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Durability Performance of Metakaolin-based Geopolymer Mortars Containing Cement

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ملخص البحث :

في هذا البحث ، تم تصنيع جيوبوليمر قائم على الميتاكولين (MK-GPM) بنسب مختلفة من 0، 20 ، 40 و 60٪) من الاسمنت باستخدام سيليكات الصوديوم / هيدروكسيد الصوديوم ومعالجتها في ظروف المعالجة المحيطة (23 ± 4) وكذلك المعالجة الحرارية عند 60 درجة مئوية. تم قياس المتانة من حيث الامتصاصية وامتصاص الماء والهجوم الكيميائي العدواني (10٪ كبريتات الماغنسيوم و حمض الكبريتيك PH = 3 و 10٪ كلوريد الصوديوم) من حيث فقد الكتلة وتدهور قوة الانضغاط في 10 أسابيع من التعرض. تمت مقارنة النتائج مع تضمين مصفوفة OPC التقليدية ، على الرغم من تفوق مونة الاسمنت (الامتصاصية وامتصاص الماء) في آليات الشعيرات الدموية التي تقيد دخول الماء إلى المونة ، عند تطبيقه على الهجمات الكيميائية ، أظهر الجيوبوليمر أداء متانة فائقة. أظهرت هذه النتيجة ثبات مصفوفة المونة لـ MK-GPM للهجوم الكيميائي.

ABSTRACT:

In this paper metakaolin based geopolymer mortars (MK-GPM) with different percentages of OPC (0, 20, 40 and 60%) were made by using NaOH/sodium silicate and cured at ambient curing condition (23 ± 4 C^O) as well as at 60 °C heat curing. Durability has been measured in terms such as sorptivity, water absorption and aggressive chemical attack (10% $MgSO_4$, H_2SO_4 PH = 3 and 10% NaCL) from where mass loss and degradation of compressive strength at 10 weeks of exposure solutions. The results were compared with conventional OPC matrix inclusion, despite the superiority of OPC mortar (sorptivity and water absorption) in capillarity mechanisms that restrict water admission to mortar, when applied to chemical attacks, geopolymer demonstrated superior durability performance. This result showed the steadiness of the mortar matrix of MK-GPM for chemical attack.

KEYWORDS: Metakaolin (MK), Microstructure properties, Durability Properties, Aggressive environment, MK based geopolymer matrix (MK-GPM)

INTRODUCTION

Geopolymers are an inorganic bundle, produced with the chemical activation of the raw material aluminosilicate with highly alkaline solution at high or room temperatures for a structure that is mostly formless and has a comparable purpose as the OPC [1,2]. The key consideration for designing concrete structures is durability. Environmentally friendly concrete protects the natural resource, waste reduction and the reduction of the environmental impact of repair and maintenance [3].

For example, the geopolymers product metakaolin (MK) have better resistance to sulfuric acid than OPC [4]. Parallel assumptions were complete by Stroeven et al. [5] which demonstrated outstanding durability properties for geopolymers (MK). While previous the stated advantages of geopolymer, curing methods always prevent its use in filed applications. The common curing method need a heat-curing time of 24–48 h at 60–100 C^O which in actual field applications becomes very difficult. Therefore, this research aimed to perform the curing process in two ways heat and ambient curing and investigate the effect of different curing on the different durability properties. Researchers are extra focused to create geopolymers with different additives like GGBS, MK, OPC, etc. [6-9]. The goal of this study is development durability of geopolymer mortar and to track the impact of calcium content added (in OPC form) under both curing conditions. Properties are studied like change in compressive strength, porosity and aggressive environments attack such as sulphate (10% $MgSO_4$), acid (H_2SO_4) pH = 3 and chloride attack (10% NaCL) for 10 weeks after 28 days curing for both conditions.

2. Experimental Program

Materials

Local Ordinary Portland Cement CEM I 42.5N complying with ESS 373/91 [10] was used in the study. The metakaolin (MK) used was purchased from local market. The physical properties and chemical composition of OPC and MK are illustrated in Tables 1 and 2 respectively. X-ray diffraction (XRD) for OPC and MK are revealed in Fig.1. Characteristic peaks of phases alite (A), belite (B), and ferrite (F) are found in cement. In comparison, MK's XRD patterns indicate that MK's crystalline phases composed of kaolinite (K), mica (M), and quartz (Q).

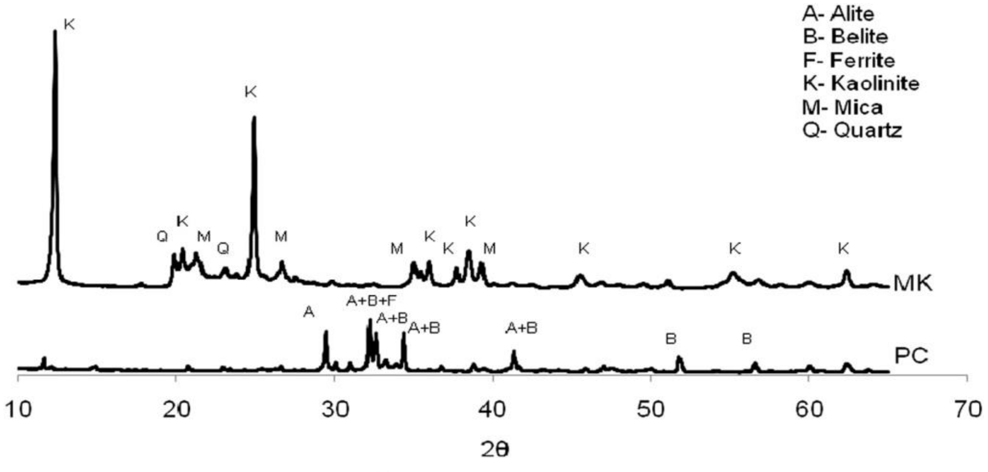

Fig.1 The X-ray diffraction patterns of OPC and MK

The fine aggregate was clean siliceous sand with fineness modulus of 1.74, 1.5% water absorption and 2.5 specific gravity. The used alkali activator consists of two solutions; (NaOH) and (Na_2SiO_3). The alkali solutions were mixed with (Na_2SiO_3/NaOH) ratio of 2.5.

Table1 Physical properties of OPC and MK

Property	OPC	MK
Specific gravity	3.15	2.5
Blaine fineness (m^2/kg)	350	12000
Average particle size (µm)	12	1
Color	Gray	White

Table 2 Chemical composition of OPC and MK

Compound (%)	**OPC**	MK
SiO_2	20.7	53
Al_2O_3	5.4	44
Fe_2O_3	3.8	0.5
MgO	2.1	0.2
CaO	64.3	0.2
Na_2O	0.2	0.3
K_2O	0.4	0.2
SO_3	2.1	0.2
Loss on ignition	0.9	1.1

Mix proportions

Five geopolymer mortar mixtures were prepared by replacing MK with OPC. The replacement ratio varied from 0 to 60% by weight with increment 20%. OPC mortar with the same mix ingredients was also prepared as shown in Table 3. All mixes have the same binder content (730 Kg/m^3), water/binder ratio of 0.35, activators content (292 Kg/m^3) and sand content (1178 Kg/m^3) and no extra water or superplasticizer was added.

Table 3 Details of MK and OPC Based proportions of geopolymer mortar mix

Mix no.	**Code**	**Quantity of mortar mixture (kg/m^3)**					**W/S**
		Sand	**MK**	**OPC**	**SS**	**SH**	
1	100 MK	1178	730	0	208.6	83.4	0.35
2	20 OPC	1178	584	146	208.6	83.4	0.35
3	40 OPC	1178	438	292	208.6	83.4	0.35
4	60 OPC	1178	292	438	208.6	83.4	0.35
5	OPC	1178	0	730	-	-	0.35

Mixing and Casting

Because the mixing of alkaline solutions releases significant heat [11,12], alkaline solutions combined to reduce their temperature at air temperatures 24 hours before use. Geopolymer mixes were mixed manually. First, dry powders and fine aggregates were blended together, thereafter alkaline solutions were added to the binder and remixed together till complete homogeneity achieved. After then the fresh mortars were casted into the molds and compacted using tamping rod. The specimens are then covered with sheets of polyethylene and kept in molds at room temperature of 23 ± 4 C^O for about 24 hours then samples were demolished and cured

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Curing conditions

Half the specimens for each mix were cured at normal ambient temperature 23 ± 4 C^O to 28 days and then put it into solutions (H_2SO4,$MgSO_4$and NaCL) till time of testing(2,4,6,8and10 week) and the other half were cured at 65 C^O for 24 hours inside oven and then left at normal ambient temperature the remaining period (28 days) and then put it into solutions (H_2SO4 ,$MgSO_4$ and NaCL) till testing age(2,4,6,8and10 weeks).

Testing Properties and Procedures

Permeation Properties

The sorptivity rate have been calculated according to ASTM C1585 [13] at 56 days. In this experiment, specimens were positioned upright with a properly sealed curved surface on the sides, so that water could only penetrate through the bottom. The constant head of 5 mm over the bottom of the specimen, and sufficient distance of approximately 100 mm among the specimens, were kept on the plate for its simple absorption by capillary action. Water absorption experiments were performed at 56 days on 50 mm^3 cube specimens. A procedure under ASTM C642 [14].

2.5.2 Durability Properties

Test series have been proposed for the durability assessment of MK-GPM and OPC mortars. Samples were exposed to 10% solution of magnesium sulfate,10% solution sodium chloride and sulfuric acid (PH=3) after 28 days from casting and kept immersed up to 10 weeks. Different tests, such as the change in weight, were used to monitor the effects of solutions on the specimens regularly over the exposure period which cleanly wiped their specimens before weight measurement. The specimens were weighed over the exposure time and the weight change percentage as a percentage of original weight was calculated. Cubics 50 mm were used according ASTM C157-08[15] each 2 weeks till 10 weeks of exposure and compressive strength loss accordance with ASTM C 109 [16]as well.

Results and discussion

Permeation Properties

Sorptivity

Fig.2 illustrates the results of the sorptivity test for different mixes for both ambient and heat curing conditions at 56 days. It is clear that MK-GPM containing 100% MK indicates a major increment in the sorptivity at both curing conditions and it decreases as the metakaolin content decreases. This is because MK reacts very quickly with calcium hydroxide produced during hydration and is converted into a variety of soluble, stable cement products so the permeability of GPM is expected to increase [17,18]. In addition, this could be referred to there is a more free water available at this particular W/B to particles lubricate into fresh concrete and then the ITZ could have become thicker [19].

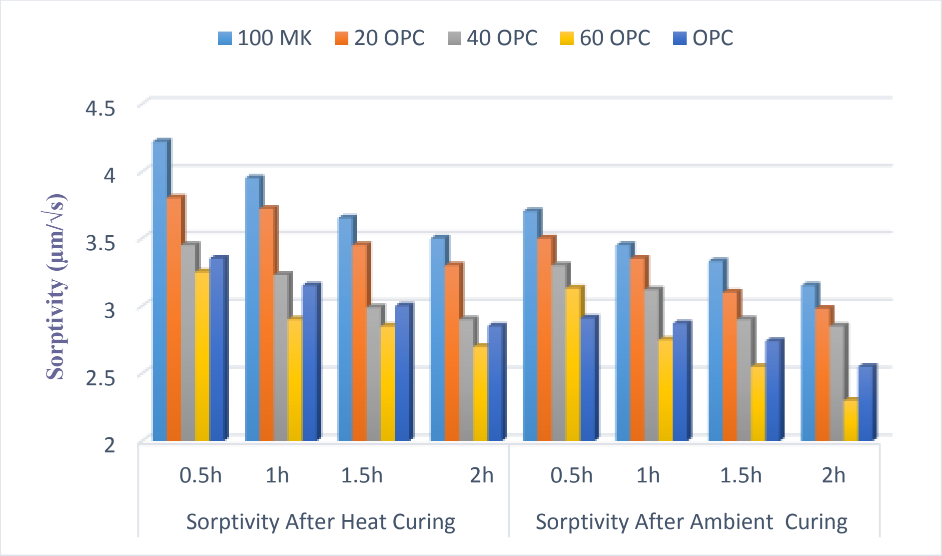

Fig.2 Sorptivity of geopolymer mortars after 56 days

Water Absorption

From Fig. 3, it's obvious that the OPC samples under both curing conditions, has a abundant lesser absorption rate than the consistent MK-GPM. This is partly due to the variance in compressive strength and also to the capillary process which detect that the absorption rate of OPC is lower than that of MK-GPM [20].

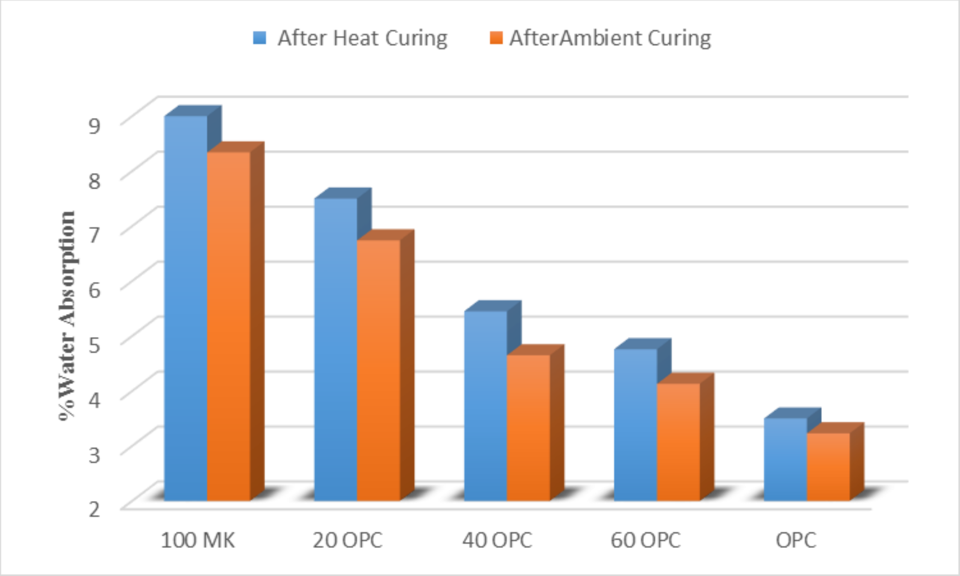

Fig.3 Water absorption of geopolymer mortars after 56 days

Also, because MK reacts very fast with calcium hydroxide produced during hydration and is rehabilitated into a variety of soluble, stable cement products so the permeability of

GPM is expected to rise [17,18]. Fig. 4 emphasizes the direct proportion between water absorption and Sorptivity, this relation gives credibility to teste result.

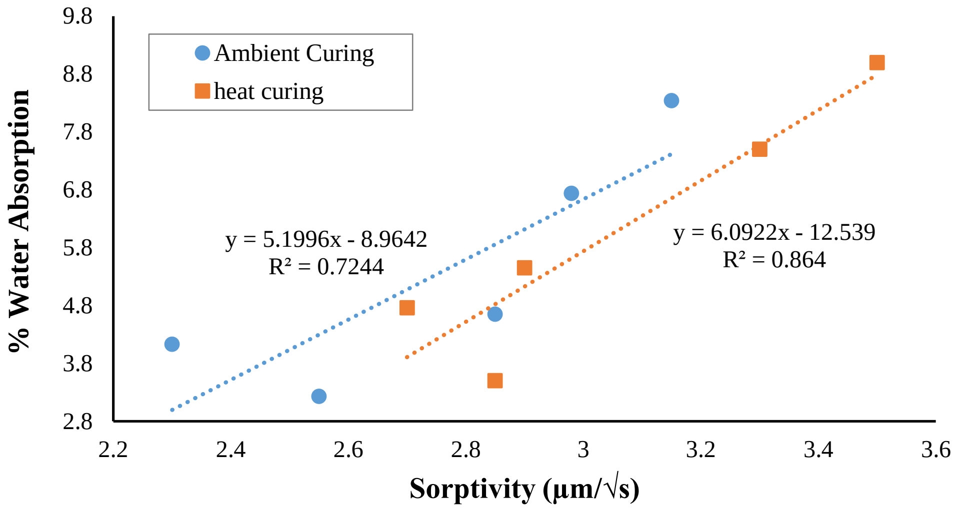

Fig.4 Relation between water absorption and sorptivity

Durability Properties

Mass loss

Fig. 5-7 illustrated the results of mortar weight change exposed to sodium chloride, magnesium sulphate and sulfuric acid solutions, for 10 weeks after 28 days of casting. From these figures, it can be seen that the weight of specimens tends to increase in all chemical solutions. The weight increase may be referred to the implication into the mass of chemical particles that penetrated the mortar and led to an increase in samples weight which benefits the volumes of mortar [21].

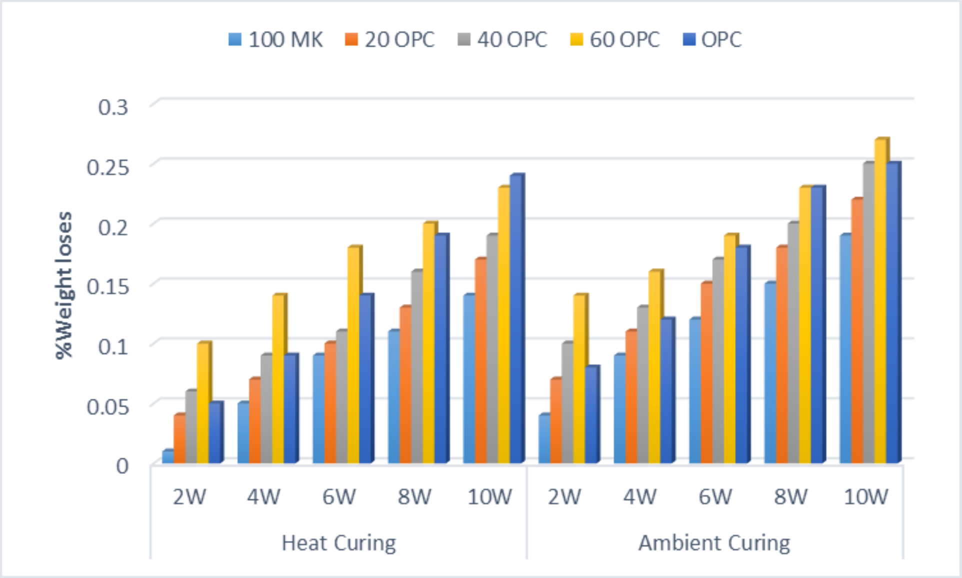

Fig.5 Mass loss of different mortars exposed to sodium chloride.

This phenomenon is called reaction-diffusion among the solutions and the mortar binder. The reaction leads to the calcium sulphate (gypsum), which leads to increased volume of samples [22,23]. A more distractive reaction is subsequently within the cement matrix between gypsum

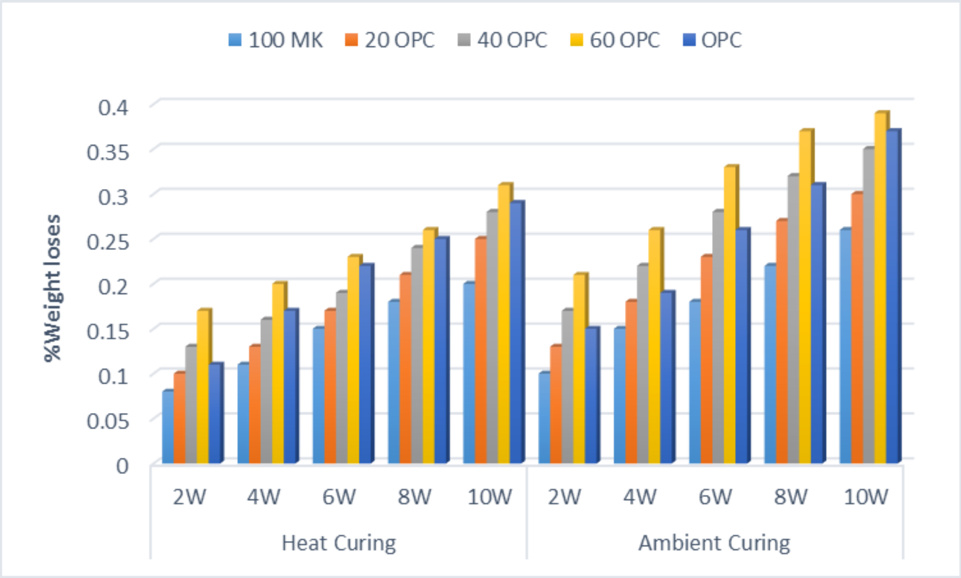

Fig.6 Mass loss of different mortars exposed to magnesium sulphate.

and tricalcium aluminates, which resulting in a higher volume of calcium sulphoaluminate (etringite)product [24]. The most pronounced weight changes due to exposure was occur in sulfuric acid followed by magnesium sulphate followed by sodium chloride. Also, it is evident that MK-GPM at both curing conditions have completed better than OPC mortar in an aggressive environment [25].

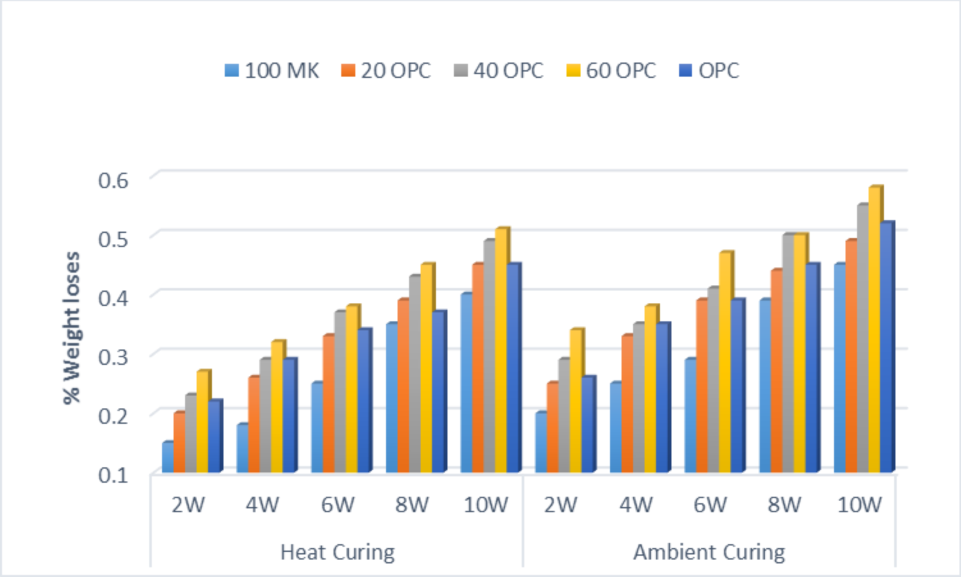

Fig.7 Mass loss of different mortars exposed to sulphuric acid.

Compressive Strength degradation

By means of regular measurement of compressive strength, the strength degradation of specimens exposed to different chemical solutions has been measured. Degradation of compressive strength for geopolymer mortar specimens immersed in different chemical solutions are revealed in Fig. 8-10 for several modules of mortar. The compressive strength degradation was determined as a compressive strength percentage of 28 days before exposure to solutions. It is clear that MK-GPM containing 100% MK indicates a major improvement in resistance of compressive strength degradation at all ages and for both curing conditions.

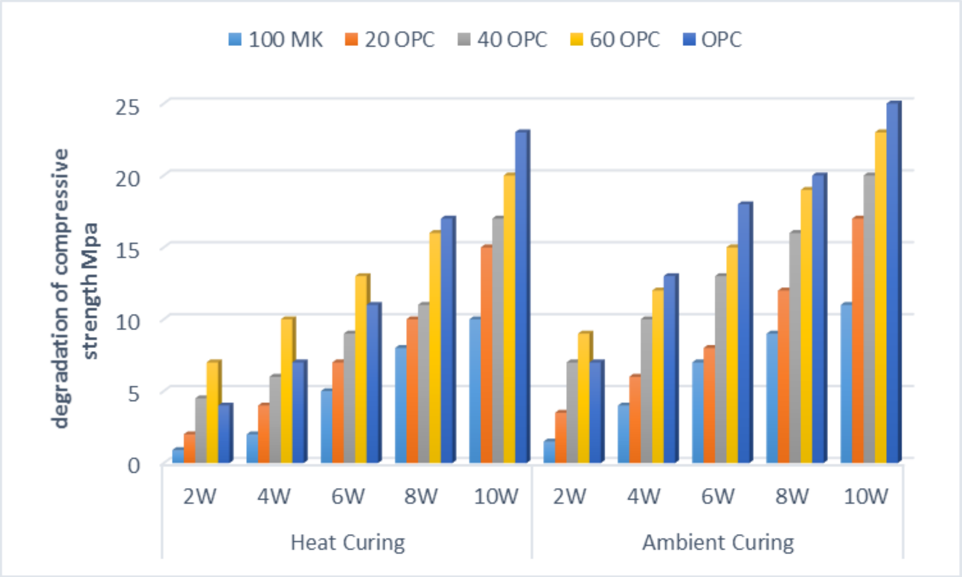

Fig.8 Compressive strength degradation of different mortars exposed to sodium chloride.

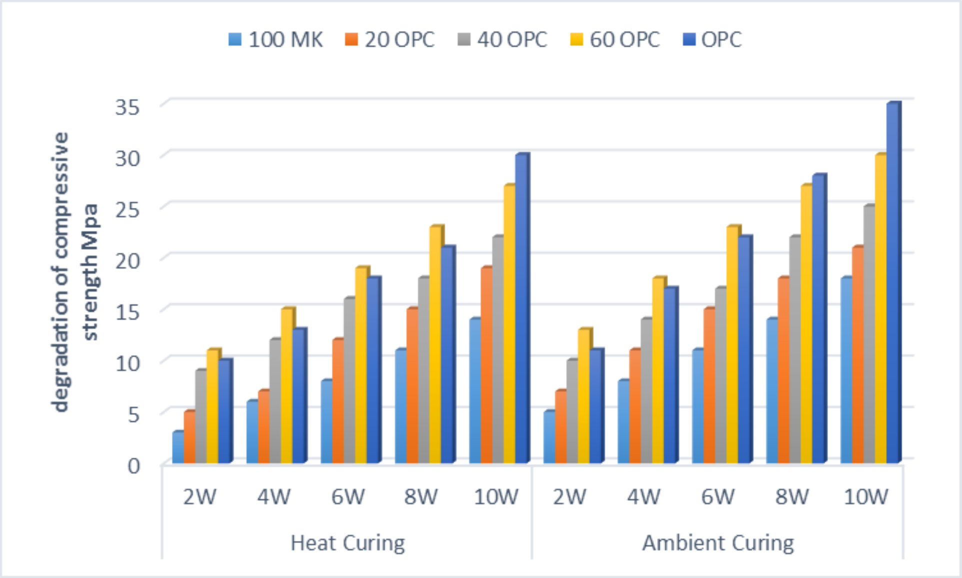

Fig.9 Compressive strength degradation of different mortars exposed to magnesium sulphate.

The amount of improvement is slightly significantly at heat curing than ambient temperature curing. The marked decrease in degradation of compressive strength is largely due to the high pozzolanic reactivity of MK as well as its micro filler effect. However, the degradation in compressive strength of geopolymer mortars increases by increasing OPC content whatever the curing condition [26-28]. It is clear that the degradation of compressive strength due to sulphuric acid exposure is the most pronounced followed by magnesium sulphate followed by sodium chloride. When sulphuric acid is attacked, hydrated of concrete containing high amounts of calcium hydroxide generates gypsum ($CaSO_4$). Moreover, in OPC mortar, calcium silicate hydrate (C-S-H) produces SiO_2, which has a negative effect, in a sulphuric acid environment [29-30].

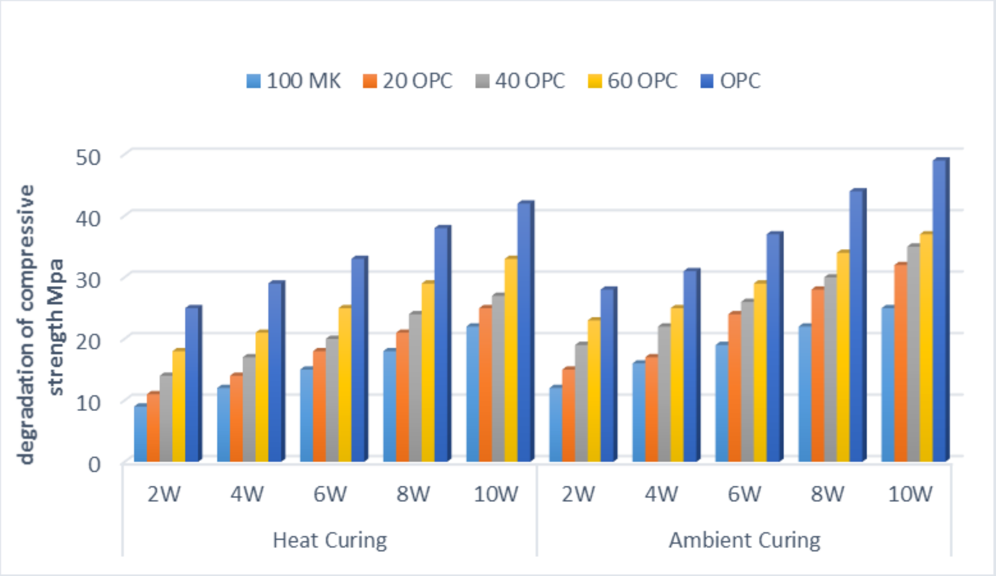

Fig.10 Compressive strength degradation of different mortars exposed to sulphuric acid.

This dissection occurs with less effect in sodium chloride and magnesium sulphate because sulphuric acid is more aggressive than any of anther both environments.The degradation of compressive strength for all studied GPM (100MK, 20OPC, 40OPC and 60OPC) reaches 22, 25, 27, 33 % respectively, while reaches 34% for OPC conventional mortar at 10 weeks without the need for heat curing. The rate of compressive strength degradation gained with time decreases at heat curing compared with ambient curing for all studied GPM mixes. However, this rate increases with OPC inclusion.

CONCLUSIONS

This study investigates the durability of geopolymer mortar matrix and compares the results with OPC mortar. The following conclusions may be written:

Egyptian metakaolin can be regarded as an active pozzolanic mineral admixture that could be applied in geopolymer concrete manufacturing for durability purpose.

In the face of aggressive environments, the geopolymer matrix shows a considerable chemical durability, and would make it possible for it to be used as a construction material in aggressive environments.

The improved performance of MK - GPM in sulphate, chloride and sulphuric solutions was directly attributable to a more stable for polymer structure of cross-linked aluminosilicate compared to the OPC hydration.

Heat curing improves the durability properties of MK-GPM more than ambient temperature curing at all aggressive environments. However, the difference is slightly significant and couldn't restrict the use of geopolymers in field applications.

After 10 weeks of sulfuric acid exposure for different geopolymer mortars, show a gypsum crystal formation. When Compared to 40% OPC and OPC mixes, a 100 % MK produces very dense microstructure and narrow micro cracks.

Despite the superiority of OPC concrete (sorptivity and water absorption) in terms of the capillary technique that limits the access of water to concrete, the geopolymer shows better durability when exposed to environmental attacks. This performance imitates the steadiness of the concrete chemical matrix of the geopolymer.

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