



Dealuminated Metakaolin Modified Geopolymer

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ABSTRACT

De-aluminated metakaolin (DK) is a byproduct of the Aluminum Sulphate industry that is highly non-environmentally friendly where amorphous silica, silicon dioxide, SiO_2 , is its primary component. DK was used as an addition to metakaolin (MK) in fabrication of MK based Geopolymer with 9 ratios of the MK content from 0 to 60 %. Casting was done in two different environments, namely winter and summer, with two different optimum additional water contents. For the DK-modified MK geopolymer fresh mortar properties, flow rate and initial setting time were tested. The ultimate compressive strength (UCS) of standard mortar cubes was tested at 3 and 7 days of age under ambient and wet curing conditions. The rate of gaining UCS for optimum DK content was tracked at early ages, namely; 6, 12, 18 and 24 hours until 10 days of age. The percentage of natural water absorption was investigated for the water cured specimens.

1. Introduction

Davidovits used the term "Geopolymer" in 1978 to describe a category of mineral fasteners with a material organization similar to zeolites but a shapeless microstructure. Geopolymer are made up of two fundamental components: source materials and basic liquids. The raw materials for an alumina silicate based geopolymer should be high in silica SiO_2 and alumina Al_2O_3 . The source materials used to make geopolymer are chosen based on factors such as accessibility, cost, type of application, and explicit interest of the end users. Each source material has advantages and disadvantages. For example, metakaolin as a source material has a high dissolvability in the reactant arrangement, delivers a controlled Si/Al proportion in the geopolymer, and is white in color [1]. Davidovits [2], classified the

application of geopolymer according to the Si/Al ratio. Geopolymerization includes the synthetic response of aluminum and silicon oxides with soluble base polysilicates, yielding polymeric [-Si-O-Al-] building units. Polysilicates are sodium or potassium silicate provided by the synthetic industry or fine silica powder resulting from Ferro-silicon metallurgy [2].

The Most applicable alkaline solutions in geopolymerization is Na(OH) and K(OH). Both produce similar results in fresh and hardened properties, with simple preferences for K(OH) in early ages, but Na(OH) is more practical because it is less expensive and does not require heat during the process, whereas K(OH) produces better results when exposed to heat. Na(OH) also gives better results at late ages than K(OH) [3]. The highest compressive strength is achieved by Geopolymer with the use of Na(OH) or K(OH) solution as an alkaline activator,

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which results in a more porous geopolymer, but Geopolymer with a mixture of Na(OH) or K(OH) solution and soluble silicate (glass water) as an activator have a denser and more homogeneous structure.[4]. P. Duxson et al. [5] demonstrated that Na(OH) geopolymer have a higher dissolution rate and are more reactive to create more ordered species. The concentration of Na(OH) is critical for the alumina silicate source's dissolution capacity, as low molarity causes insufficient Na^+ ions to be interacted with Si^{4+} and Al^{3+} to complete the geopolymerization reaction. On the other hand, too high a concentration causes a faster setting because reaction products form rapidly around unreacted particles, inhibiting further reaction [6], In case of Na-Geopolymer, raising the Na(OH) content from 4 M to 10 M results in a 165 percent increase in compressive strength [7]. It was found that adding water decreased compressive strength, while increasing curing time or temperature increased compressive strength [7]. Initial curing at higher temperatures did not produce significantly higher compressive strength than curing at room temperature. Curing at 70°C appears to significantly improve compressive strength when compared to curing at 30°C for the same amount of time [5].

De-aluminated Metakaolin DK is a by-product of the aluminium sulphate industry that results from metakaolin acid leaching. Kaolin ore is obtained from quarries in South Sinai, specifically from Abu Zenima. It is a sedimentary rock ore, whereas its composition is aluminum silicate, which belongs to the family of clay minerals and is rich in aluminum. As can be seen from Figure 1, thermal hydroxylation at 700°C for 2 hours eliminated the water of crystallization, causing the interior structure to become brittle and the formation of amorphous phase called metakaolin. The calcinated kaolin was milled in a ball mill to produce ground metakaolin. MK reacted with concentrated sulphuric acid to produce aluminum sulphate and alum residue. This aluminium sulphate alum is used in the purification of drinking water. The process produces a solid silicate material (SiO_2) known as dealuminated metakaolin DK. This is followed by a filtration step to separate the aluminium sulphate (alum), from the DK, followed by washing the DK to remove the alum and the solid sludge, which is mostly silica. DK may cause an agricultural soil risk as it interferes with soil nutrient and water cycles, as well as water balance. Dumping DK into the soil involves incorporating it into deeper soil layers to avoid dusting. The risks may involve increasing acidity and toxicity in the soil ecosystem and cultivation.

Despite positive outcomes from the small number

of studies carried out on the use of DK in the construction industry, there are still many aspects that need to be investigated. According to Vollet [9], the de-alumination procedure raises the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio and specific surface area values. The specific surface area rose as a result of the acid attack, which was discovered due to an increase in pore volume without a notable increase in the mean pore size distribution [9]. Stefano Cangiano, and Antonio Princegallo [10] studied DK from two separate sources in order to study how it may be applied in the concrete industry. Because of its high silica concentration, the two samples of DK tested exhibit extremely high fineness and a high level of pozzolanic activity. They came to a conclusion by employing DK as a potential principal component of cement and demonstrating that when ground with clinker, the compressive strength values for mortar tests EN 196-1 are almost equivalent to those obtained for samples containing silica fume and cement. When it comes to Portland cement, the rheology is a little off. [10].



Fig.1: Production of alum and de-aluminated metakaolin [8]

2. Objectives

The main objective of the research work is to produce DK modified MK geopolymer mortar. This may include a review of the literature on the production and properties of metakaolin geopolymer. To document the production aluminum sulphate product from MK and the DK as a by-product, to investigate the production of DK modified MK geopolymer using different DK contents from 0 to 60 % of MK content for determination of the optimum DK content. And to investigate the effect of additional water content on

DK modified MK Geopolymer mortar in order to obtain the optimum additional water content. The study included the fresh properties of DK modified MK geopolymer, namely, flow rate and initial setting time for two different curing regimes through winter and summer, such as ambient and wet curing, on the geopolymer's compressive strength at 3 and 7 days. The natural water absorption of DK modified MK geopolymer after 3 and 7 days was determined during wet curing. The rate of gaining CS for DK modified MK geopolymer at ages 6, 12, 18, 24 hours, and at 3, 7, and 10 days.

3. Materials

The experimental program given in Table 1 focuses on the determination of the optimum parameters that affect the DK modified metakaolin geopolymer. For a constant metakaolin MK content of 200 g and a natural sand of 550 g were used for each mortar cube. These parameters are as follows;

- DK content varies from 0 to 120 g, which represents a ratio of 0 to 60% of metakaolin content.
- Alkali activator content Na(OH) was 100 g that represent 50% of Metakaolin content.
- The concentration of alkali activator Na(OH) was 50%, that represents a mass density of 1.43 g/mL.
- Additional water content ranging from 30 to 35 g, representing 15 to 17.5% of MK content.
- Two curing regimes were used for DK modified MK mortar namely; ambient and wet curing.
- Two ambient conditions: winter time and summer time.

Fresh and hardened properties measured for MK geopolymer mortar were flow test; that was carried out according to ASTM C1437-20 [11] for single specimen of each fresh mortar by using the flow table instrument, initial setting time that was carried out according to ASTM C807-21 [12] for single specimen of each fresh mortar by using Humboldt Vicat's apparatus, percentage of natural water absorption was carried out according to ASTM C128-15 [13] which was measured for wet curing regime samples at 3 and 7 days prior to the UCS test, and compressive strength was carried out for standard mortar specimens according to ASTM C109-20 [14] at 3 and 7 days. Three standard mortar samples were used in the UCS at each age for each mortar mixture.

The standard sand was sieved through a 1.18-mm No. 16 sieve and then retained through a 600- μ m No. 30 sieve. It is a type of natural sand with a sharp, angular, coarse, and clean appearance. It is devoid of any organic matter. The sand was thoroughly washed

and oven-dried before being mixed with the binder.

Kaolin was supplied from "Sinai Co. for Manganese". Kaolin was used to produce aluminum sulphate in Aluminum Sulphate Co. of Egypt ASCE. Figure 2 (a, and b) represent MK and DK used in experimental program.

The chemical analysis was performed using an Axios PW4400 WD-XRF Sequential Spectrometer at the Egyptian Mineral Resources Authority's central laboratories. The chemical composition of MK and DK is given in Table 2, while Table 3 represents their physical properties.

XRD investigation was done on a PANALYTICAL X-Ray diffraction equipment model x pert PRO with monochromatic diffract meter using Cu-radiation ($\lambda=1.542\text{\AA}$) at 45 K.V., 35 M.A. at a scanning speed $0.04^\circ/\text{sec}$. The test was conducted at the central laboratories in the Egyptian mineral resources authority. The reflection peaks were between $2\theta = 2^\circ$ and 60° in which corresponding spacing (d, \AA) and relative intensities of (I/I°) were obtained. Silica was used as an internal standard. Data were identified according to the XRD software. The peak and pattern lists of MK and DK in the XRD test are presented in Figure 3 (a, and b).

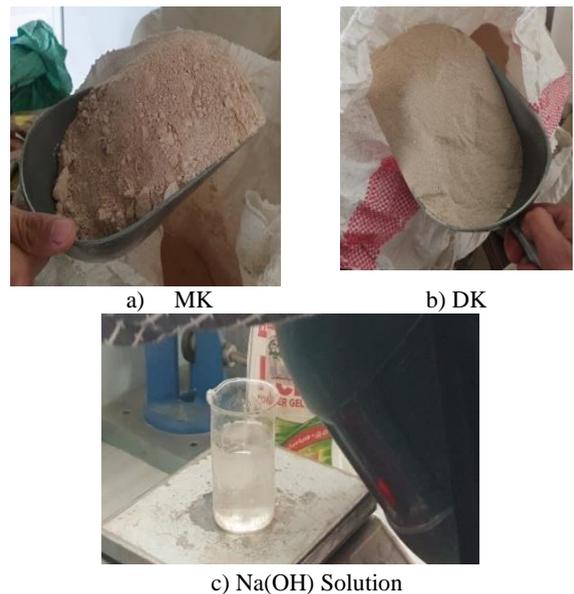


Fig. 2: Materials used

The thermal analysis was done by LINSEIS STA PT1600. The test was conducted at the central laboratories of the Egyptian mineral resources authority. Each powdered sample was heated up to 1000°C at a rate of $10^\circ\text{C}/\text{min}$; the weight, change in Wt., and the thermal behaviour of the samples were recorded in Figure 3 (c and d).

The infrared spectroscopic analysis was carried out using the FTIR-Thermo scientific model

NICOLET6700).Wave number 4000-400 cm-1 (mid region) was used for the analysis. The test was carried out at the central laboratories in the national research center's of housing and construction. The test result is shown in Figure 3 (e and f).

The chemical examination was carried out by Axios (PW4400) WD-XRF Sequential Spectrometer at the central laboratories in the Egyptian mineral

resources authority. The chemical compositions of MK and DK are given in Table 2. Na(OH) solution was acquired from “New Star Company for Chemical Industries” Cairo, Egypt. The Sodium hydroxide Na(OH) solution chemical and physical properties are presented in Table 4. The Na(OH) solution used is shown in Figure 2 (c).

Table 1 Parameters used for the experimental program

MK Content	Natural Sand	DK Content	Alkaline Activator		Additive Water	Curing regime	Ambient condition
			Na(OH) Concentration	Na(OH) Content			
(g)	(g)	(g)	(%)	(g)	(g)		
200	550	0, 20, 40, 60, 80, 90, 100, 110,120	50%	100	30 35	Wet and Ambient	Winter and summer

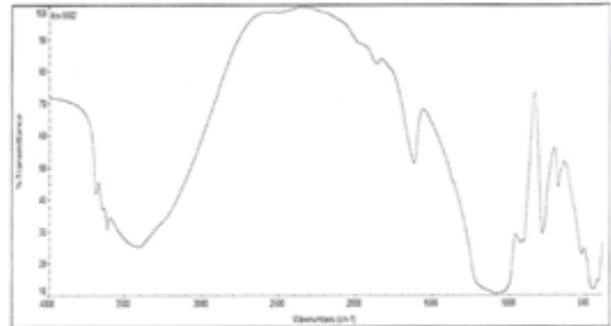
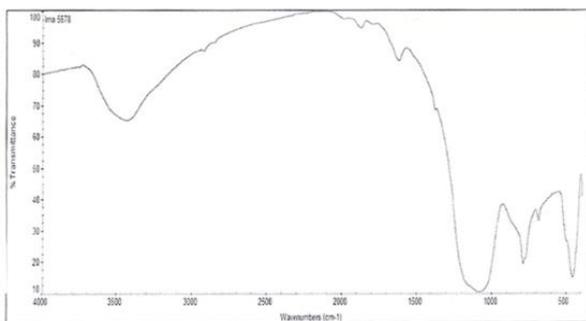
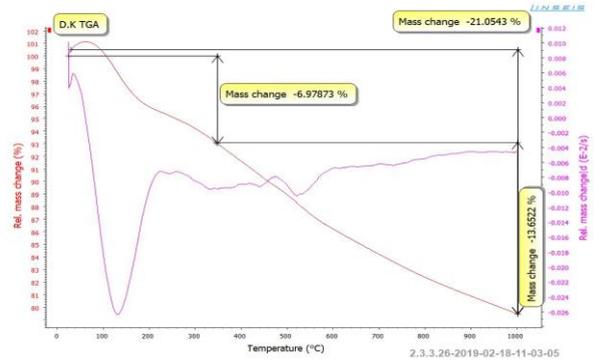
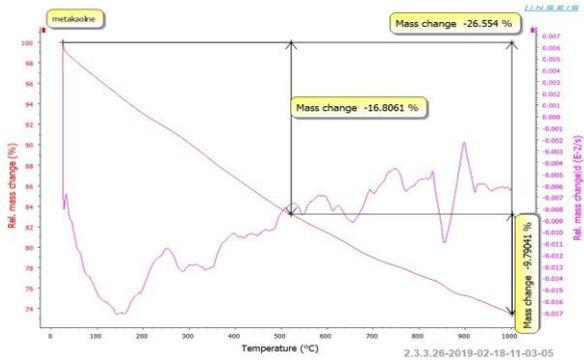
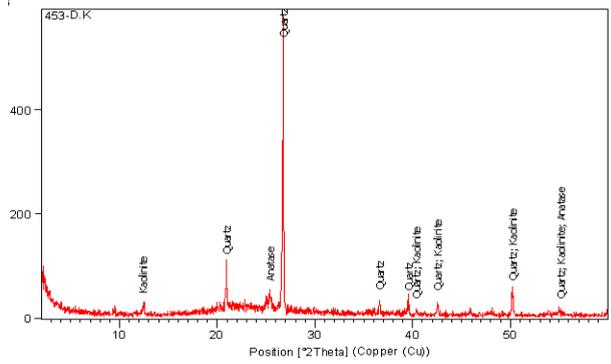
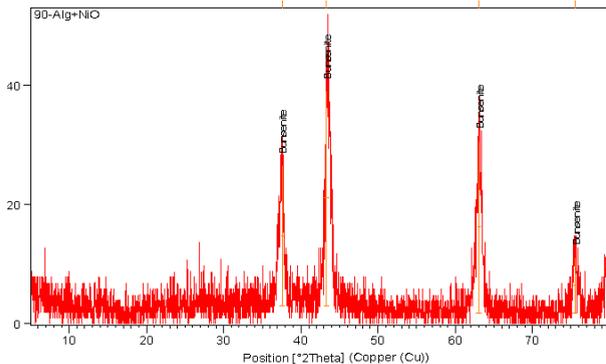


Fig. 3: Materials charchtarization

Table 2 Chemical composition of MK and DK using XRF (Percentage By Weight)

Compound	MK	DK
SiO ₂	62.06	76.78
SiO ₂ amorphous	40.34	49.4
TiO ₂	2.98	3.59
Al ₂ O ₃	30.0	7.91
Fe ₂ O ₃	2.00	0.95
MnO	0.03	0.02
MgO	0.35	0.37
CaO	1.15	1.16
Na ₂ O	<0.01	<0.01
K ₂ O	<0.01	<0.01
P ₂ O ₅	0.02	<0.01
SO ₃	0.25	1.96
LOI	0.90	6.96

Table 3 Physical properties of MK and DK

Property	MK	DK
Specific gravity	2.50	2.10
Mean grain size	2.54 µm	---
Specific surface (cm ² /g)	12000	42000
Brightness	80 Hunter L	---

Table 4 Chemical and physical properties for Na(OH)

Property	
Na(OH) Concentration	50%
Total solid	52.0
Water content	48.0
Specific gravity	1.52
Density g/mL	1.50
Viscosity(cps)	78
Appearance	Liquid gel
Molecular weight	40
Boiling point	145 °C for aqueous solution

4. Experimental work

The cubic moulds used were 70.6x70.6x70.6 mm that were coated with a releasing agent to prevent metakaolin mortar from sticking to the sides of the mould and to facilitate mould removal as shown in Figure 4 (a). When the release agent dries, it forms a thin coating that lubricates the mould, allowing mortar to easily slide out. A manual batching process for each mortar mixture was carried out using weight batching. The MK binder was added to the sand and mixed vigorously in the electric mixer. The pouring was carried out in two layers, where each layer was compacted manually with 2” compacting rod as shown in Figure 4 (b). The fresh mortar mixture inside the moulds was covered with plastic bags until the next day as shown in Figure 4 (c). The specimens were demoulded after 24 hours. The ambient curing specimens were exposed to ambient environment until the date of testing as shown in Figure 4 (e). The wet curing specimens were

submerged in water until the date of testing as shown in Figure 4 (d).



a) Oiled standard moulds



b) Pouring and compaction



c) Covered specimens inside moulds



d) Wet curing regime



e) Ambient curing regimes
Fig. 4: Experimental work

5. Results and Discussion

5.1 Flow Rate

Flow rate test was carried on one single fresh mortar sample for each mortar mixtures in the experimental programme. Figure 5 represents the flow rate of fresh MK mortar for alkaline activator concentration of 50% and Na(OH) content 100 g. For each DK content 2 bar charts represent the additional water content of 30 and 35 g respectively. Increasing the DK content from 0 to 120 g has decreased the consistency of the mortar mixtures due to increasing of fine particles content. Flow rates for geopolymer mortars were very sensitive to the additional water content that increasing additional water content from 30 to 35 g causes significant increasing of flow rate mainly due to increasing in the water-to-binder ratio. A comparison of different flow rate at different DK content at additional water content of 35 g is shown in Figure 6. Table 5 presents the different consistencies' classifications of fresh mortar according to the flow rates limits by the ECP [15]. Increasing the DK

content in the mortar gradually changed the mortar consistency from plastic consistency to dry consistency.

Table 5 Flow rate limits [15].

Flow rate	0 – 20 %	15 -60%	50 -100%	90-120%	110 -150%
Consistency	Dry	Stiff	Plastic	Wet	Sloppy

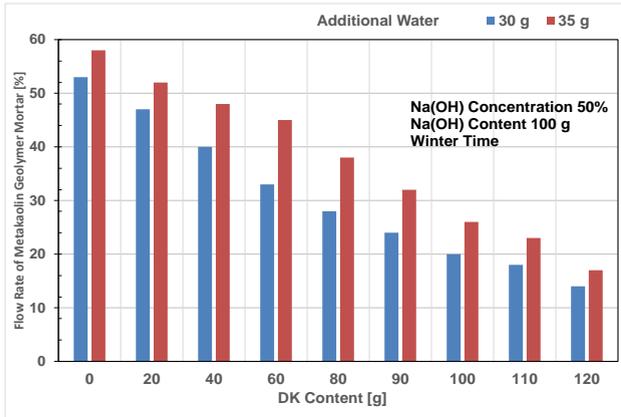


Fig. 5: Flow Rate of MK Geopolymer mortar for optimum contents

5.2 Initial Setting Time

Initial setting time test was carried on one single fresh mortar sample for each mortar mixtures in the experimental programme. Figure 7 (a and b) represents the initial setting time of fresh MK mortar for alkaline activator concentration of 50 % and Na(OH) content 100 g through winter and summer. For each DK content two bar charts represent the additional water content of 30 and 35 g, respectively. Increasing the DK content from 0 g to 120 g has decreased the setting time of the mortar mixtures due to increasing of fine particle content. Setting time for geopolymer mortars was very sensitive to the additional water content. Increasing the additional water content from 30 g to 35 g caused a significant increase in the setting time rate, mainly due to the increased water-to-binder ratio.

Table 6 OPC Initial setting time limits reference [15]

Cement grade	Initial setting time
N32.5 & R 32.5	≥ 75 minutes
N42.5 & R 42.5	≥ 60 minutes
N52.5 & R52.5	≥ 45 minutes



a) Plastic flow at 0 g DK



b) Stiff flow at 100 g DK



c) Dry flow at 120 g DK

Fig. 6: Flow rate at different DK content at 35 g water

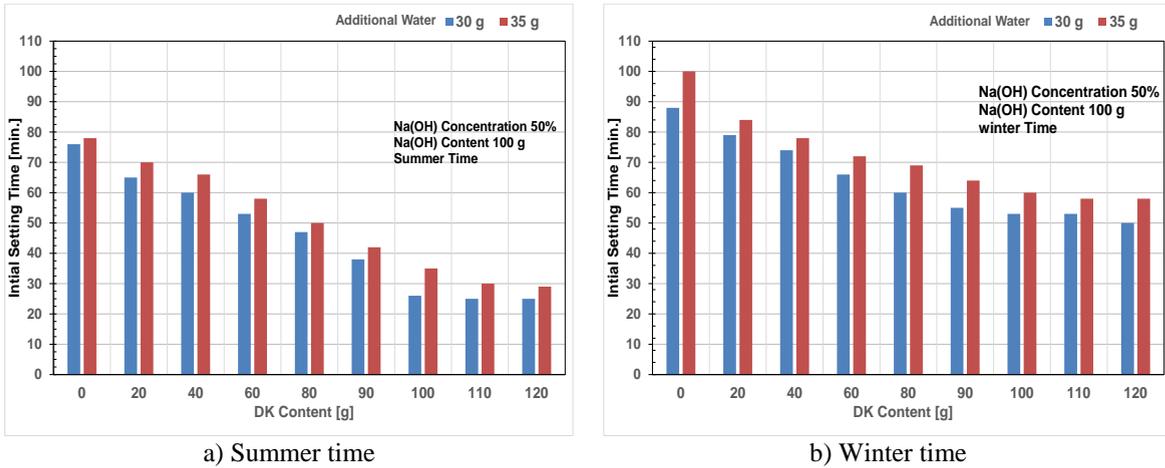


Fig. 7: Initial Setting time

Table 6 presents initial setting time limits according to the Egyptian Code of Practice [15] for different grades of cement specified in the code. As can be seen from Table 6, all mixtures, during winter time, satisfies the initial setting time requirements of the ECP [15] regardless of the DK content. During summer time, MK geopolymer mixture with low DK content <40 % of the MK binder content satisfies the initial setting time requirements of the ECP [15].

5.3 Natural percentage water absorption:

Figure 8 represents the natural water absorption percentage of fresh MK mortar for an alkaline activator concentration of 50% and Na(OH) content of 100 g. Figures 8 (a and b) represent the natural water absorption percentage of fresh MK mortar for additional water content of 30 and 35 g, respectively. For each DK content two bar charts represent the 3 and 7 days of water absorption, respectively. Increasing the DK content from 0 g to 120 g has decreased the water absorption of the mortar mixtures due to the increased fine particles content. The water absorption of geopolymer mortars was very

sensitive to the additional water content, and increasing the additional water content from 30 g to 35 g caused a significant decrease in the water absorption, mainly due to increasing the water-to-binder ratio and the saturation of the pores at early ages reduced the percentage of water absorption. The geopolymer samples were de-moulded after 24 hours. Curing of the geopolymer mortar mixtures for 3 and 7 days has been done by submersion of the specimens for 2 and 6 days, respectively. The 7-day water absorption gave higher values than the 3-day natural water absorption. Yet, the majority of the natural water absorption of specimens was recorded in the first 3 days for all geopolymer mortar mixtures, with a lower rate of increase in natural percentage water absorption with time. This may be due to an improved polymerization process that results in a denser solid phase that reduces the rate of natural percentage water absorption. This may refer to the completion of the chemical reaction process in the geopolymer mortar.

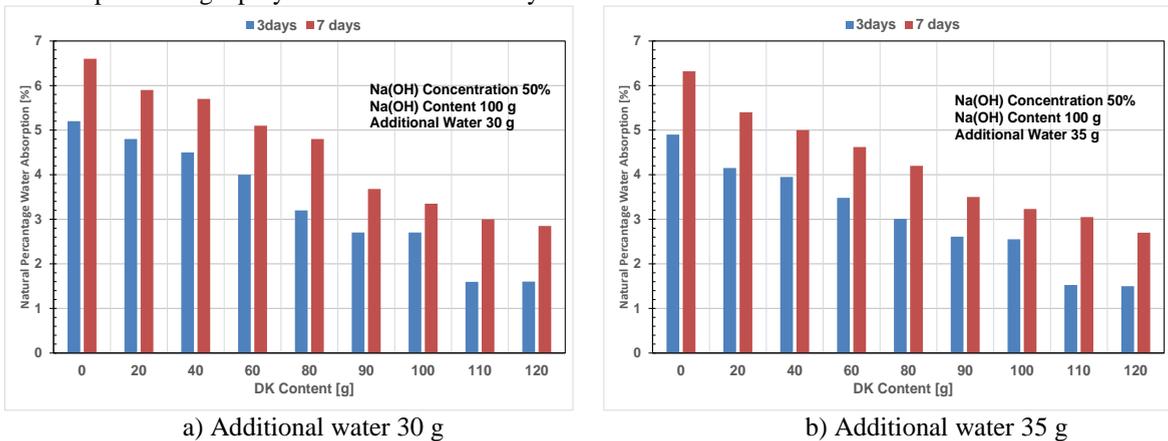


Fig. 8: Natural water absorption

5.4 Ultimate Compressive Strength:

The test samples were cured for periods of 3 days and 7 days using two different curing regimes, namely, ambient and wet curing. Figure 9 represents the typical mode of failure mode for AAS mortar samples under axial compressive stresses. Figure 10 represents the 3 and 7 days ultimate compressive strength UCS for ambient and wet curing of fresh MK mortar for alkaline activator concentrations of 50%. Figures 10 (a, and b) represent the compressive strength of fresh MK mortar with additional water content of 30 during winter and summer time respectively.

Figures 10 (c, and d) represent the compressive strength of fresh MK mortar with additional water content of 35 during winter and summer time respectively. In each figure, the DK content is represented on the horizontal axis where four bar charts represents the 3- and 7-day UCS using two different curing regimes. Table 7 presents the average coefficients of variance for the UCS tests results presented in Figure 10.



Fig. 9: Compressive strength test

Increasing the DK content from 0 g to 100 g increased the UCS of mortar mixtures, but then it began to decrease until it reached a lower value at DK content of 120 g. Thus, the 3-day and 7-day UCS were reached at a DK content of 100 g, which represents, again, 50% of the MK content of 200 g. This may suggest that the effect of microsilica in the geopolymer mixtures that increased geopolymerization products in the mortar was optimised at a DK content of 50% of the MK content.

The UCS for geopolymer mortars was very sensitive to the additional water content. The UCS decreased as the additional water content increased from 30 or 35 g caused a significant decrease in the UCS. This behaviour is mainly due to increasing the water-to-binder ratio results in the formation of large internal voids. The optimal amount of additional water was 15% of the MK content.

The 7-day UCS gave higher values than the 3-day UCS. Yet, the majority of the UCS was recorded in the first 3 days for all geopolymer mortar mixtures, with a lower rate of increase in compressive strength with time. This may be due to an improved polymerization process that results in a denser solid phase and reduces the rate of gain in compressive strength. This may refer to the completion of the chemical reaction process in the geopolymer mortar. The 3-day and 7-day of ambient cured UCS recorded were consistently higher than the 3-day and 7-day of wet-cured UCS, respectively. The geopolymer samples were demoulded after 24 hours of casting. The 3-day and 7-day UCS specimens for wet cured geopolymer mortar mixtures have been submerged in water for 2 and 6 days, respectively. Ambient curing is a great advantage when producing in-situ geopolymer concrete.

Table 8 represents the UCS limits for different cement grades according to the Egyptian Code of Practice ECP [15]. As can be seen from table 8, the DK modified Geopolymer mortar with the optimum DK content of 50% of the MK binder content satisfies the UCS requirement for the ECP [15] limits for all grades of cement specified in the code.

5.5 Effect of additional water on UCS.

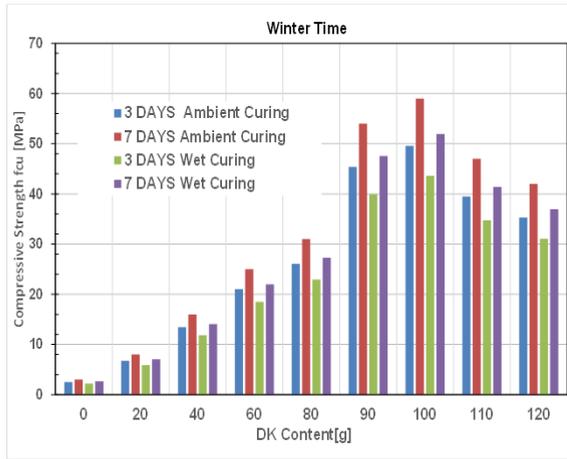
Figures 11 (a, and b) represent the 3- and 7-day UCS for ambient curing of fresh MK mortar for an alkaline activator concentration of 50% both in the winter time and the summer time. Table 9 presents the average UCS and the coefficients of variance for the UCS tests results presented in Figure 11. For each additional water content two bar charts represent the 3-day and 7-day UCS, respectively. The UCS for geopolymer mortars was very sensitive to the additional water content and increasing the additional water content from 0 g to 30 g or 35 g caused a significant increase in the UCS. The UCS began to decrease as water content increased beyond 35 g, then it reached its lowest UCS at a water content of 50 g. This behaviour is mainly due to increasing the water-to-binder ratio, which results in the formation of large internal voids. The optimum additional water contents ranged between 30-35 g that represents 15-17.5 % of the MK binder content.

Table 8 Early compressive strength limits for different OPC grades [15]

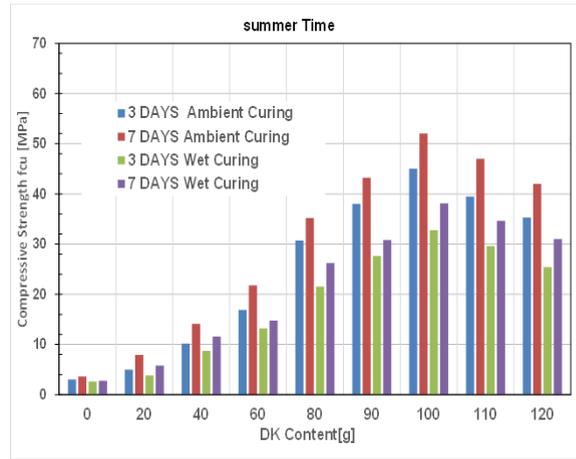
Cement grade	Early compressive strength	
	3 days	7 days
N32.5	-	≥ 16
R32.5	≥ 10	-
N42.5	≥ 20	-
R42.5	≥ 20	-
N52.5	≥ 30	-

Table 7 Average coefficients of variance for the UCS tests results for DK modified MK mortar

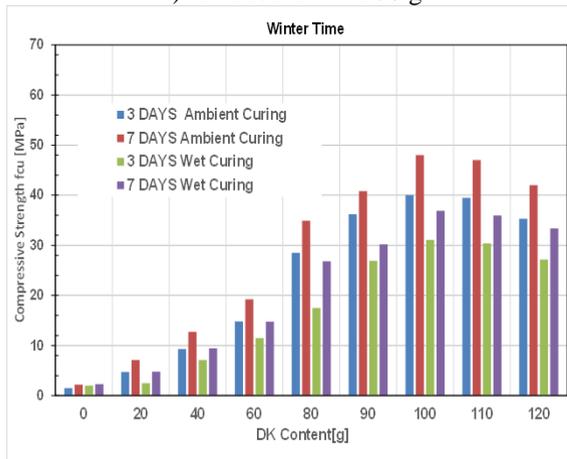
Additional water g	Environmental Conditions	Curing regime	Coefficients of variance		Curing regime	Coefficients of variance	
			for 3 day %	for 7 day %		for 3 day %	for 7 day %
30	Winter	Ambient curing	3.72	2.84	Wet curing	10.96	8.73
	Summer		12.6	13.8		4.15	2.45
35	Winter		7.2	9.05		3.98	5.12
	Summer		5.13	6.62		6.45	4.31



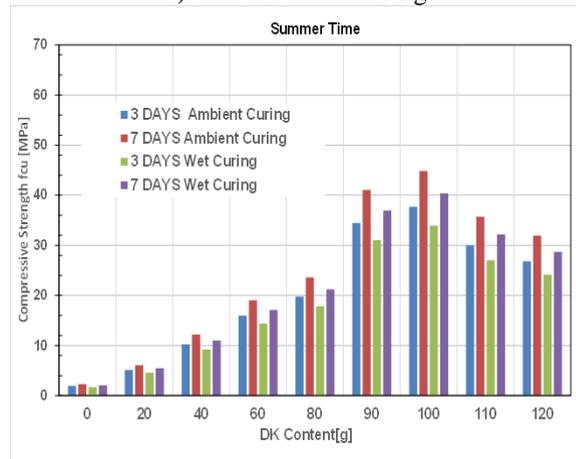
a) Additional water 30 g



b) Additional water 30 g



c) Additional water 35 g



d) Additional water 35 g

Fig. 10: Compressive strength through winter/summer time

Table 9 Average UCS and coefficients of variance for DK modified MK mortar

Additional water g	Winter time				Summer time			
	3 day		7 day		3 day		7 day	
	Average UCS MPa	Coefficients of variance %						
0	12	2.47	16	7.14	6.5	1.84	8	3.50
25	24.7	6.18	39.5	6.52	13.2	3.62	20.7	7.19
30	47.3	3.85	63	9.63	45	10.37	52	5.33
35	41.2	4.41	55.1	5.71	40	8.91	48	9.20
50	28.5	8.33	35.6	6.49	22.9	5.79	27.5	7.66
65	24	3.66	33.2	4.55	15.2	2.30	23.6	6.10
85	21.9	5.19	29	3.97	10.1	3.46	19	4.72

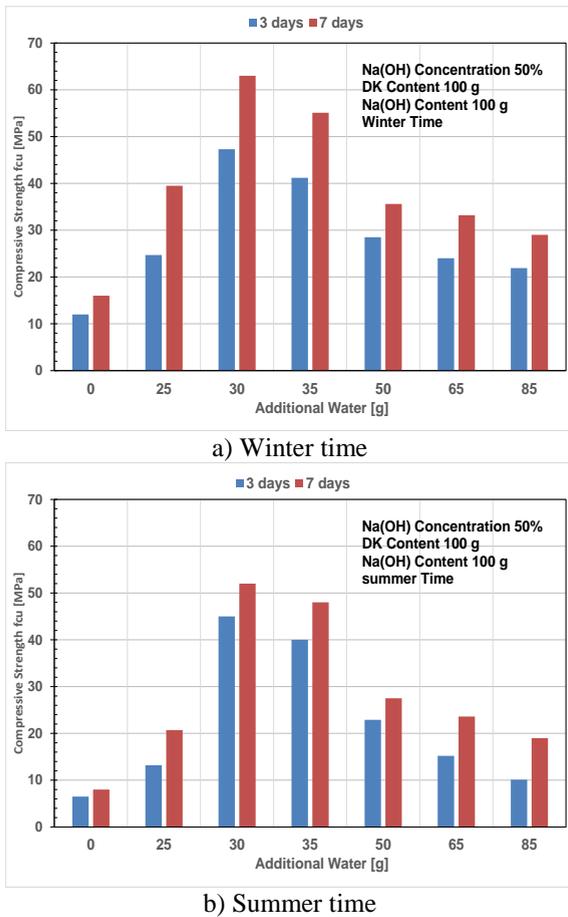


Fig. 11: Compressive strength of optimum mix for different additional water

5.6 Rate of gain Compressive strength

In this series, the optimum DK content and the optimum additional water content were used namely; 50% and 15/17.5% of the MK binder content. Two different curing regimes were investigated namely; ambient and wet curing. In addition, two environmental conditions were checked namely; at winter time and at summer time. The specimens were sealed in the first 24 hours of age in plastic bags. The specimens were tested at 6, 12, 18 and 24 hours. Only tested specimens were de-moulded from their moulds prior to testing. After the first 24 hours, the rest of samples were de-moulded and cured for periods of 10 days using two different curing regimes, namely, ambient and wet curing. Figure 12 represents the rate of gain in ultimate compressive strength of fresh MK mortar for an alkaline activator concentration of 50%.

The ultimate compressive strength increased as the age of the mortar increased. The highest rate of gain of strength took place during the first 24 hours of age. Recall, the specimens were kept inside the moulds during that period. In addition, the rate of

gain of strength until 3 days was much higher than the rate of gain of strength at later ages. Ambient curing gives higher results than wet curing at all ages. The rate of gain of strength is sensitive to the additional amount of water where the higher rate of gain of strength is associated with the lower additional water content. The rate of gain strength through winter time was faster than during summer time.

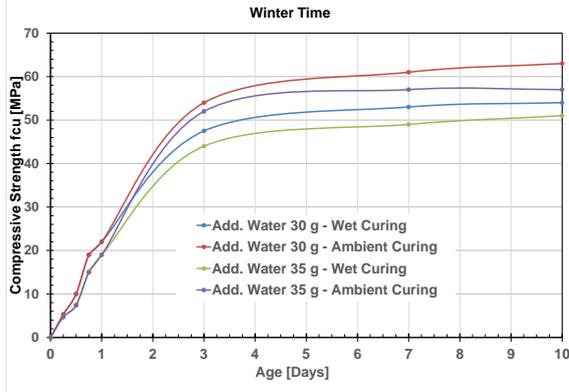
5.7 Geopolymerization Evidence

Figure 13 represents Scanning Electron Microscope SEM images carried out in the national research center's for housing and construction. According to the micrograph illustrated in Figure 13, the microstructure of the geopolymer is a fine, porous, heterogeneous mixture of the reacted MK grains, residual alkaline precipitates, and geopolymer gel. Figures 13 (a and b) show the geopolymer microstructure at 500 and 2000 times magnification, respectively. The dense and bulky base is composed of gel-like substances and can be characterised as geopolymer binders. It consists of a dense, continuous gel-like layer with microcracks and micropores that can be clearly observed on the surface. The microcracks shown in Figures 13 (a and b) were caused by the drying shrinkage, a physical property inherent in the gel. The gel shrinkage occurs because the removal of water during polycondensation creates capillary tension within the gel matrices, as seen in SEM micrographs of DK/MK-based geopolymers at 2000 magnification. The unreacted alkali precipitated in the form of white, rose-shaped particles. Based on the analysis of the microstructure of the DK/MK geopolymer synthesized, the geopolymer products consist of many features: voids, pores, microcracks, and dense and bulky geopolymer binders. The adherence of the crust to the sphere does not appear to be very strong, and the bonding between grains is produced through the geopolymerization reactions. From the analysis of the microstructure of DK/MK geopolymer binder, the geopolymer structure consists of many features such as dense geopolymer binder matrix, MK particles, voids, micro pores and NaOH precipitate. The excess NaOH may cause future developing of compressive strength.

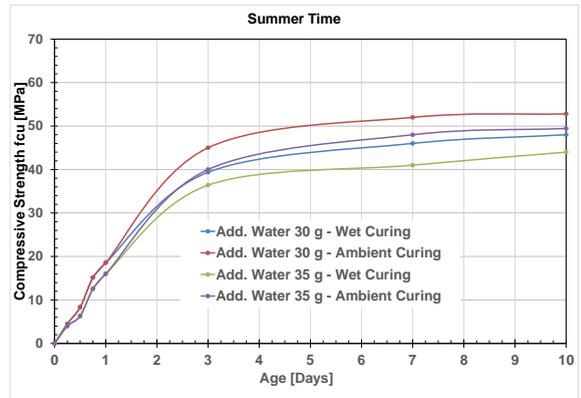
EDAX, energy dispersive X-ray analysis is a leading provider of innovative material characterisation system. Quantitative EDAX analysis is listed in Figure 14 (a). The overall Si/Al and Na/Al ratios determined by EDAX analysis match those of the initial precursor mixtures. It is noticed that elements are affected by the introduction of MK to form a mixture with the DK, and the effect

increases when curing at the thermal state. Qualitative EDAX analysis at Figure 14 (b) confirmed the presence of major elements Si, Al, Na, and O, which promote geopolymer formation. However, other elements appeared in impressive proportions, such as Ca, Mg, S, and Fe. This collection of elements achieved the geopolymer structures of compressive strength of 62 MPa. The

ratios of Si/Al and Na/Al noticed from EDAX analysis are matched to those of the initial precursor mixtures. The EDAX analysis showed that different proportions of silicon, aluminium, sodium, and oxygen elements are proportional to different ratios of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ in the geopolymer mixes.

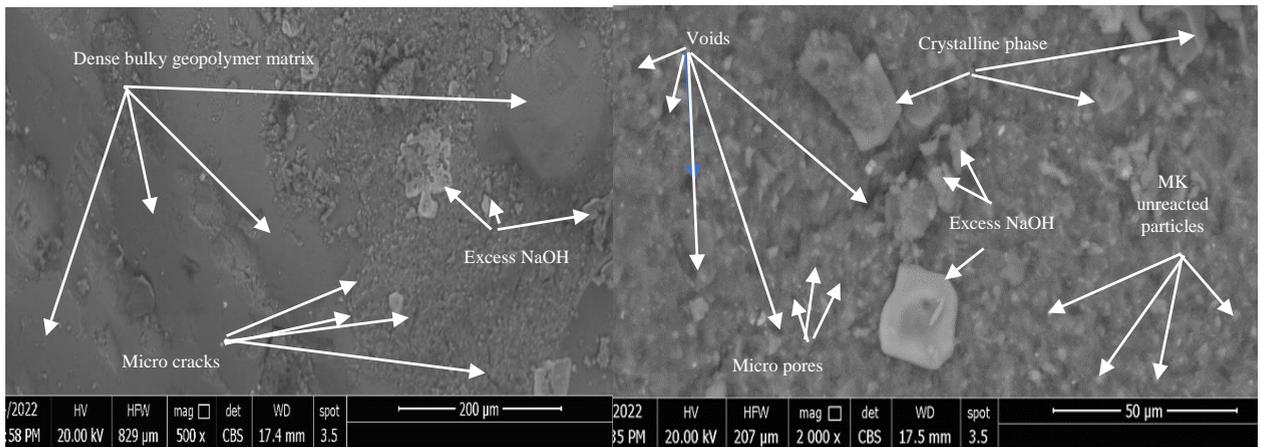


a) Winter time



b) Summer time

Fig. 12: Rate of Gain Strength for optimum mix (DK content = 100g)



a) At 500 magnification

b) At 50 magnification

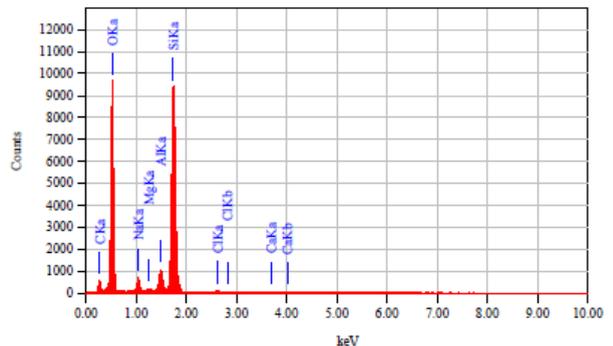
Fig. 13: SEM micrographs of MK/DK based geopolymer

Thin Film Standardless Standardless Quantitative Analysis

Fitting Coefficient : 0.0221

Element	(keV)	Mass%	Counts	Sigma	Atom%	Compound	Mass%	Cation	R
C K	0.277	5.31	3102.36	0.06	8.97				3.1932
O K	0.525	40.62	61363.90	0.19	51.51				1.2344
Na K	1.041	2.49	4896.93	0.06	2.20				0.9497
Mg K	1.253	0.37	746.91	0.03	0.31				0.9256
Al K	1.486	4.16	7955.96	0.08	3.13				0.9754
Si K (Ref.)	1.739	46.44	86598.94	0.26	33.55				1.0000
Cl K	2.621	0.33	455.98	0.03	0.19				1.3620
Ca K	3.690	0.28	305.06	0.04	0.14				1.6876
Total		100.00			100.00				

a) Quantitative



b) Qualitative

Fig. 14: EDAX of MK/DK based geopolymer

6. Conclusions:

The current research work may lead to the following conclusions;

1. Increasing the DK content from 0% to 60% of MK content has decreased the consistency of the mortar mixtures due to increasing of fine particles content. The consistency of mortar was plastic at 0% of DK content. While at DK content of 60% of MK content, the mortar consistency reaches dry consistency according to the ECP [15].
2. Increasing the DK content from 0% to 60% of the MK binder content has decreased the setting time of the mortar mixtures. Initial setting times for Geopolymer mortars were very sensitive to the additional water content that increasing additional water content from 15% to 17.5% of the MK binder content caused significant increase of the initial setting time mainly due to increasing the water-to-binder ratio. The initial setting time in winter was much higher than setting time in summer. This may be speculated due to the effect of ambient temperature on the geopolymerisation process. During winter time, the initial setting times satisfied requirements of the ECP [15] regardless of the DK content. During summer time, MK geopolymer mixtures with low DK content $\leq 40\%$ of the MK binder content satisfied the initial setting time requirements of the ECP [15]. This may require further investigation for innovative solutions to increase initial setting time for Geopolymer mortar to satisfy ECP [15] limits.
3. Increasing the DK content from 0% to 60% of the MK content has decreased the natural percentage water absorption of mortar mixtures. This may suggest that increasing fine content in the mortar may reduce the natural percentage water absorption similar to densification effect of micro silica in the geopolymer.
4. Increasing DK content from 0% to 50% of the MK binder content has increased the UCS of mortar mixtures then it began to decrease until it reached lower value at DK content of 60%. The highest 3-day and 7-day UCS were reached at the optimum DK content of 50% of the MK binder content. This may suggest that the effect of microsilica in the DK in the Geopolymer mixtures had increased geopolymerization products in the

mortar and that was at an optimum DK content of 50% of the MK content.

5. The UCS for geopolymer mortars was very sensitive to the additional water content. Increasing the additional water content to 15/17.5% of the MK binder content caused significant increase in UCS. The UCS began to decrease as additional water content increased beyond 17.5% of the MK binder content. The 7-day UCS gave higher values than 3-day UCS. The UCS was always higher at winter time rather than at summer time and with ambient curing rather than wet curing. The UCS of Geopolymer mixtures with optimum DK content in both environmental conditions satisfied the ECP [15] strength limits for cement grade R52.5.
6. The UCS increased as the age of the mortar increased. The highest rate of gain of strength took place during the first 24 hours of age. In addition, the rate of gain of strength until 3 days was much higher than the rate of gain of strength at latter ages. Ambient curing gives higher results than wet curing at all ages. The rate of gain of strength is sensitive to the additional amount of water where the higher rate of gain of strength is associated with the lower additional water content. The rate of gain strength through winter time was faster than during summer time. This may aid in buildings' construction that need early high strength that will help in form work removal at early ages.

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