Investigating the effect of elevated temperatures on the properties of mortar produced with volcanic ash

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1 **Investigating the effect of elevated temperatures on the properties of**
2 **mortar produced with volcanic ash**

3 **Abstract**

4 During the recent years, the use of pozzolanic materials (e.g. volcanic ash) in concrete
5 and cement manufacturing has increased significantly since it can reduce the
6 environment hazard associated with using Portland cement. In this paper, the effect of
7 elevated temperatures on the physical and mechanical characteristics of building mortar
8 produced with volcanic ash is experimentally explored. In order to evaluate the
9 performance of the mortar, four different proportions of volcanic ash (0, 5, 15 and 25%)
10 -as weight replacement of the cement- were prepared. A series of tests were conducted
11 after 28, 90 and 120 days under different temperatures (25, 200, 500 and 800C°). This
12 paper demonstrates that the replacement of cement by a proportion of volcanic ash can
13 sustain an acceptable level of compressive strength and improve the overall
14 characterization of the mortar while reducing the amount of CO₂ released. The mortar
15 with 15% ratio of the volcanic ash replacement showed better flexural and the tensile
16 strength. This paper also heights that the volcanic ash replacement affects the late-age
17 properties of the mortar more than the early-age ones at both ambient and elevated
18 temperatures.

19
20 **Keywords:** Elevated temperature, Volcanic ash, natural pozzolan, Mortar.
21

1. Introduction

Concrete exhibits superior resistance compared to more popular construction materials like steel and wood. Being non-combustible, concrete forms a barrier that prevents the further spread of fire, hence its use does not increase risk of fire in buildings[1]. Concrete from Portland cement is commonly utilized in the construction of buildings, as it is capable of satisfying the crucial need for safety from fire hazards, in a manner better than most of its competitor materials [2,3]. The change in concrete's temperature, and the changes stemming from its exposure to fire, have been a key interest for the researchers in the field of fire safety. The main ingredients of concrete, including the paste of cement, undergo a series of reactions of decomposition, which are in most cases permanent and cannot be reversed.

Additionally, previous studies have investigated some of the physical and mechanical changes of the mortar subjected to high temperatures. These studies included studying the influence of the compressive strength on the color change in mortars [4]; the influence of the temperature on pore structure; the concrete permeability; the mechanical properties of concrete of normal strength and also on concrete of high performance [5,6] and the behavior of spalling in structural elements like columns or beams [1,7–10] . The thermal response of concrete under elevated temperatures is dependent on the proportions used in its mix, and on the characteristics of its constituents [11]. Exposure to elevated temperatures is a critical process of physical deterioration, and one that inflicts direct effects on the durability of the structure of concrete. Upon exposure to elevated temperatures, concrete could exhibit detrimental failures in its structural integrity [9]. The major outcomes resulting from exposure to high level of temperatures include the changes that occur in the concrete color and spall, the losses in mass or weight of the concrete, and the deterioration in performance due to the degradation of the compressive strength. The building materials might be exposed to the effects of elevated temperatures during fire incidences, or inside reactors and when in proximity to furnaces. The deformation, the elastic modulus, aggregate interlock and bonds between the cement paste and the aggregate are also influenced by high temperatures [2].

The rate at which temperature increase is another factor affecting the type and extent of the changes occurring in the mechanical properties of concrete when subjected to high temperatures. When concrete is subjected to an elevated temperature ranging between 100 and 200°C, the evaporation of free water takes place slowly; hence structural damage does not occur. On the other hand, when heating takes place rapidly, the resulting higher vapor pressure leads to the formation of cracks in the surface morphology of the concrete. After the temperature reaches 400°C, the concrete was shown to begin to lose its compressive strength [12].

At temperatures ranging from 400 to 500°C, concrete was shown to exhibit a reduction in its compressive strength by approximately 15 to 70% of the value in concrete that had not been subjected to heating [3]. As the temperature is increased, the evaporation of the trapped water in the paste of the concrete starts to take place at around 400°C; therefore, leading to the dehydration of calcium silicate hydrate (CSH), which is the compound used in concrete mixture to achieve the bonding together the diverse components constituting the concrete paste. When dehydration takes place in the crystals of CSH, it was shown to irreversibly lower the strength of the concrete. At 530 C°, Ca(OH)_2 converts to CaO , resulting in a shrinkage in the volume of the concrete paste by approximately 33% [1,10].

Khoury *et al* reported significant changes in the chemical and physical structure of concrete [13], when exposed to high temperatures. They observed that when the binding

paste of concrete faces temperatures above 110°C, it causes the chemically bound water to be released from calcium silicate hydrates. Internal micro-cracks were reported to occur within the structure of the concrete, which was observed particularly when the temperature increased up to 300°C [14], which was due to dehydration of calcium silicate, and the expansion of the aggregates under high levels of temperature. The use of pozzolanic materials (e.g. volcanic ash) is becoming more appealing as they reduce the cost of cement, results in a better durability, retard reaction (important with hydraulic structures) and most importantly removes the waste product from environment [10,12,15]. There are limited studies on the effect of elevated temperature on the mortar containing natural volcanic ash; therefore, this paper aims at studying the physical and mechanical properties of mortar containing different percentages as a replacement for cement.

2. Description of Volcanic Ash in Concrete

Volcanic ash (rock dust) powder is a naturally existing volcanic rock that consists of more than 70 types of minerals. Volcanic ash rock dust powder Azomite is mined from ancient volcanic ash deposits in central region of the State of Utah, USA. It is used in many countries including China, Mexico, Germany, Italy, and Greece, to achieve the reduction in cost and improvement of the durability and the quality of concrete [16].

The use of volcanic ash is merited by its chemical or pyro chemical properties, also its physical properties, including the light color, friability, fine size and angularity of its particles. In ceramics, it is used as abrasive, in cellular blocks, concrete, and lightweight aggregates and in vitreous enamels and glass in general.

pozzolanic properties: volcanic ash is one of natural pozzolana, and has pozzolanic properties with silica oxide (SiO_2) content, which influences significantly the strength of the designed concrete [16].

One of the most significant aspects that would be affected by the addition of the volcanic ash to mixture is the development of the strength. The main factors that affects the development in the strength in mortar with volcanic ash are illustrated below [17,18]:

1. The volcanic ash as admixture
2. The volcanic ash as aggregate.
3. The fineness of the natural Pozzolana has a significant effect on early strength development.
4. The process of curing (and the related parameters) plays an important role in the development of strength of any binding material. Longer curing time (in addition to the type of curing) provides higher strength values than when the same concrete mix is cured for a shorter time period. Lime Pozzolana mixtures in particular, are affected by curing largely, because of their slow strength gain versus that of cement. The process and parameters of curing also affect the permeability of pozzolanic mixtures, which is reflected in the robustness of the structure [18].

The physical and chemical characteristics of the volcanic ash used in this study are shown in Table 4.

The pozzolanic behavior likability of the used volcanic ash was assessed based on the standard specification test ASTM C618. Chemical analysis results showed that the summation of the SiO_2 , Al_2O_3 , and Fe_2O_3 contain was 78.65%, which fulfills the minimum prerequisite of 70%, fixed by the standard specification [19]. Additionally, the amount of SO_3 is 0.21, and the loss on ignition (LOI) is 6.43 more than that of

cement, which is within the limits of the ASTM C618 [20]. It can also be noted that the strength activity index is utilized to assess the reactivity between the cement and the mineral admixture used.

3. Experimental Program

3.1. Material

In this section, the used materials including cement, fine aggregate, volcanic ash and chemical admixtures characteristics are explored as shown below.

A- Cement: Ordinary Portland Cement Type 1 (OPC) was used in this study (ASTM C150 – Type 1) [21]. The used cement was checked according to IOS 5:1984 [22] as shown in Tables 1 and 2 .

B- Aggregate: In this research, natural sand was used as a fine aggregate. The sand grading shown in Table 3, according to IOS No.45/1984 [23]. The test results for the physical and chemical properties of the sand used including absorption %, specific gravity, dry loose-unit weight kg/m^3 , and sulfate content were found to be 2.00%, SO_3 2.69, 1725kg/m^3 , and 0.24%, respectively.

C- Volcanic Ash (VA): The AZOMITE is a mineral composition came from the combination of seawater and fed by hundreds of rivers rich in minerals, which present in volcanic ash and this geologic characteristic is an outcropping known as a “hogback” (see Fig.(1)). Table 4 shows Chemical analysis of the volcanic ash.

D- Chemical Admixture: Glenium 51 (high range water reducing admixture) was used as a super-plasticizer for the mixture, Table 5 show the main characteristics of the product.

3.2. Mix design and sample preparation

According to ASTM C109 [24], all the test specimens were prepared then the standard procedure of all mixing was carefully performed according to ASTM C-305. The mix proportions used in this paper for cement: sand: W/C: Glenium 51 are 1: 2.75: 0.35:2-3%. Table 6 shows the four concrete mixtures with various proportions of VA ranging from 0%, 5%, 15%, and 25%. The choice of these ratios is according to the limited cement percentage to be replaced as adding more than 25% would results in a very low strength of the mortar [16]. To examine the effect of VA replacement for cement (for 28, 90 and 120 days of curing) the following tests were performed; compressive strength, flexural strength, ultrasonic pulse velocity, absorption, and weight loss, cubes 50 x50 x 50 mm, and prisms 40x40x160 mm.

4. Testing procedure and method

An experimental planner was designed to investigate the physical and mechanical characterization of blended cement mortar containing VA following exposure to elevated temperatures. For this purpose, the electric arc furnace shown in Fig. (2) was used. Four mixtures with 0 %, 5 %, 15, and 25 % VA as a replacement by weight for cement were prepared. A total of 192 cubes and 144 prisms were tested to examine the physical and mechanical properties of the blended cement mortar containing VA. During the first stage, the specimens were retained after casting in the molds for 24 hr. at a room temperature of 25°C. After de-molding the specimens, they were cured for 28, 90, and 120 days in water. In the second stage, the specimens were dried at 25°C° for 24 hours first, then exposed to different elevated temperatures (200, 500, and 800°C) for 1.5 hr. at a rate of 10 °C / min, as shown in Fig (3). At the last stage, the specimens

were left in the furnace to cool down gradually. Finally, the following tests were conducted:

4.1 Slump flow test

Slump flow test was followed the ASTM C1437 [25] for the mortar flow of all mixes and was performed by using the flow table equipment.

4.2 Absorption test

Absorption test followed the ASTM C642 testing standard [26]. The average water absorption of the three cubs for each mixes and ages was calculated following Equation 1 calculated according to ASTM C642 [26]:

$$\text{Absorption ratio} = \frac{(\text{wet weight} - \text{dry weight})}{\text{dry weight} \times 100} \dots\dots\dots \text{Equation 1}$$

4.3 Loss in Weight

The weight of each sample before and after exposing to elevated temperatures was measured. Weight loss was then calculated as shown in Equation (2) according to [11]

$$W_{\text{loss}} = \frac{\text{Standard weight} - \text{Weight at exposure temprature}}{\text{Standard weight} \times 100} \dots\dots\dots \text{Equation 2}$$

4.4 Compressive Strength

Compressive Strength test was performed according to ASTM C109/ C109M-05 testing standard [24] on 50×50×50 mm cubes, using a digital compression testing machine 2000 kN in capacity. The test was conducted at ages of 28, 90 and 120 days (three specimens were tested for each age).

4.5 Flexural strength

The flexural strength test was performed according to ASTM C348-02 testing standard [27], on 40×40×160 mm prisms, under central line loading using flexural testing machine 10 kN in capacity.

4.6 Ultrasonic pulse velocity

The ultrasonic pulse velocity [28] of three 40×40×160 mm prism specimens, was determined automatically by using a device that measured the time of ultrasonic waves to pass throughout the tested specimen length, from the wave transmitter to the receiver nozzle. Then, the Equation 3 was used to calculated the wave speed [28].

$$V = (h/t) \times 10^6 \dots\dots\dots \text{Equation 3}$$

5. Results and discussion

5.1. Flow of mortar

Since the fineness and the large surface area of pozzolan materials, might result in lower flow and higher water demand. This would lead to a lower workability with increasing percentage of VA. As a result of the fineness of pozzolan materials particles greater amount of water demand obtain to the desired consistency, and the reduction in flow in proportion to their amount [29]. Table 6 shows the flow of all mortar mixes, the VA-0 mix was 112% only when using 2% Glenium 51 as a super-plasticizer and to ensure

getting the same flow for all mixes the a higher percentage of the super-plasticizer was used, this is in line with [29] findings.

5.2. Absorption

Absorption results of specimens at 28 days age for all four tested sets under the effect of elevated temperatures is shown in Fig (4). The results demonstrate an increase in absorption with the increase of temperature and percentage of replacement of VA at 28 days age, except for the higher temperature case (500 and 800°C) where the control specimens exhibit the maximum absorption. Heating the specimen would increase the rate of absorption, as the water within the concrete would start evaporating after 100 °C [30]. The water within the hydrated cement can be classified into two categories; the evaporable water (water in the capillary pores and some water in the gel pores) and Non-evaporable water (nearly all chemically combined water and some water not held by chemical bonds)[18]. The higher absorption for the control mixture at high temperatures is an indicator that the mortar with a higher percentage of VA replacement is more durable than the normal mortar at post-fire condition as the higher water absorbed by mortar the less durable it becomes.

5.3. Loss in weight

Fig (5) to (7) demonstrate the effect of various elevated temperatures on the weight loss of all mixes. It can be seen that the unit weight values decrease with increasing temperature for all mixes, as a result of weight reduction taking place in the specimens after releasing their water contain [28]. Eventually, after the water being released from the cement paste, air void would start forming. The concrete specimens containing VA experienced higher weight losses than the control concrete specimens, this finding is in line with reference [10]. At temperature range between 20 to 200°C, the weight loss is fundamentally due to evaporation of moisture from the surface of concrete specimen to the atmosphere. The specimens exposed to 500 °C has shown the highest loss in weight that could be a result of the evaporation of residual moisture content retained at a temperature level of 200°C. Meanwhile, the degradation in the weight of specimen was lower at a temperature range of 200°C to 500°C compared to 500°C to 800°C. The evaporation of free water and CSH water in structure of CSH and subsequent decomposition of $\text{Ca}(\text{OH})_2$ can be the reasons of the percentage of loss in weight. It changes the stiffness and mechanical properties of the substance and might results in a lower compressive strength values [19].

The specimen with maximum percentage of VA replacement 25% (VA-25) has shown the maximum loss of water, which could be due to higher retention of water content in the presence of volcanic ash. It can also be noted from Figs (5) to (7) the relationship between the percentage of loss weight with ages. Moreover, it can be seen that the specimens cured for 120 days age have the highest percentage of loss weigh. Therefore, the increase in the percentage of loss weight was observed with increased specimens curing age and that can be due to the saturation of the specimens increased with the time of curing.

5.4. Compressive strength

The main components of the hydrated cement paste are Calcium Silicate Hydrate, Calcium Hydroxide, and Calcium Sulfate Aluminate Hydrate. Besides that, the saturated paste encloses a significant amount of free, capillary and gel water. Therefore, the effect of $\text{Ca}(\text{OH})_2$ can be reduced by using different pozzolans, such as fly ash, slag, silica fume, clay, and volcanic ash, as a replacement of cement in concrete. SiO_2 present

in these pozzolans reacts with Ca(OH)_2 , and a byproduct of hydration reaction forms calcium silicate hydrates. As a result, the amount of Ca(OH)_2 is reduced, and C-S-H is increased, which improves the performance at elevated temperatures[31,32].

Figs (8) to (10) displays the relationship between compressive strength and the effect of elevated temperatures for different percentages of replacement VA with cement. It can be seen that the compressive strength would increase with curing time; meanwhile, compressive strength decreases with increasing the amount of VA replacement. Therefore, the minimum value of compressive strength was for the mix with higher VA contain (25%). This makes sense since replacing the cement with VA reduces the cement content in the mix at short-term. However, looking at the long-term durability the pozzolanic action of VA helped to improve the strength [31], keeping in mind that the reduction of strength in blended cement due to the replacement is not compensated in the current study.

Figs (8) to (10) illustrate that the compressive strength increased between 25 to 200 °C, this can be due to the formation of Tobermorite (calcium silicate hydrate mineral), which is formed by reaction between un-hydrated VA particle at high temperature. At temperature range 200 to 500 °C, there is a decrease in the compressive strength for all specimens and for all curing ages, but the highest reduction occurred at 28 day (see Fig (11)). This reduction can be due to the pore structure roughening in concrete [33]. All mixes at 500–800 °C temperature show severe loss in compressive strength. The severe deterioration in mortar is with reason to the dissociation of CSH gel [32].

5.5. Flexural strength

Figs (12) to (14) show the relationship between the flexural strength and the effect of elevated temperatures, for different percentages of replacement VA with cement. All the mixes show improved flexural strength with curing time. The results show the decrease in the flexural strength with the increase in the amount of the VA replacement at 25 °C. The decrease in the flexural strength at 28 days were 13.34, 5.92 and 24.52% for VA-5, VA-15 and VA-25, receptively. Specimens with 15% VA as a replacement had the best result. Fig. 12 shows the effect of different temperatures on flexural strength at 800°C. The residual flexural strength was 75.74, 76.34, 87.53 and 87.85% for VA-0, VA-5, VA-15 and VA-25, receptively. The percentage of the residual flexural strength was calculated relatively to retained flexural strength with respect to the unheated specimen flexural strength (25 °C). At 90 day, the residual flexural strength values for specimens were 66.66, 79.71, 91.5 and 95.1%, for VA-0, VA-5, VA-15 and VA-25, receptivity. At 800 °C and at 120 day, the values were 64.2, 79.1, 89.0, and 90.38 for VA-0, VA-5, VA-15 and VA-25, receptivity. The loss of strength observed at higher temperatures might be attributed to the loss of bound water, increased porosity, and consequently, increasing permeability [28].

5.6. Ultrasonic pulse velocity

As seen in Fig. (15) to (17), the Ultrasonic pulse velocity (UPV) of the specimens that were used in the study deteriorated due to increasing temperature and percentage of VA replacement, resulting a lower in UPV (especially at 800 °C). This decrease in UPV is due to the degeneration of the CSH gel at above 500 °C temperatures, which causes would increase air voids in the specimens cement paste. This would slow down transmission speed of sound waves along of the specimens. The increase in UPV was noticed to be higher for VA-0 specimens at 500 °C and 800 °C, meanwhile the

maximum decrease in UPV was for VA-25. This reduction in the UPV can be due to the formation of a porous structure as a result of the decomposition the C-S-H gel [28].

6. Conclusions

This study has presented an experimental program set to explore the effect of elevated temperatures effects on the mechanical and physical properties of cement mortar containing volcanic ash. The results of 192 cubes and 144 prisms tested post-heating after exploring to four different temperatures including room temperature (25, 200, 500 and 800 °C). Three different weight replacements of the cement were considered (5, 15 and 25%) of the volcanic ash along with the control specimens. For the limitations for this research, the following conclusions can be drawn:

1. The addition of volcanic ash to the mixtures showed a limited reduction in the mechanical and physical properties, which indicates that it could be suitable to use as partial replacement of cement, paste and mortar to reduce the carbon emission resulting from the Portland cement reactions.
2. At 800°C, the control specimens exhibit a higher water absorption compared to the ones contain volcanic ash, which denote that they are more durable since the higher water absorbed by results in less durable mortar.
3. The concrete becomes more brittle after exposure to elevated temperatures due to the loss of water and show changes in color and density loss. When the temperature exceeded 500 °C, the fine cracks on concrete specimen appear on surface.
4. The compressive strength of mortar at 200°C heating was increased compared to the initial strength determined at 25°C, for all mixes due to the formation of Tobermorite.
5. At 500°C, the compressive strength decreased in all cases in relation to the value determined before post-heating. However, at 800°C, the maximum value of reduction in the compressive strength was below the value determined on unexposed specimens in all cases, with the maximum drop reaching 79.54 % for VA-0 at 28 days age, versus 59.32% and 52.76 for VA-25 and VA-0, at 90 days age. As the temperature, goes higher the bond within cement paste is weaken causing the mortar to be more brittle.
6. At temperatures above 500 °C, the amount of air voids in the specimens' were increased, due to the degeneration of the CSH gel and that decreases the compressive strength and flexural strength, and increases the weight loss.

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List of Tables

Table 1: Ordinary Portland cement (OPC) chemical composition according to ISO 5/1984

Oxides composition	Content%	ISO 5/1984
CaO Lime	61.27	-
Fe ₂ O ₃ Iron oxide	3.12	-
Al ₂ O ₃ Alumina	5.05	-
MgO Magnesia	2.06	<5.00
SiO ₂ Silica	21.27	-
SO ₃ Sulphate	2.07	<2.80
Loss of Ignition	3.21	<4.00
Insoluble residue	1.22	<1.5
Lime Saturation Factor, L.S.F.	0.98	0.66-1.02
C ₃ S Tricalcium Silicate	43.42	-
C ₂ S Diacalcium Silicate	28.31	-
C ₃ A Tricalcium Aluminate	8.11	-
C ₄ AF Tetracalcium Aaluminoferrite	9.48	-

Table 2: Ordinary Portland cement physical characteristics.

Physical characteristics	Experimental values
Setting time (min),	
Initial	3.20
Final	4:40
Compressive strength, MPa	
3- days	26.4
7- days	33.4
28-days	44
Soundness , %	0.19
Specific surface area, m ² /kg	330

Table 3: Sieve analysis of the used sand compared with of IOS No.45/1984.

Sieve size ,mm	Cumulative passing,%	IOS No.45/1984
10.00	100	100
4.75	97	90-100
2.36	92.2	75-100
1.18	77	55-90
0.60	52.2	35-59
0.30	10.6	8-30
0.15	2	0-10

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Table 4: Chemical composition of the used **Volcanic Ash***.

Oxide composition	Oxide content %
SiO ₂	65.85
Al ₂ O ₃	11.43
Fe ₂ O ₃	1.37
Na ₂ O	2.07
K ₂ O	5.23
CaO	3.87
MgO	0.78
SO ₃	0.21
BaO	0.09
C	0.61
H	0.38
N	0.15
Cl	0.22
O	0.73
Mn ₂ O ₂	0.02
L.O.I.	6.43

*values supplied by the manufacturer.

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Table 5: Characterizations of **Glenium 51***.

Characteristics	
Density	1.05 - 1.16 kg/liter
PH. Value	6.4
Chlorine Content% (EN 480-10)	<0.1
Colour	dark brown
Alkaline Content % (EN 480-12)	<3
Viscosity, cps	at 20°C 138± 30

* Given by the manufacturer.

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Table 6: Mortar mix proportion, percentage of flow

MIX ID	VA	W/C	Cement Kg/m³	Sand Kg/m³	SP%	Flow%
VA-0	0	0.35	400	1100	2	112
VA-5	5	0.35	380	1100	2	112
VA-15	15	0.35	340	1100	2.3	112
VA-25	25	0.35	300	1100	3	112

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