effect of the replacement level of pozzolanic materials on concrete strength increased with increasing the age of the concrete. The results indicate that using 12% of pozzolanic materials, as opposed to 25%, can lead to a more pronounced improvement in concrete strength at all stages of strength development. The high percentage of pozzolanic materials, when combined with a specific w/c ratio, could have an inverse impact on the strength of concrete, which may effect as a filler in the mixes because of its high surface area, as mentioned in Yogiraj and Wang (Figure 10) [3].

The present study used the activation energy, represented by *k*-value, to estimate the concrete strength development from the early hours of maturity under standardized conditions, using Eq. (2), reported in ASTM C1074 [4], and then compared to the actual strength, as shown in Table 6. The results in the table show that utilizing the *k*-value of mortar results in the least difference in strength compared to the actual strength for all mixes at all ages; however, utilizing the *k*-value of the final setting time of cement paste at early age produced less difference strength compared with the actual than utilizing *k*-value of initial setting time. Figure 11 represents the estimating and actual strength of concrete mixes at all ages.

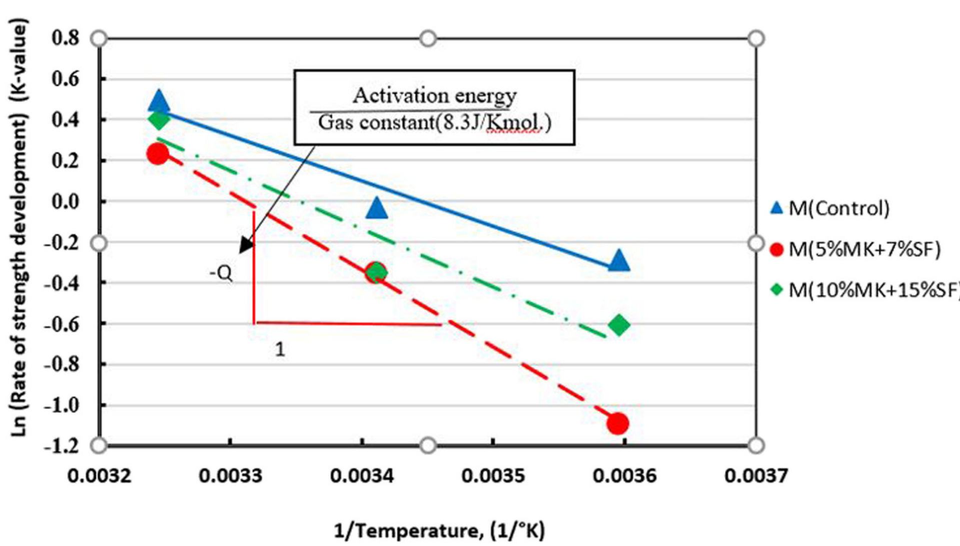

Figure 9: Natural logarithm of *k*-value *versus* the inverse absolute temperature.

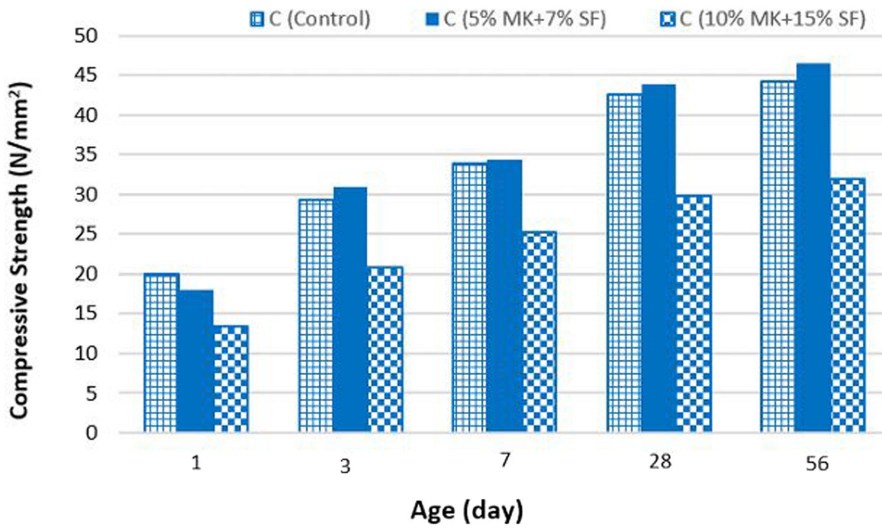

Figure 10: Compressive strength of all concrete mixes at different ages.

Table 6: Comparison between the actual and estimated strength based on the *k*-value for concrete mixes

Mixes	Age								
	% Difference between estimated strength from (Eq. (2)), as per ASTM C1074, and actual strength of concrete								
	Early ages						Later ages		
	1 day			3 days			7 days	28 days	56 days
	k-value is calculated from			*k*-value is calculated from			*k*-value is calculated from		
	(Paste) Initial set	(Paste) Final set	(Mortar) Strength	(Paste) Initial set	(Paste) Final set	(Mortar) Strength	(Mortar) Strength	(Mortar) Strength	(Mortar) Strength
C(control)	−19.5	18	3.5	−9.5	12.7	−6.6	−8.9	3.5	5.5
C(5% MK + 7% SF)	−43.5	−10.6	7.5	−9.4	5.9	−5.3	−5.3	4.8	8.2
C(10% MK + 15% SF)	−37.5	7.2	11.2	−13.8	9.5	2.2	0.9	4.5	9.1

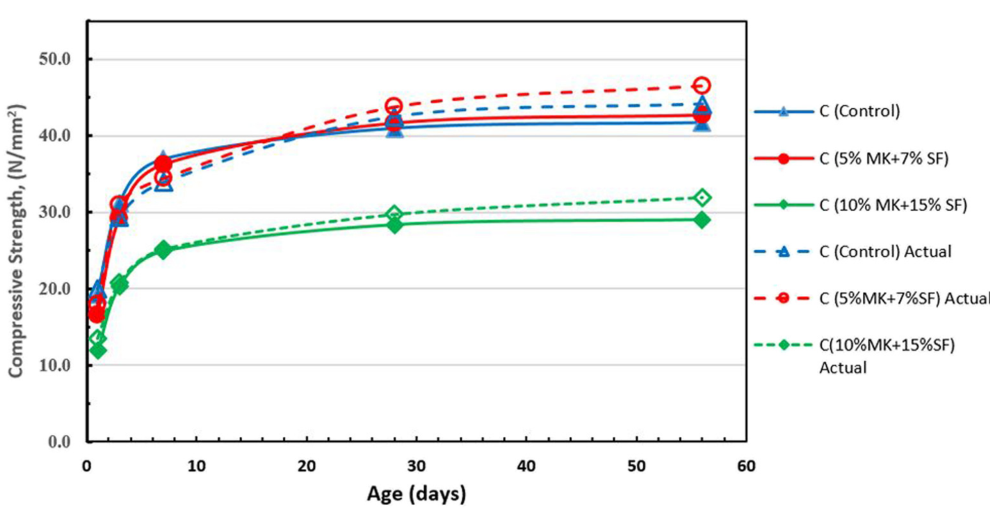

Figure 11: Comparison between estimating and actual strength of concrete mixes.

$$S = S_u + \frac{k(t - t_o)}{1 + k(t - t_o)}, \quad (2)$$

where S = cube compressive strength at age t (N/mm^2), t = test age (days), S_u = ultimate strength at 56 days (N/mm^2), t_o = age when strength development is assumed to start (days), and k = rate of strength development (1/day).

The results show a satisfied agreement between the actual and estimated strength over all ages; resulting in the ability to use the activation energy to estimate concrete strength at any age.

4 Conclusions

The impact of using locally pozzolanic materials in two replacements, 12 and 25% of cement weight, on the rate of strength development (k-value) and activation energy (E_a) was evaluated on paste, mortar at different curing temperatures of 5, 20, and 35°C. Then, it was evaluated on concrete mixes at a standard curing temperature of 20°C only. The study resulted in the following conclusions:

1. The rate of strength development (k-value) increases with the increase in pozzolanic materials content in all mixes.
2. The rate of strength development (k-value) significantly increases with elevated temperature, and the increase is more observed with high temperature.
3. The variability of the rate of strength development (k-value) is nonproportionate with the activation energy (E_a). The highest activation energy is obtained with a 12% replacement level of pozzolanic materials; while the highest k-value is observed at 0%. Thus, these materials are the main factor in retarding the rate of strength development.
4. The high activation energy (E_a) of the mixes containing a 12% replacement level of pozzolanic materials is a substantial indicator for strength development in the later ages, reflecting the impact of these materials in enhancing strength, especially at later ages.
5. The activation energy (E_a) is reduced with high replacement of pozzolanic materials due to the filler effect of these materials with high surface area. However, the E_a was the least in 0% replacement.
6. The ability to use Eq. (A1.1) as designated in ASTM C1074, to estimate the early and later strength based on the rate of strength development (k-value) and activation energy (E_a), is a vital approach because of the satisfied correlation between the actual and estimated strength for all concrete mixes.

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