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Performance of Geopolymer Mortars Subjected to Elevated Temperatures.

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PERFORMANCE OF GEOPOLYMER MORTARS SUBJECTED TO ELEVATED TEMPERATURES أداء المون الجيوبوليمرية المعرضة لدرجات الحرارة المرتفعة

Ameer Albadawy, *Metwally Abd Elaty*, and Mohamed Taman

KEYWORDS: *Geopolymer based materials; heat temperature; compressive strength and residual strength.*

الملخص العربي-:يعرض هذا البحث نتائج البرنامج المعملي لدراسة أداء المون الجيوبوليمرية المعرضة لتاثير درجات الحرارة المرتفعة .هذا والمتغيرات األساسية التي تم دراستها تشمل تأثير الموالرية لهيدروكسيد الصوديوم) 6 ، ،10 16 ، 20 مول(، نسبة سيليكات الصوديوم إلي هيدروكسيد الصوديوم)،1،2.5 4.5(، نسبة المحلول القلوي إلي الرماد المتطاير)،0.4 ،0.5 0.6(، درجة حرارة المعالجة) ،25 ،60 90 ° م (، مدة الراحة)صفر، ،24 48 ساعة(، نسبة الماء الزائد)،0.1 ،0.125 0.15(كنسبة من وزن الرماد المتطاير. عالوة علي المون الجيوبوليمرية التى تم دراستها فقد تم تجهيز خلطة مونة من األسمنت البورتالندي ودراسة أدائها تحت تأثير درجات الحرارة المرتفعة للمقارنة. هذا ولقد تم تعريض جميع العينات لدرجات حرارة بلغت ،200 ،400 ،600 800،1000 ° م وذلك لمدة ساعتين .هذا ولقد شملت الدراسة تعيين مقاومة الضغط عالوة علي النقص في الوزن تحت تأثير درجات الحرارة المرتفعة . ولقد بينت النتائج أن نسبة مقاومة الضغط المتبقية لعينات المونة الجيوبوليمرية قد تراوحت بين 60.1 % ، 90.9 % من مقاومتها قبل الحرق وذلك بعد التعرض لدرجة حرارة 1000 ° م. في حين بلغت نسبة مقاومة الضغط المتبقية صفر% لعينات األسمنت البورتالندي بعد تعرضها لدرجة حرارة 800 ° م . عالوة على ذلك أتضح أن هناك عالقة عكسية بين مقاومة الضغط وكمية الماء االضافي المستخدمة. كما تالحظ أيضا أن هناك عالقة عكسية بين نسبة المحلول القلوي إلي الرماد المتطايرومقاومة الضغط. كما أوضحت نتائج عينات المون الجيوبوليمرية انخفاضا نسبيا في نسبة النقص في الوزن مقارنة بالعينة االسمنتية وهو مايتفق مع نتائج مقاومة الضغط المتبقية

*Abstract***— This paper presents the results of an experimental program that was carried out to investigate the performance of geopolymer (GP) mortar mixes subjected to elevated temperature. The main investigated parameters were the molarity levels of NaOH(6M, 10M, 16M and 20M), the sodium silicate to sodium hydroxide solution ratios (1, 2.5 and 4.5), the alkaline liquid to fly ash ratio (0.40, 0.5 and 0.6), the curing temperatures (25, 60 and 90 oC), the rest period (RP) (0, 24 and 48 hours) and extra water (0.1, 0.125 and 0.15). Parallel to preparing the different GP mortars, ordinary Portland cement mortar was made to compare results. The hardened GP and OPC mortars were subjected to different elevated temperatures of 200, 400, 600, 800, 1000 oC for two hours. The properties of GP**

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mortars including the compressive strength and the loss of weight were studied. The residual compressive strengths of the GP samples ranged between 60.1% and 90.9% after exposure to 1000 oC of the compressive strength before exposure to elevated heat temperature, whereas, it reached zero% for OPC samples after exposure to 800 oC. On the other hand, an inverse relationship between the compressive strength with the used extra water was recorded. Moreover, another inverse relationship between alkaline solution to fly ash ratio and the compressive strength was noticed. The GP mortars showed also relatively lower mass loss values compared with OPC specimens which agree well with the residual strength results. later. Otherwise, use this document as an instruction set. The electronic file of your paper will be formatted further at IEEE. Paper titles should be written in uppercase and lowercase letters, not all uppercase. Avoid writing long formulas with subscripts in the title; short formulas that identify the elements are fine (e.g., "Nd–Fe–B"). Do not write "(Invited)" in the title. Full names of authors are preferred in the author field, but are not required. Put a space between authors' initials. Define all symbols used in the abstract. Do not cite references in the abstract. Do not delete the blank line

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I. INTRODUCTION

ONCRETE is one of the most widely used construction materials in the world and Ordinary Portland cement (OPC) is its main binder. However, the cement industry contributes between 5 and 7% of the total global CO2 emission into atmosphere. Research efforts are continuing to make the concrete more sustainable by reducing the amount of the used cement. Partial replacement of cement by various cementitious materials such as, fly ash, slag, silica fume,...etc. in concrete is now common practice in industry as their use in the concrete improves the mechanical and durability properties. The development of a new type of inorganic cementitious binder called (geopolymeric binder) was first introduced by Davidovits (1978). GP binder is a new material that does not use Portland cement. Instead, a source of material such as fly ash, that is rich in silicon (Si) and aluminium (Al), is reacted by alkaline solutions (Duxton et al., 2007). It has been estimated that the manufacture of geopolymeric cement emits about 80% less CO2 than the manufacture of OPC (Davidovits 1994 and Van D. JSJ et al. 2010). Fly ash based geopolymers have extremely low embodied energies.Van D.JSJ et al. (2010) found that producing GP-based concrete (produced from fly ash and asoluble silica-like activator, and cured under mild heating), consumed 70% less energy when compared with OPC-based concrete of similar strength C

Considerable research has been conducted on the parameters affecting producing fly ash based geopolymer (GP) concrete and its influence on the fresh and hardened properties. Hardjito et al (2004) found that higher concentration (in terms of molarity) of sodium hydroxide solution and the higher the mass ratio of sodium silicate-tosodium hydroxide liquid results in a higher compressive strength of GP concrete. The factor of curing temperature was examined by Hardjito and Rangan (2005). The results showed that the higher curing temperature in addition to longer curing times resulted in larger compressive strength for GP concrete. The added extra water remains outside of the geopolymeric network, acting as a lubricating element improves the workability of the mixtures (Hardjito and Rangan, 2005, and Davidovitts, 2011). There must be sufficient time (rest period) available between casting of products and sending them to the curing process (Hardjito and Rangan 2005, Prakash R.V., and Urmil V. D., 2013).Many of the durability problems associated with OPC concrete arise from its calcium content in the main phases. Geopolymeric materials possess low calcium containing materials that may prevent geopolymers from experiencing such negative effects. The test results for GP concrete indicated excellent resistance of the chloride attack, with a longer time to corrosion cracking relative to OPC concrete (Reddy D.V et al 2013). Low calcium fly ash based GP concrete exhibits high resistance to sulphate attack. Specimens exposed to sodium sulphate for up to 6 months

showed no visual signs of surface spalling, deterioration and cracking. The best performance in different sulphate solutions was observed in the GP material prepared with sodium hydroxide and cured at elevated temperature (Wallah and Rangan, 2006). The previous studies Kong D, Sanjayan J.G (2008) of fly ash based GP concrete have been conducted to investigate their residual mechanical properties after exposure to elevated temperatures. A number of publications studied the advantages of geopolymers in high temperature applications and showed evidences of their ability to have high residual compressive strengths after exposure to 800ºC. Behaviour of geopolymer at high temperatures while hot is important to assess the fire endurance of structures made with this material. While OPC concrete degenerates and degrades at high temperature from last different study fly ash GP concrete can maintain its desired compressive strength at 400 °C. Kong D., Sanjayan J.G. (2010). Considering the echo friendly geopolymer based materials and its application fields at which it may be subjected to high rise temperature during its life time. So this research in devoted to study the performance of the Geopolymer mortars under high rise temperature

II. EXPERIMENTAL PROGRAM

A. Materials

1) Fly Ash

In the present study, low-calcium fly ash (FA) class F according to ASTM C618 (2008) produced in a coal-fired power plant was used as a source of a pozzolanic material with specific gravity of 2.31. The chemical composition of the used fly ash as determined by X-Ray Fluorescence (XRF) analysis is shown in Table (1)

2) Alkaline solution

The used alkaline solution was a combination of sodium hydroxide NaOH (NH) and sodium silicate. Sodium hydroxide was in pellets form with 98% purity. NH has molecular weight of 40. In present research four different molarities were used 6 M, 10M, 16M and 20M. Sodium silicate solution Na2Sio3 (NS) (SiO2=29.4%, $Na2O = 14.7%$ and water=55.9%) obtained from chemical industries was used as the alkaline activators. Both of the liquid solutions were then mixed at laboratory together at the required ratio to prepare the alkaline solution

3) Fine aggregate

The fine aggregate used in this study was river siliceous sand complied to ECC-Appendix 3 (2007) with specific gravity of 2.55, a fineness modulus of 2.36 and water absorption of 0.90%. The physical and mechanical properties of the used sand are shown in Table (2). Moreover, the grain size distribution curve of the used sand is presented in Fig (1)

 $T_{ADID}E(1)$

TABLE (2) PHYSICAL AND CHEMICAL PROPERTIES OF THE USED SAND

Property	value		
Specific gravity	2.55		
Unit weight, (t/m^3)	1.72		
Void ratio, (%)	32.55		
Fineness modulus	2.36		
clay and fine matter % (by weight)	2.		
Water absorption, (%)	0.90		

4) Ordinary Portland cement

Ordinary Portland Cement (OPC) CEM1 42.5N with initial and final setting times 75 and 220 mins, respectively was used. The used cement complied with the requirements of (EN 196-1:2005). OPC was used for the preparation of the control mixture. Tables (3) and (4) show the properties of the used cement.

Fig.1: Grading of the used sand

5) Water

Potable water according to the requirements of the [ECP 203/2007], with PH value of 7.5 was used as extra water (EW) for GP mixtures to enhance the flow ability of the GP mortar in fresh state. On the other hand, it was used for mixing and curing the OPC mortar specimens

6) Mix proportions, mixing procedure and casting

The compositions of GP mortar mixes containing fly ash, fine aggregate, NS, NH solution and extra water are shown in Table (5). The conducted mixtures consisted of six groups. The first group investigated the effect of concentration of NH, and the second group studied the effect of sodium hydroxide to sodium silicate ratio. Whereas, the third group investigated the effect of alkaline liquid to fly ash ratio by mass and the fourth group explored the effect of extra water to GP solids ratio by mass. On the other hand, the fifth group investigated the heat-curing temperature (°C) and the sixth group studied the effect of the heat curing time.

The fine aggregate and the fly ash were firstly mixed together in dry state. The alkaline solution was mixed with the extra water. The liquid component of the mixture was then added to the dry materials and the mixing continued for another 8 minutes. After mixing, the flow test was conducted to evaluate its percentage flow in fresh state according to ASTM C1437 (2015).

After conducting the flow test, the fresh mortar was cast in the moulds and compacted by manual strokes. The GP specimens were left open to air temperature for 24 hours then were heat cured in an oven at different

temperatures. After that the specimens were removed from the moulds and taken out from the oven. The specimens were left open to room temperature up to testing. The mixing process used for OPC mortar was similar to that for GP mortar. The control mortar mix (OPC) was removed from its moulds and was cured using tap water for 28 days

B. 1) Mechanical Properties of GP mortar

The compressive strength test was carried out at 3, 28 and 56 days age, the test setup is presented in Fig.2. To study the compressive strength and the flexural strength of cement and GP mortar samples, prismatic specimens of 40×40×160 mm were prepared using the proportions mentioned in Tables (5) and (6) according to EN 196- 1:2005. The flexural strength test was carried out at 3, 28 and 56 days age. Each sample was tested using threepoints load bending setup to obtain flexural strength. The test set up for flexure is shown in Fig. 3

Fig. 2: Compression test setup

Fig. 3: Flexural test setup

2. Elevated temperature heating methods.

Mortar specimens with dimensions of $40 \times 40 \times 160$ mm were prepared and tested to investigate the effect of elevated temperature on the performance of geopolymer mortar mixtures according to Gorhan G. and Kurklu G., (2014). An electrical heater furnace designed for a maximum temperature of 1200 °C was used as shown in Fig.4. Specimens were exposed to different temperatures of 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C at an incremental rate of 4°C per minute from room temperature. The temperature was sustained for 2 hrs after reaching the desired elevated temperature. The exposed specimens were allowed to gradually cool to room temperature inside the furnace

Fig. 4:Automatic electric furnace for elevated temperatures exposure

III. .RESULTS AND DISCUSSIONS

A.Effect of elevated temperature on properties of GP mortars mixes.

The following paragraphs present the performance of GP mortar mixes subjected to elevated temperature. Different parameters are checked. Mechanical properties as well as physical ones are included. The results of the compressive strength as affected by high rise temperature on GP mortar mixes as well as Portland cement mortars are shown in Fig. 5. It can be clearly seen from Fig. 5 that after exposure to elevated temperature of 200oC both of the GP specimens and OPC specimens showed a remarkable increase in the compressive strength. Where GP specimens recorded an increase in compressive strength about 122.3% compared to that of the control specimen (unexposed or room temperature specimens). But in case of OPC specimens, only 47.4% increase in the compressive strength value compared to the unexposed specimens was recorded. This attributes to the positive effect of the elevated temperature on the geopolymerization and hydration processes.

At elevated temperature of 400oC, the compressive strength of the OPC specimens started to decrease and recorded 12.1 % reduction in compressive strength compared to unexposed specimens. This reduction is due to the release of the chemically bound water Guerrieri, M. and Sanjayan, J.G. (2010) and this bond of hydrated water is fully broken at 800oC (zero compressive strength recorded). The exposure to elevated temperature of 400℃ and 600oC still showed a positive effect on the compressive strength, resulting in increasing values of about 48.4% and 34.4% compared to the control specimens respectively. The good firing resistance of GP specimens was assured after exposing to 800ºC and1000 ºC. Specimens yielded a reduction about 1.2 % and 9.1% compared to unexposed specimens at 800ºC and 1000ºC, respectively. The relation between the elevated temperature

and the mass loss for the GP and OPC specimens is shown in Fig. 6. It can be concluded that the least percentage of mass loss is observed for GP specimens

1) Effect of concentration of sodium hydroxide solution

The measured compressive strengths of GP mortars containing different molaries of 6M, 10M, 16M and 20M after exposing to 200 oC, 400 oC, 600 oC, 800 oC and 1000oC temperatures are shown in Fig. 7. It can be seen that the residual compressive strengths of GP mortars with concentrations of 6M, 10M, 16 M and 20M are increased by about 23.08, 51.01, 122.27 and 52.92 % at 200oC compared to the unexposed specimens. This may be due to the activation as a result of high rise temperature. The exposure to elevated temperature of 600 oC leads to an improvement at the aluminia silicate networks in geopolymerization Rickard et al. (2012). The maximum compressive strength achieved by 16M specimens followed by 10M, 20M then by 6M and this sequence was repeated under the investigated elevated temperatures.

At 1000oC temperature, the specimens of mixture 6M showed the minimum residual compressive strength followed by the specimens of mixture 20M then 16M and 10M. From Fig. 8, it is noticed that the least percentage of mass loss is observed in the specimen of mixture 16M followed by the specimens of mixture 10M then 6M and 20 M

2) Effect *of sodium silicate to sodium hydroxide solution ratios on properties of GP.mortars*

Effect of alkaline liquid to fly ash ratio on properties of GP mortars the results illustrated in Fig. 9 proved that, the use of NS helps to improve the geopolymerisation process by accelerating the dissolution of source material, it was observed by Hardjito and Rangan. Conversely, when the NS/NH ratio was more than 2.5 the compressive strength tended to decrease; instead, the dissolved precursors tend to form zeolites which are weaker in compressive strength.

The residual compressive strengths of GP mortar in case of NS/NH ratios of 2.5 and 4.5 are increased by about 122.27 and 78.11 % at 200oC. The findings in Fig. 10 indicated that the least percentage of mass loss is observed in case of NS/NH ratio of 2.5 followed by the case of NS/NH ratio of 4.5.

3) Effect of alkaline liquid to fly ash ratio on properties of GP mortars

The obtained results in Fig. 11 clarified for each specified temperature, that there was an inverse relationship between the effect of alkaline liquid to fly ash ratio and the compressive strength of GP mortar. The residual compressive strengths of GP mortar in case of alkaline liquid $/$ fly ash ratio of 0.4, 0.5 and 0.6 are increased by about 122.27%, 80.63% and 68.66 % at 200oC, compared to those at ambient temperature, respectively. However, with further heating at 400, 600, 800 and 1000oC temperatures, the least percentage of mass loss is observed in case of alkaline liquid / fly ash ratio of 0.4 followed by the case of alkaline liquid / fly ash ratio of 0.5 and 0.6 as shown in Fig.12

4) Effect of curing regime on properties of GP mortars

The reason for the need of the heat curing is that the activation of fly ash which represents an endothermic reaction and it is very important for the geopolymerization process of the fly ash based GP specimens. Fig. 13 demonstrates that the GP specimens with curing temperature of 25oC at room temperature exhibited higher compressive strength at 200oC than other specimens cured at temperatures of (60oC and 90oC). From 400oC to 1000oC the residual compressive strengths of specimen with curing temperature of 60oC is higher than other specimens at 25oC and 90oC. The obtained results in Fig. 14 clarified that the least percentage of mass loss is observed in case with curing temperature of 60oC followed by the case with 25oC and 90oC

Experimental program and mix proportions of GP mixtures										
Groups	Mix ID	Molarity	NS/NH	S/FA	FA/Sand	RP (hours)	CP (hours)	EW/FA	CT ($°C$)	
G1 Molarity	6M	6M	2.5	0.40	0.50	24	48	0.10	60	
	10M	10M	2.5	0.40	0.50	24	48	0.10	60	
	20M	20M	2.5	0.40	0.50	24	48	0.10	60	
G2 Na2SiO3/NaOH (NS/NH)	NS/NH 1	16M		0.40	0.50	24	48	0.10	60	
	GP control	16M	2.5	0.40	0.50	24	48	0.10	60	
	NS/NH 4.5	16M	4.5	0.40	0.50	24	48	0.10	60	
G3 Activators/ Fly ash (S/FA)	S/F 0.5	16M	2.5	0.50	0.50	24	48	0.10	60	
	S/F 0.6	16M	2.5	0.60	0.50	24	48	0.10	60	
G ₄ Curing Temperature (CT)	$C 25^{\circ}C$	16M	2.5	0.40	0.50	\sim	\sim	0.10	Air cured	
	$C 90^{\circ}C$	16M	2.5	0.40	0.50	24	48	0.10	90	
G5 Rest period (RP)	RP ₀	16M	2.5	0.40	0.50	$\mathbf{0}$	48	0.10	60	
	RP ₂	16M	2.5	0.40	0.50	48	48	0.10	60	
G6 Extra water (EW)	EW 12.5%	16M	2.5	0.40	0.50	24	48	0.125	60	

Table (5)

TABLE(6) Mix proportions for Ordinary Portland cement mixture **Mix ID Cement Fine aggregate Water Curing Temperature** (${}^{\circ}$ C) **M-Cement 1 1 2 1 0.5 1 2 2 2 2 2 2 2 2 1**

5. Effect of rest periods on properties of GP mortars

The effect of rest periods on the compressive strength of GP mortars at elevated temperatures are also evaluated in this study and are shown in Fig. 15. The results showed that the residual compressive strengths of GP mortar in case of rest periods of 0, 1 and 2 days are increased by about 55.47%, 73.9% and 122.27 % at 200oC compared to ambient temperature strength. The no rest period showed the smallest compressive strength value for all steps of the elevated temperature. Fig. 16 demonstrates that the least percentage of weight loss is observed in case of the effect of rest period of 1 day followed by the case with 2 days and no rest period. Moreover, and at temperature levels beyond 200oC, the compressive strength values reduced compared to those at 200oC. The mixes subjected to 1 day as a rest period showed the best performance at higher temperature levels

6. Effect of extra water on properties of GP mortars

The extra water/fly ash ratio affects the volume of pores and the porosity in the matrix which directly influences the strength of the GP mortars. It facilitates the workability of the mixtures and it does not become a part of the resulting GP structure (Kong et al. 2008). Fig. 17 revealed that there is an inverse relationship between the extra water and the compressive strength at elevated temperature. The residual compressive strengths of GP mortar in case of extra water 0.1, 0.125 and 0.15 are increased by about 122.27, 104.12 and 94.37% at 200oC compared to unexposed (room temperature), respectively. At elevated temperature of 1000oC, the GP specimens still had a sufficient residual compressive strength for all extra water usages. The results in Fig. 18 show that the least percentage of mass loss is observed with extra water of 0.1 followed by the case with 0.125 and 0.15.

Generally, using extra water enhances the workability of GP mortar mixes and reduces its compressive strength. Moreover, an inverse relationship is found between the compressive strength and the used extra water at all temperature levels

IV. DAMAGE AND COLOUR CHANGES OF GP MORTARS AT DIFFERENT TEMPERATURE LEVELS

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Fig. 19 shows the colour changes occurred in the GP mortars after exposure to elevated temperatures from a normal dark grey to salmon pink at 1000 oC. It can be seen that up to 600oC no cracks were formed in the GP specimens' surface. However, GP showed signs of cracks at 800oC and it became worst at 1000oC as shown in Fig.20. On the other hand, in the OPC mortar, colour changes occurred after exposure to elevated temperatures from a normal dark grey to light grey at 1000 oC. It can be seen that up to 400 oC no cracks were formed in the OPC specimen's surface. However, OPC specimens showed worst signs of cracks at 600 oC and it were damaged at 800 oC and at 1000 oC

Fig. 5: Compressive strength for GP and OPC mortars exposed to elevated temperatures

Fig. 6: Loss of mass for GP and OPC mortars exposed to elevated temperatures

Fig. 7: Effect of concentration of sodium hydroxide solution on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig. 8: Effect of concentration of sodium hydroxide solution on mass loss of GP mortar mixes exposed to elevated temperatures

Fig. 9: Effect of NS/NH ratio on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig. 10: Effect of NS/NH ratio on mass loss of GP mortar mixes exposed to elevated temperatures

Fig. 11: Effect of solution /FA ratio on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig. 12: Effect of solution /FA ratio on mass loss of GP mortar mixes exposed to elevated temperatures

Fig. 13: Effect of curing temperature on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig. 14: Effect of curing temperature on mass loss of GP mortar mixes exposed to elevated temperaturees

Fig. 17: Effect of extra water on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig. 15: Effect of the rest periods on the compressive strength of GP mortar mixes exposed to elevated temperatures

Fig.18: Effect of extra water on mass loss of GP mortar mixes exposed to elevated temperatures

Fig. 16: Effect of the rest periods on mass loss of GP mortar mixes exposed to elevated temperatures

Fig.19: The effects of elevated temperatures on colour changes of GP mortar specimens

Fig. 20: Damage pattern of GP and OPC mortar mixes as affected by higher temperatures

V.CONCLUSIONS AND RECOMMENDATIONS

The Based on the results of the experimental work and the analysis presented in this paper, the following conclusions can be drawn:

- **1.** GP mortars showed a good result from compressive strength view point at different elevated temperatures compared to that of cement mortar. This attributes to the positive effect of that elevated temperature on the geopolymerisation processes.
- **2.** The molarity of NH affects significantly the compressive strength of the exposed specimens to elevated temperature. Moreover, 16 M showed the best performance.
- **3.** Using higher NS/NH ratio of 2.5 yielded the maximum residual compressive strength of GP mortar after exposure to 1000 oC.
- **4.** The alkaline liquid to fly ash ratio inversely proportional to the compressive strength. The ratio of 0.40 showed the maximum residual compressive strength for the specimens exposed to 1000 oC.
- **5.** The GP specimens which were cured at ambient laboratory temperature exhibited higher compressive strength at 200 oC compared to that cured at other curing temperatures.
- **6.** The effect of rest period had an important role for geopolymerization processes. Rest period of one day enhances the residual compressive strength and the percentage of mass loss showed the least values
- **7.** There is an inverse relationship between effect of the extra water and the elevated temperature resistance. The residual

compressive strengths of GP mortar were decreased with increasing the extra water percent.

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