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Chemical and Thermal Characterization of Cement Mortar Containing Ground Palm Oil Fuel Ash as a Partial Cement Replacement

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Abstract: This study investigates the influence of using ground palm oil fuel ash (G-POFA) from 10%-30% as cement replacement (by weight) on the cement mortar's pH under various curing conditions. These findings were supplemented by thermal gravimetric analysis (TGA). Moreover, the resistance of G-POFA blended cement mortars to water absorption and sorptivity was determined. Further, the *k*-value test was carried out to explain the pozzolanic and filler behavior of G-POFA and to support the results obtained from TGA. It was found that there was no significant impact of several curing conditions on the pH of mortars. The mortar with 10% G-POFA in replacement of cement (G-POFA-10) exhibited the best resistance against water absorption and sorptivity.

Key words: curing condition; Ca(OH)₂ content; *k*-value; pH; palm oil fuel ash

1 Introduction

Naturally, the pH of CBMs such as mortar, paste and concrete is high from about 12.5 to 13.5^[1]. The presence of alkali metals content and oxide minerals of portlandite are primarily responsible for the high pH of concrete^[2]. The high pH of concrete provides good protection to its reinforcing steel bars against corrosion and other defect causing agents, such as concrete surface cracking and spalling of concrete cover^[1]. However, the concrete's pH doesn't stay steady, and it might change because of the entrance of carbon dioxide, chlorides and water permeability. Both low and high pH values of concrete exhibit negative impact on the strength, durability and service life of concrete buildings^[3].

The POFA is a loss as debris acquired by consuming palm oil deposits as a fuel in the palm oil industry furnace^[4]. According to the research^[5], generally, the palm oil residues consist of 85% palm oil fiber and 15% palm kernel shell. Moreover, about 5% ash is produced by weight of the solid waste by palm oil mill boiler.

Various types of POFA have potential to be used as supplementary cementitious materials (SCMs) in CBMs^[6]. According to the research reports^[5,7,8], the optimum replacement level of ground palm oil fuel ash (G-POFA) is 20% and it should not be more than 30%. The use of high substitution levels of G-POFA reduces the early-age compressive strength and increases the demand of water in CBMs. However, the limitations of using G-POFA can be controlled by its heat treatment and grinding again after heat treatment to gain particles with higher fineness and improved pozzolanic properties than G-POFA. This newly developed type of POFA is called treated POFA (T-POFA). The permissible limit of T-POFA in CBMs is about 50%-70%^[9,10].

A significant contribution of different types of POFA in CBMs can be found in the literature. However, there are still many chemical and thermal char-

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acteristics to be studied in detail. Among the chemical properties, the pH as one of the important factors influencing durability of concrete structures, was not reported for POFA blended CBMs. There is lacking in information about the influence of different curing conditions on the pH of CBMs. Study on the pH of POFA blended CBMs is very important due to the reduction in pH of CBMs incorporating SCMs^[11-13]. The reduction in pH of concrete containing POFA will significantly reduce the life serviceability of concrete structures^[14,15].

This study is aimed at investigating the effect of G-POFA incorporation (10%-30% by weight). TGA has been employed to confirm the findings of pH analysis. The resistance of G-POFA blended cement mortars to the surface water absorption and water absorption through capillary suction (sorptivity) was determined. Further, the *k*-value test was carried out to explain the pozzolanic and filler behaviour of G-POFA to support the results obtained from TGA.

2 Experimental

2.1 Materials

The OPC owing 3.12 specific gravity was used for all the mortar mixes. The OPC's specific surface area (SSA) was measured to be 2 667.24 cm²/gm, using Brunauer, Emmett and Teller (BET) test. The POFA used in this study was G-POFA. The raw POFA was acquired from a factory situated in Selangor (Malaysia). Moisture was removed from the raw POFA by heating it in an oven at 105 ± 5 °C for 24 hours. The coarse residues of POFA were separated by sieving with 300 mm sieve. Then, this POFA was crushed in a Los Angeles machine to decrease the particle size^[16,17]. The machine time was set for 16 hours for grinding 6-8 kg POFA, using an electric motor (33 rpm). The SSA of G-POFA based on BET test method was 4 940 cm²/gm. The specific gravity of G-POFA was measured to be 1.81.

Locally available mining sand having grain size of 4.75 mm (maximum) and specific gravity of 2.68 was used as the fine aggregate. To get good workability for cement mortars, an aqueous solution of modified polycarboxylate copolymers was used as superplasticizer (SP) with density of 1.09 kg/m³. The water from the

pipeline of the laboratory was utilized to mix and cure the specimens. The specifications of OPC and G-POFA are given in Table 1. X-ray fluorescence spectrometry (XRF) was used to determine the elemental composition of OPC and G-POFA. The loss on ignition (LOI) was determined by heating the samples of OPC and G-POFA at temperature of 900-1 000 °C. The chemical composition and LOI of OPC and G-POFA are presented in Table 2.

Table 1 Physical properties of OPC, sand and G-POFA

Sample	Color	SSA according to BET test/(cm ² /gm)	Specific gravity	pH
OPC	Grey	2 667.24	3.12	12.7
Sand	White	-	2.68	7.6
G-POFA	Dark grey	4 940	1.81	10.4

2.2 Mixing proportions and processes

In total, four different mortar mixes were produced. The OPC was replaced with 10%, 20%, and 30% of G-POFA by weight in a control mortar with cement to sand proportion (*c/s*) of 1:3 and a water to binder proportion (*w/b*) of 0.48. The mix proportions of all mortars in one batch are displayed in Table 3. The flow of the OPC control and all mortars containing G-POFA was 220±10 mm. SP, on the other hand, was used to ensure that all mortar mixes flowed the same way. Same flow was achieved due to the same application, workability, and usage of all the prepared mortar mixes.

Binder and sand were dry blended for two minutes prior to being joined. The SP compound was then added to the mixture, along with around 70% of the mixing water, and the mixing was maintained for another 3 minutes. The remaining water was then added to the mixture, and it was mixed for another 5 minutes to achieve uniform blending. Then, the workability was evaluated using flow table test.

For pH and TGA testing, samples were prepared in 50 mm cube metallic moulds. 100 mm cubes were cast for water absorption and sorptivity testing. Cylinders of 100 mm length, 200 mm height, and 100 mm diameter were manufactured for the *k*-value test. The moulds were all made in two layers. A vibrating table was used to compress each layer. All the specimens

Table 2 Chemical composition of OPC and G-POFA along with LOI/wt%

Sample	SiO ₂	CaO	Al ₂ O ₃	MgO	Fe ₂ O ₃	P ₂ O ₅	MnO	K ₂ O	TiO ₂	SO ₃	SrO	LOI
OPC	20.14	60.82	3.89	3.10	3.35	0.064	0.14	0.24	0.16	2.25	0.02	2.3
G-POFA	59.17	5.80	3.73	4.87	6.33	-	-	8.25	-	0.72	-	16.1

Table 3 Mix proportions of all mortars in a single batch

Mix name	Binder <i>b</i>		Sand/(kg/m ³)	<i>b/s</i>	Water/(kg/m ³)	<i>w/b</i>	SP/(% of binder)
	OPC/(kg/m ³)	G-POFA/(kg/m ³)					
Control	12.50	-	37.5	0.33	6	0.48	1
G-POFA-10	11.25	1.25	37.50	0.33	6	0.48	0.6
G-POFA-20	10.00	2.5	37.50	0.33	6	0.48	1.2
G-POFA-30	8.75	3.75	37.50	0.33	6	0.48	1.8

were demolded one day after casting.

The 50 mm cubes for pH and TGA tests were cured in four different curing conditions such as water curing at room temperature (WC), curing in air at room temperature (AC), water curing at room temperature after hot water curing at 60 °C for 20 hours (HWC) and curing in air at room temperature after hot water curing at 60 °C for 20 hours (HAC). The curing was continued until the samples were used for tests.

The cylinders for *k*-value test were cured in three different curing conditions. These curing conditions included WC, AC and PC (water curing at room temperature for 7 days after demolding and then air curing at room temperature). The 100 mm cubes for water absorption and sorptivity tests were cured under WC and PC curing conditions. The curing was continued until the samples were used for tests.

At the ages of 2, 28 and 150 days, pH and TGA testing were performed. TGA analysis was performed on the control and G-POFA-30 mixtures. It was due to the reason that 30% replacement level of SCMs with OPC is more practical and common in construction practices^[18]. The age of 2-days was considered to see the effect of hot water curing at 60 °C for 20 hours on the pH and Ca(OH)₂ contents of mortar mixes. Moreover, the later age of 150-days was considered for pH and TGA testing due to the maturity of specimens. It is also expected that there are no significant changes in the chemical properties of mixes at this age. In addition, understanding of the pH of CBMs at later ages is very important to analyze the durability of materials. The water absorption, sorptivity and *k*-value tests were conducted at the age of 56 days. This age was selected due to the reason that samples should get maturity at this age.

2.3 Test methods

2.3.1 Flow table test

The workability of mixes was checked using a flow table test. With a flow of 220±10 mm, all the mixes were kept at an acceptable workability. The average

of at least three tested samples was used to obtain the pH value for each mix.

2.3.2 pH measurement

After crushing the specimens with a compression machine, the inside portions of the cubes for each curing condition were collected for pH measurements. The chosen samples were then crushed using a grinding machine^[19]. The pH was determined using 20 g of produced powder. This powder was mixed with 40 g water (dilution ratio of 1:2) as recommended by the research^[20]. This solution was stirred for 15 minutes using a magnetic stirrer. Then, the solution was filtered using number 40 filter paper with 110 mm diameter. Finally, at least three pH readings were taken by digital pH meter. The prepared solution was not stirred during the measurement process. For each time of the pH reading, the pH value was recorded after the pH meter displayed a stable reading. It ought to be noticed that the digital pH meter was calibrated before each pH measurement using two buffer solutions of pH 7.01 and 4.01^[21].

2.3.3 Thermal gravimetric analysis (TGA) test

At the ages of 2, 28 and 150 days, the Ca(OH)₂ content of control and G-POFA-30 was evaluated using TGA. The TGA testing is used to measure the changes in weight, in relation to the changes in temperature. It gives the most prominent point of weight loss, the decomposition rate and measures of the mass loss steps precisely. The obtained curves comprised of mass loss and derivative weight. During testing, sample of around 100 mg was heated at 10 °C/min from about 30 to 1 000 °C in the nitrogen atmosphere at constant rate of 20 mL/min. According to several research reports^[22-24], the Ca(OH)₂ contents are measured from the percentage weight loss between 300-550 °C.

2.3.4 Water absorption test

The initial (30 min) and final (72 h) water absorption test was conducted for all mortars cured under WC, AC and PC curing conditions. This test was done according to Ref.[25] to find the ability of mortars to resist water penetration. At first, initial volume, weight

and density of cubes were measured. Each mix's measured water absorption value was based on the average of at least three tested samples. Then, all the samples were oven dried at a temperature of 105 ± 5 °C for 24 hours. After taking out from oven, the samples were cooled to measure oven dry weight and density. The samples were then immersed in water for 30 min to measure initial water absorption. Following that, all of the samples were submerged in water for 72 hours and their saturated surface dry weight and density were determined. The percentage increase in weight of specimens after water absorption was estimated using the equation below.

$$\text{Water absorption} = \frac{M_a - M_0}{M_0} \times 100 \quad (1)$$

where M_a is mass of sample after immersion in water (g), and M_0 is mass of oven dried sample (g)

2.3.5 Sorptivity test

The sorptivity test was carried out to determine the sensitivity of all mixes to the penetration of water by capillary action. The test determined the increase in water depth, resulting from water absorption when only one surface of the cube was exposed to the water. The sorptivity test was performed according to the guidelines stated in ASTM standards^[26]. Fig.1 demonstrates the testing setup.

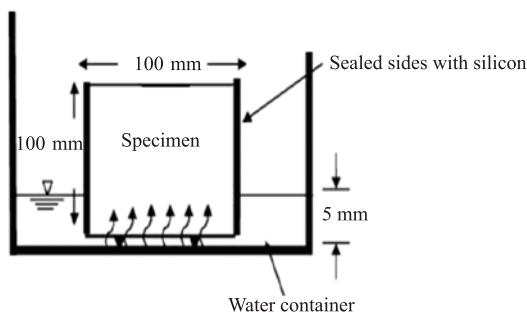


Fig.1 Sorptivity test setup

Each mix's value was calculated as the average of at least three samples. All of the samples were first oven dried for 24 hours at 105 °C. After taking out from oven, the samples were cooled and their oven dry weight was measured. Then, the samples were prepared for test in such a way that the side of sample to be immersed in water was clear. All other sides of the sample were coated with water-proof silicone to prevent water intaking from other sides of the sample. Then, water was filled in a plastic tray. Some steel rods with 6 mm diameter were kept in tray to keep the samples about 5 mm high from the base of tray. The depth of water lev-

el in tray was maintained up to about 1-2 mm.

After putting all samples in tray, the water intake rate was monitored by measuring weight of samples at regular intervals of 1, 5, 10, 20, 30, 60, 120, 180, 240, 300 and 360 minutes (0-6 hours). The surface of sample dipped in water was wiped out with a soft piece of cloth to remove excessive water before measuring weight. After measuring weight, the samples were put back immediately in the tray to maintain the uniformity of water intake with time. A graph between weight of sample (W_s) at any time (t) and \sqrt{t} was plotted for each mix. The slope of graph yielded the sorptivity co-efficient. The following equation was used for the calculations.

$$i = \frac{M_t - M_0}{Ad} \times 10^3 \text{ mm}^3/\text{mm}^2 \quad (2)$$

where i is cumulative absorption; M_t is mass of sample at time t (g); M_0 is mass of oven dried sample (g); A is cross-sectional area of the surface of sample (mm^2); d is density of water (kg/m^3).

2.3.6 Thermal conductivity (k -value) measurement

The k -value of all samples was calculated using the KD2-PRO analyser and the TR1 needle. TR1 sensor (2.4 mm diameter, 100 mm length) can detect k -values between 0.1 and 4.0 W/m.K. To produce a hole of the size of the TR1 sensor, a pilot pin was placed into newly cast cylinders. Thermal grease was used in the hole to ensure the contact between the needle and specimen. The average of at least three tested samples determined a mortar's k -value.

3 Results and discussion

3.1 The pH values of OPC control and G-POFA blended cement mortars

Table 4 shows the pH test results for all mortar mixes at the ages of 2, 28, and 150 days. According to the results, the pH of control mix was more than all blended cement mortars, containing various amounts of G-POFA as previously described by the researchers^[11-13]. There was a decreasing trend in pH of control and all blended cement mortars from 2-150 days.

The results showed that there was no significant effect of various curing conditions on the pH of mortars. The highest difference in pH of all mortars was found to be 2.6% due to the curing conditions of WC, AC, HWC and HAC, as can be seen in the mix of G-POFA-10 at 150 days. All the results agreed with the previous studies^[27,28] describing that the use of SCMs

reduces high alkalinity, fills the voids and hardens the matrix.

Table 4 The pH results of all mortars at 2, 28 and 150 days

Mix name	Curing condition	pH		
		2 day	28 day	150 day
Control	WC	12.6	12.3	12.1
	AC	12.6	12.4	12.0
	HWC	12.5	12.3	12.1
	HAC	-	12.4	12.2
G-POFA-10	WC	12.5	12.1	11.6
	AC	12.5	12.0	11.8
	HWC	12.4	12.0	11.9
	HAC	-	12.0	11.8
G-POFA-20	WC	12.5	12.1	11.8
	AC	12.5	12.0	11.6
	HWC	12.4	12.0	11.7
	HAC	-	11.9	11.6
G-POFA-30	WC	12.5	12.2	12.1
	AC	12.5	12.2	12.2
	HWC	12.4	12.3	12.2
	HAC	-	12.4	12.3

It is clear from the findings that the obtained pH values of all mixes were in the safe and essential range for passivation of steel rebar^[29], protection from carbonation, biodegradation^[30] and chloride ingress^[31]. It should be noted that even a small reduction in pH of CBMs is of great importance since the pH of 11.5-13 is the safe range for the formation of portlandite^[2,30,32-36].

3.2 Ca(OH)₂ contents in OPC control and G-POFA-30 mixes from TGA test

Table 5 Ca(OH)₂ contents from TGA of control and G-POFA-30 mixes at the ages of 2, 28 and 150 days

Mix name	Curing condition	Ca(OH) ₂ by TGA testing/%		
		2 day	28 day	150 day
Control	WC	1.45	1.69	2.00
	HWC	1.47	-	-
	HAC	-	1.81	2.20
G-POFA-30	WC	1.40	1.34	1.11
	HWC	1.43	-	-
	HAC	-	1.37	1.31

For the determination of filler and pozzolanic effect of OPC and G-POFA-30 mortars, TGA test was conducted for the WC and HWC curing conditions at the age of 2 days, and WC and HAC curing conditions at 28 and 150 days. Table 5 summarises the TGA data for Ca(OH)₂ content in mortars. The results revealed that the Ca(OH)₂ level of the control mix was higher

than the G-POFA-30 mix and enhanced with curing age. It was due to the higher amount of OPC in control mix than in G-POFA-30. The Ca(OH)₂ was produced by hydration of OPC through reaction of di-calcium silicate (C₂S) and tri-calcium silicate (C₃S)^[37].

Ca(OH)₂ concentration decreased with increased curing time in the G-POFA-30 mix, according to TGA data. It was owing to its consumption in the pozzolanic reaction, which resulted in the formation of the C-S-H gel. Furthermore, the reduction was attributable to cement dilution, which resulted in less Ca(OH)₂ production during the cement hydration process. The pozzolanic reaction of G-POFA increased with curing time under all curing conditions, as shown by the data.

3.3 Water absorption

The rate at which dry surface of a mortar absorbs water can be considered as an indicator of its durability^[38]. The results of water absorption in 30 min (initial water absorption) and after 72 h (final water absorption) for all mixes under WC and PC curing conditions are given in Figs.2 and 3, respectively. According to the research^[39], the quality of mortar and concrete was classified as poor, average and good for initial water absorption of 5% and above, 3%-5% and 0%-3%, respectively. For final water absorption, all the mortars and concretes having values up to 5% were categorized as good quality. According to the research^[40], most of the good quality concretes and mortars have water absorption less than 10%. The obtained results showed that all the mixes were of good quality according to the above standards.

It is clear from the results that the quality of OPC mortar in terms of water absorption increased by addition of G-POFA in cement replacement due to its more considerable pozzolanic reaction. However, the quality of G-POFA-10 mix significantly improved as compared to G-POFA-20 and G-POFA-30 mixes in terms of water absorption in both curing conditions. Overall, all the mortars showed less water absorption under WC curing condition. The high water absorption values can be attributed to the more porosity and slow pozzolanic reaction that results in less amount of hydration products. Both initial and final water absorption were measured at the age of 56 days, since at this age the pore structure of blended cement mortars is densified by more hydration products than that at initial ages of 2 and 28 days.

3.4 Sorptivity

The sorptivity results of all mortar mixes under WC and PC curing conditions are shown in Figs.4 and 5, respectively. The sorptivity of each mix was calculated

using the slope of line best fit to the points between 0-6 hours. The results showed that G-POFA-10 mix had the lowest sorptivity under WC curing condition among all mixes. It is due to the pozzolanic reaction of G-POFA which produced a denser microstructure resulting in reduction of sorptivity compared to all other mixtures^[41].

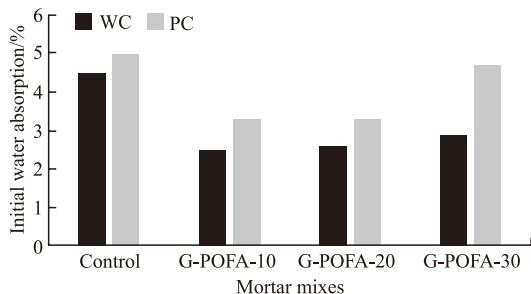


Fig.2 Initial water absorption for all mixes under WC and PC curing conditions at the age of 56 days

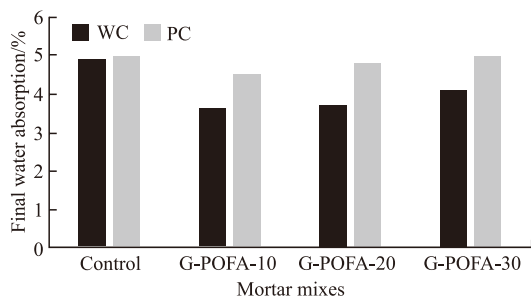


Fig.3 Final water absorption for all mixes under WC and PC curing conditions at the age of 56 days

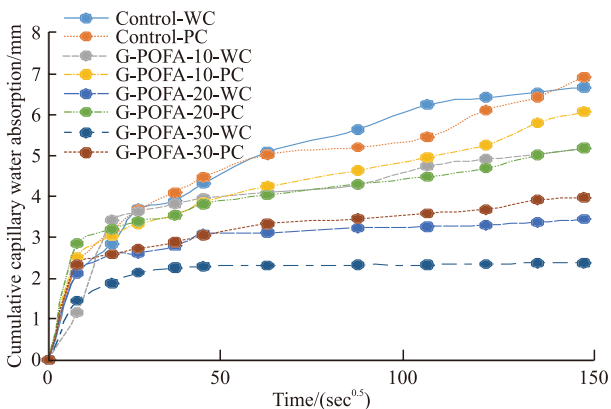


Fig.4 Cumulative capillary water absorption for all mixes under WC and PC curing conditions at the age of 56 days

The sorptivity of control mix was higher than all mixes under both curing conditions which showed its porous structure due to CSH gel^[42]. The improved behavior of G-POFA mixes in terms of water absorption and sorptivity is a result of high SiO₂ contents than control, that yields additional CSH gel resulting in dense microstructure^[43]. Overall, the mixes showed improved behavior in terms of sorptivity under WC curing condition.

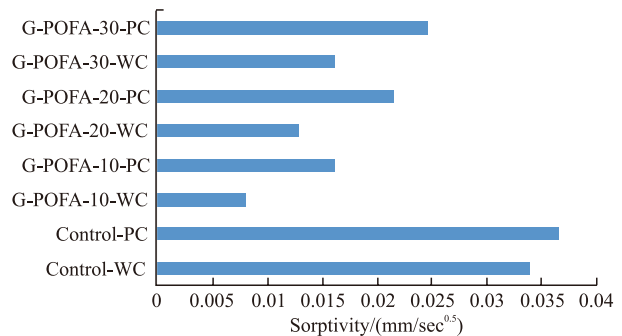


Fig.5 Sorptivity of all mixes under WC and PC curing conditions at the age of 56 days

3.5 Thermal conductivity (*k*-value)

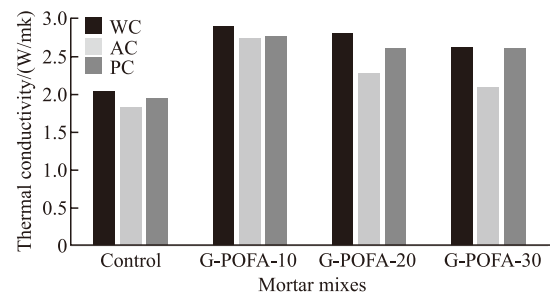


Fig.6 Thermal conductivity of all mortars at the age of 56 days

The *k*-values of all mixes at the age of 56 days are shown in Fig.6. The results showed that the *k*-value of control mortar was less than all G-POFA blended cement mortars due to the existence of more CSH gel and consequently more porous structure existing in the CSH gel. The *k*-values increased with percentage replacement of G-POFA in blended mixes due to pozzolanic reaction of active silica-alumina in G-POFA particles that increased the packing of cement matrix and reduced the porosity. Thus, it can be concluded that reduction of porosity resulted in increased *k*-value.

4 Conclusions

The aim of this study was to investigate the chemical (pH, water absorption and sorptivity) and thermal (*k*-value) properties of G-POFA blended cement mortars under different curing conditions. For this purpose, OPC cement was replaced with 0, 10%, 20% and 30% (by weight) of G-POFA. Samples were produced at various curing conditions, including WC, AC, PC, HWC, and HAC. The pH values of these mortars were measured at the ages of 2, 28 and 150 days. The relation between pH and Ca(OH)₂ contents was investigated in detail. Therefore, at 56 days, the water absorption, sorptivity, and *k*-value tests were performed. Based on the test results, the following conclusions can be drawn.

a) There was a decreasing trend in pH of control and all blended cement mortars from 2-150 days.

Therefore, to examine the pH of cement-based materials (CBMs), its monitoring and measurement should be for both short and long terms.

b) There was no significant effect of various curing conditions on the pH of mortars.

c) The difference in pH of all mortars was the highest, *i e.*, 2.6% due to the curing conditions of WC, AC, HWC and HAC in case of G-POFA-10 at 150 days.

d) The mortar with 10% G-POFA in replacement of cement (G-POFA-10) showed the best resistance against water absorption and sorptivity.

e) All pure and blended cement mortars showed less water absorption and sorptivity under WC curing condition.

f) The *k*-value of control mortar was less than all G-POFA blended cement mortars due to the existence of more CSH gel and consequently more porous structure formation.

Conflict of interest

All authors declare that there are no competing interests.

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