



Production and Properties of Alkali Activated Slag Modified Using DK and MK

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ABSTRACT

De-aluminated Metakaolin (DK) and Metakaolin (MK) were studied as partial replacement of slag binder using 7 and 4 different ratios respectively to produce alkali activated slag (AAS) mortar. Different material characterization tests were carried out to investigate the properties of the Slag, DK and MK by using X-ray fluorescence (XRF), X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) and Fourier-transform infrared spectroscopy (FTIR). A comparable study between DK and MK partial replacement on the AAS mortar physical and mechanical properties was carried out. Alkali activator used, was a mixture of Sodium Hydroxide Na(OH) solution and soluble Sodium Silicate using 4 different mixing ratios. Three different percentages of additive water were also checked. Flow rate and initial setting time were tested for the fresh modified AAS mortar samples. The compressive strength of modified AAS hardened mortar samples were tested at 3 and 7 days, under 2 different curing regimes namely; ambient and wet curing to investigate the effect of both curing on both mortars. The percentage of natural water absorption was tested for wet curing regime. The increase in DK and MK content had led to decrease in the flow rate, the initial setting time and percentage of natural water absorption. The increase of additive water increased the compressive strength until a certain ratio and then reduced the compressive strength beyond that. The effect was the same for both DK and MK modified mortar. The optimum ratio of the alkaline activator composition for DK modified mortar differed from MK modified mortar.

1. Introduction

The golden rule in the field of environmental preservation from waste dangers is to recycle this waste production at its source. De-aluminated metakaolin (DK) is one of the by-products generated from the aluminium sulphate industry in Egypt, as shown in Figure 1 [1]. Kaolin ore is supplied from quarries in South Sinai, notably Abu Zenima; it is a sedimentary

rock with an aluminium silicate composition. It belongs to the clay mineral family, which is rich in aluminium. Metakaolin (MK) is created after a thermal process at 700 °C that removes molecular water from crystallisation water, making the interior structure unstable.

This is followed by the chemical reaction stage, in which sulphuric acid reacts with aluminium silicate in metakaolin to produce aluminium sulphate (alum). In

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the reaction, a solid amorphous silicate material (SiO_2) is left behind, called de-aluminized meta-kaolin. This is followed by a filtration step to separate the aluminium sulphate solution (alum) from the solid sludge (DK). The DK is thoroughly washed to remove the alum solution, and the solid sludge, which is primarily amorphous silica (SiO_2), is disposed of in a sanitary landfill.

The DK had an adverse impact on the environment. It is considered one of the most harmful wastes of the aluminium sulphate industry, with an annual local productivity of about 100,000 tonnes and an estimated annual global output of around 7.5 million tonnes that harms the agricultural land on which these wastes are dumped. The reason for that is the acidity of the DK composition, which makes it extremely corrosive and damaging to soil and life forms and presents a massive problem for safe disposal [2].

DK is such a significant environmental problem that needs effective solutions. Currently, the majority of DK is dumped directly in the dump fields. It has resulted in farmland encroachment, groundwater pollution, the possibility of dam failure, and the loss of waste resources. DK have some beneficial in its component are not adequately reused. The minor recycling process of alum residue was done due to the fact that it contains raw ingredients for the manufacturing of silicon and aluminium. Yet, this is due to the fact that it is less expensive to dump alum residue as waste rather than continue mining for new ores; only about 1% to 2% of waste is recycled, and the rest is dumped.

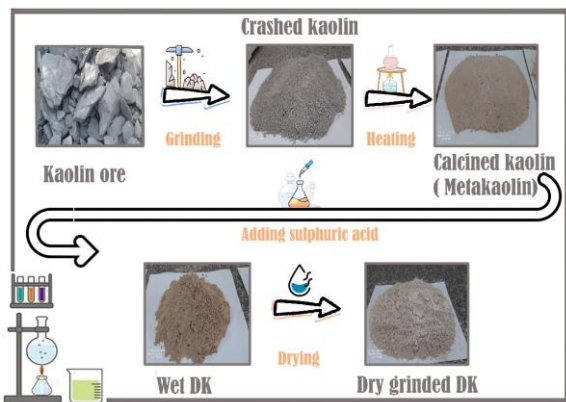


Fig. 1: Production steps of DK [1]

Over the past 40 years, researchers have focused their efforts on conducting research and investigations on alkaline activated materials (AAM), which they believe could replace cement partially or fully in mortar or concrete [3]. Alkali-activation is a technology that is growing globally rapidly and depends on the chemical

reaction between a solid alumina silicate powder binder and an alkaline activator at different curing temperatures below $100\text{ }^\circ\text{C}$, giving a hardened product [4] that leads to the production of an amorphous or semi-crystalline three-dimensional polymeric structure consisting of Si–O–Al bonds [5].

There are different types of activators, which are divided into three classes: low-hydration alkaline activators, medium-hydration alkaline activators, and high-hydration alkaline activators. The activated materials may be agricultural or industrial wastes or materials rich in silicate, such as broken glass or rice husk ash. The activators are sodium hydroxide $\text{Na}(\text{OH})$, potassium hydroxide $\text{K}(\text{OH})$, and glass water (sodium/potassium silicate). Each alkaline activator composition and type will have a different effect on the properties of alkali activated slag (AAS) mixtures [6]. One of the most important factors influencing the consistency and setting time of fresh binder, as well as mechanical properties such as compressive strength of alkali-activated binder materials, is the type of alkaline activator used [7, 8].

The workability and initial setting time were longer in samples that had a content of silica fume and slag than in samples that only contained slag [9]. The optimum partial replacement of slag content when Metakaolin was used was determined at 70% slag to 30% Metakaolin, which achieved the highest compressive strength [10]. The partial replacement of slag by Metakaolin increased compressive strength by 40% compared to mixtures that contained only slag [11]. Changing a mixture's water-to-binder ratio (w/b) can influence compressive strength development and fresh properties [12]. More than a ratio of 0.42 in w/b had a negative impact on the mechanical performance of alkali-activated slag [13]. The wet curing had led to a decrease in the compressive strength compared to the ambient curing [14]. Due to the appearance of cracks inside the sample, air ambient curing resulted in a continuous increase in compressive strength at an earlier age than wet curing [15].

2. Objectives

Any material consisting of alumina and silicate could theoretically be alkali activated [16]. DK as a by-product of aluminium sulphate production has an amorphous silica; i.e. silicon dioxide SiO_2 and aluminium oxide Al_2O_3 as the primary constituent [17]. The main objective of this research work is to use MK and DK material as partial replacement of slag binder. Also, investigating the production of DK and MK modified mortar using seven different DK content and four different MK content. The alkaline activator

solution consists mixture of soluble sodium hydroxide Na(OH) and soluble sodium silicate (Glass Water GW) with four different alkaline solution composition namely 1:0, 1:1, 1:2 and 1:3 to determine the optimum alkaline solution composition. Moreover expose samples to various fresh and hardened tests to determine the optimum mix.

3. Experimental Work

3.1. Materials

As can be seen in Table 1, the experiment work programme focuses on the use of DK and MK as part of the binder for slag alkali activated mortar. A constant binder content of 200 gm of slag and a constant standard sand content of 550 gm as a fine aggregate were used. The optimum binder was examined by gradually partially replacing the slag with DK in a ratio of 0, 20, 40, 60, 80, 100, and 120 gm, and then gradually replacing the slag with MK in a ratio of 0, 40, 60, 70, 80, and 100 gm.. A constant content of the alkaline activator made up of Sodium Hydroxide solution Na(OH) and soluble Sodium Silicate (GW) was equal to 50% of the binder content; i.e. 100 gm. The Na(OH) concentration was 52%, and four ratios of Na(OH) to (GW) composition were investigated, namely 1:0, 1:1, 1:2, and 1:3. Three different additive waters, namely 25, 35, and 50 gm, were used. Twelve standard mortar samples were cast for each mixture. Samples were exposed to two curing regimes, namely ambient and wet curing regimes, to examine the effect of curing regimes on their mechanical properties. Six specimens were used for each curing regime.

The fresh mortar property was examined by conducting the flow test according to ASTM C1437-20 [18] for flow testing for fresh mortar by using the flow table instrument for a single sample for each mixture. The initial setting time was conducted according to ASTM C807-21 [19] for testing the initial setting time for mortars by using Humboldt Vicat's apparatus for a single sample for each mixture.

Hardened mortar properties were examined by conducting the percentage of natural water absorption after conducting the wet curing regime on the samples at 3 and 7 days and before conducting the compressive strength on them. The percentage of natural water absorption was the average of three specimens at each age. The compressive strength test was conduct on 3 samples according to ASTM C109-20 [20] at 3 and 7 days after subjecting the specimens to ambient and wet curing regimes. Three cubical moulds of size 70.6 x 70.6 x 70.6 mm were used at each age.

The sand used was passing sieve 1.18 mm (No.16) and retained in sieve 600 μm (No.30) according to

ASTM C128-15 [21]. It is standard sand characterize by sharp, angular, coarse, and clean in appearance. It is completely devoid of organic stuff. The sand washed then dried to be ready to mix with the binder. Slag is Ground Granulated Blast Furnace Slag (GGBFS) and it is collected from steel production process. Slag is Indian product as shown in Figure 2 (a). MK and DK were collected from Aluminum Sulphate Company in Egypt (ASCE), as a by-product from alumni production process as shown in Figure 2 (b, and c).

3.2. Instruments

Different material characterization tests were carried out to investigate the properties of the Slag, DK and MK by using X-ray fluorescence (XRF), X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) and Fourier-transform infrared spectroscopy (FTIR). XRF, XRD and FTIR tests were carried out at the central laboratories in the Egyptian Mineral Resources Authority while the FTIR test was carried out at the central laboratories in housing and building national research centre. The chemical and physical examination of slag, DK and MK was carried out by applying Axios (PW4400) WD-XRF Sequential Spectrometer as shown in Table 2 and 3 respectively. The XRD investigation was carried out using a PANALYTICAL X-Ray diffraction equipment model x'pert PRO with monochromatic diffractometer using Cu-radiation ($\lambda=1.542\text{\AA}$) at 45 K.V., 35 M.A. and scanning speed $0.04^\circ / \text{sec}$. were used. The reflection peaks between $2\theta = 2^\circ$ and 60° in which corresponding spacing (d, \AA) and relative intensities (I/I \AA) were obtained for Slag, DK and MK as shown in Figure 3 (a, b and c) respectively. Silica was used as an internal standard. Data were identified according to the XRD software as shown in Table 4 and 5 for DK and MK. The thermal analysis was carried out using LINSEIS STA PT1600. Each powdered sample was heated by $10^\circ\text{C}/\text{min}$. up to 1000°C temperature, weight, change in Weight., and the thermal behaviour of the samples are recorded in the charts as shown in Figure 4 (a, b and c) for slag, DK and MK respectively. The infrared spectroscopic analysis was carried out using thermo-scientific model (NICOLET6700). The analysis was scanned in wave no. region $4000\text{--}400\text{ cm}^{-1}$ (mid region) as shown in Figure 5 (a, b and c) for Slag, DK and MK respectively.

Soluble sodium silicate was collected from Morgan Company for Chemical Industries, Cairo, Egypt. Soluble Sodium hydroxide was collected from New Star Company for Chemical Industries, Cairo, Egypt. The Chemical properties (Weight %) and physical properties are shown in Table 6.

3.3. Laboratory work

The first step in the mixing process is mould preparation. The mould used was 70.6 x 70.6 x 70.6 mm, as shown in Figure 6 (a), and was coated with a releasing agent to prevent alkaline activator slag (AAS) from sticking to the mould inner surface. The activator was weighted by getting 100 gm as the total content of the soluble Na(OH) with the soluble sodium silicate by using different weight ratios between both, namely 1:0, 1:1, 1:2, and 1:3 as shown in Figure 6 (b).

The alkali activator solution was allowed to cool, and the solution temperature dropped to room temperature. A manual batching process of ingredients was carried out to make the mortar mix. The dry

ingredients, which included sand, slag, and DK or MK, were thoroughly mixed together. The alkali activator was added to the dry ingredients as seen in Figure 6 (c and d) and mixed thoroughly. The additive water was weighted at either 25, 35 or 50 gm and was added to the mix. An electric mortar mixer seen in Figure 6 (e), was used to mix all the ingredients for 3 minutes. The mortar was poured in the oiled steel moulds on three layers. The compaction was carried out using manual compaction rod to compact each layer as shown in Figure 6 (f). The moulds were covered by plastic sheet for 24 hours and then demoulded. The specimens were exposed to two curing regimes namely; ambient and wet as shown in Figure 6 (g and h) respectively.

Table 1 AAS mortar mixture design

Binder	Fine Aggregate	Alkaline Activator [100 gm]		DK	MK	Additive Water	Curing Regime	
		Na(OH) Solution Concentration (52%)	GW					
Slag	Standard Sand							
gm	gm			gm	gm	Gm		
200				0	0			
180				20				
160		1	0	40	40			
140	550	1	1	60	60	25	Ambient and wet	
130		1	2		70	35		
120		1	3		80	50		
100					100	100		
80					120			

Table 2 XRF Results for Chemical composition of Slag, DK, and MK by Weight [%]

Item	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃	LOI
Slag	43.01	0.44	15.69	0.18	0.34	6.21	32.52	<0.01	0.83	<0.01	<0.01	0.48
DK	76.78	3.59	7.91	0.95	0.02	0.37	1.16	<0.01	<0.01	<0.01	1.96	6.96
MK	62.02	2.98	29.97	2.00	0.03	0.35	1.15	<0.01	<0.01	0.02	0.25	0.90



a) Slag



b) De-Aluminated Metakaolin (DK)



c) Metakaolin (MK)

Fig. 2: Materials used

Table 3 Physical properties of Slag, DK and MK

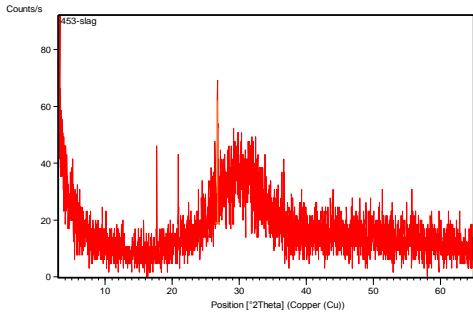
Property	Slag	DK	MK
Specific gravity	2.80	2.1	2.50
Bulk Density (t / m ³)	1.15	-	-
Specific surface (cm ² /gm)	4088	42000	12000
150µm(% retained)	4.00	-	-
Insoluble residue (%)	1.40	-	-
Loss of ignition (%)	0.50	-	-

Table 4 Pattern list of DK

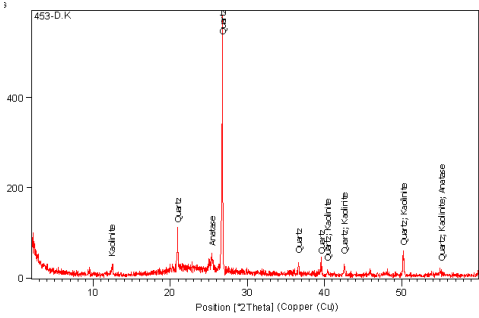
Ref. Code	Mineral Name	Chemical Formula
96-500-0036	Quartz	SiO ₂
96-900-9231	Kaolinite	Al ₂ Si ₂ H ₄ O ₉
96-900-8214	Anatase	TiO ₂

Table 5 Pattern list of MK

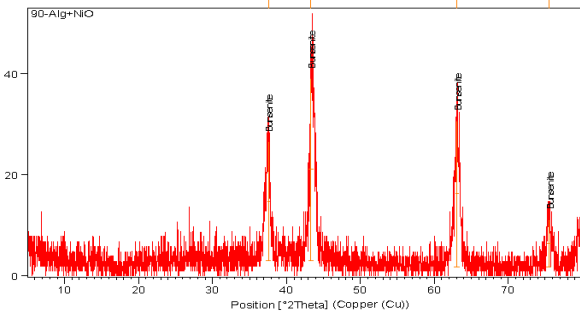
Ref. Code	Mineral Name	Chemical Formula
00-033-1161	Quartz, syn	SiO ₂
96-101-1046	Kaolinite 2M	Al ₈ Si ₈ H ₁₆ O ₃₆
96-500-0224	Anatase	TiO ₂



(a) Slag



(b) DK

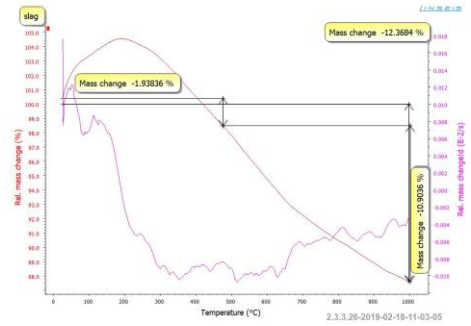


(c) MK

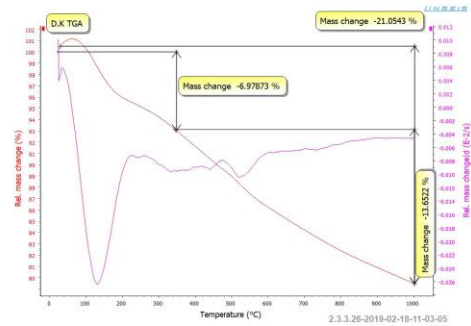
Fig. 3: XRD tests results

Table 6 Chemical properties (Weight %) and physical properties of Glass Water and Na(OH)

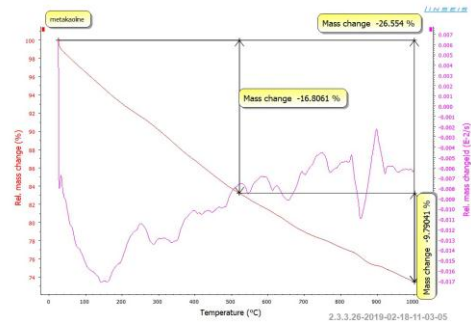
Item	Glass Water	Na(OH)
SiO	34.35	
Na ₂ O	16.37	
Ratio of Na ₂ O: SiO ₂	1:2.09	
Acids (Na(OH))		48.
Total solid	50.72	48
Water content	49.28	52
Specific gravity	1.5	1.6
Density g/cm ³	2.4	1.52
Viscosity(cps)	912	78
Appearance	Liquid gel	Liquid gel
Molecular weight	184.04	40
Boiling point	102 C	135 C



a) Slag

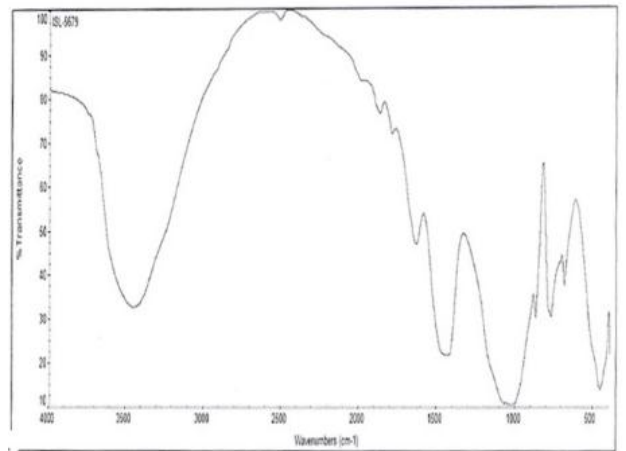


b) DK

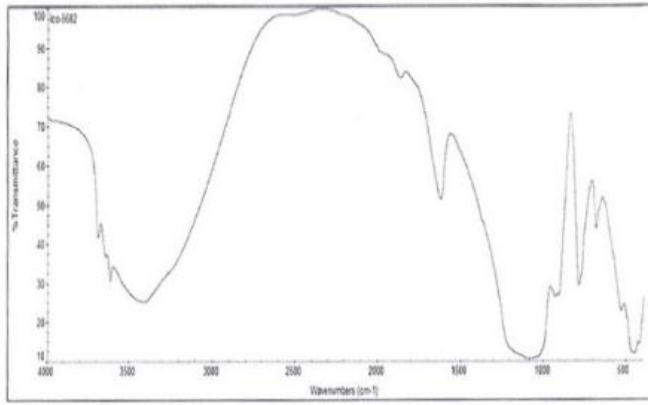


c) MK

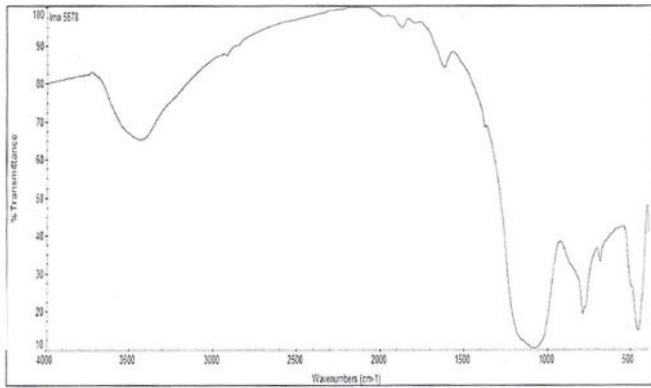
Fig. 4: Thermal Analysis tests results



a) Slag



b) DK



c) MK

Fig. 5: FTIR spectroscopic analysis tests results



(a)



(b)



(c)



(d)



(e)



(f)



(h)



(g)

Fig. 6: Experimental work

4. Results and Discussion

4.1. Flow Rate of Fresh Mortars:

Figure 7 represents the flow rate of DK modified AAS mortar for four ratios of Na(OH):GW were examined namely; 1:0, 1:1, 1:2, 1:3 respectively. Figures 7 (a), 7 (b), 7 (c), and 7 (d) represent flow rate of fresh mortar versus DK content where vertical axes represent the flow rate while horizontal axes represent the DK content for four ratios of Na(OH): GW namely; 1:0, 1:1, 1:2, 1:3 respectively.

Figure 8 represents the flow rate of fresh mortar after partial replacement of AAS mortar with MK for the optimum alkaline activator ratio between Na(OH) concentration of 52% and sodium silicate ratio of 1:2 at additional water content of 35 gm of water versus MK content from 40 to 100 gm of water, where vertical axes represent the flow rate and horizontal axes represent the MK content for the alkaline activator ratio of 1:2.. It is worth mentioning that the additional water content of 35gm and the ratio of 1:2 between Na(OH): GW had led to the optimum compressive of MK modifies AAS mortars. Table 7 represents flow rate limits and its classification according to Egyptian code of practice [22].

Table 7. Flow rate reference [22]

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

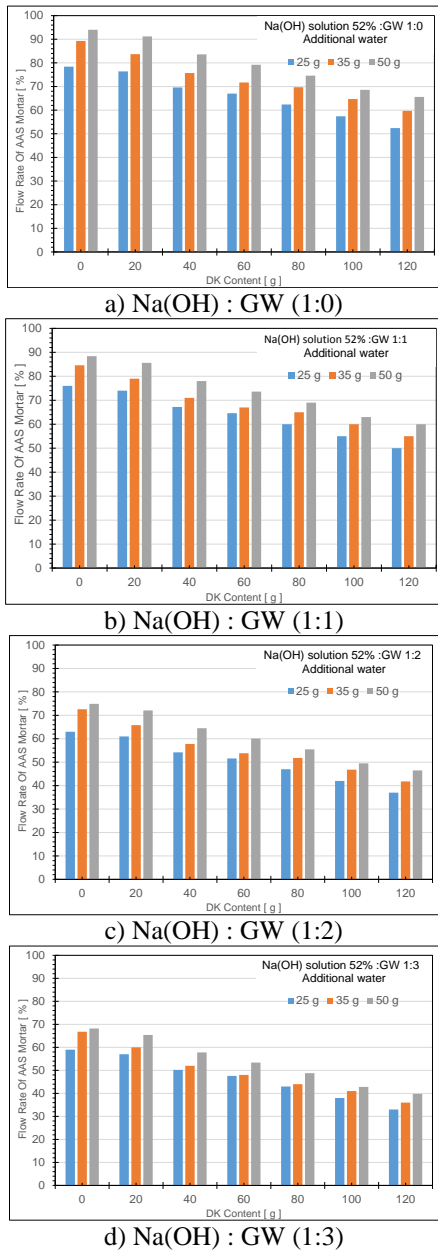


Fig. 7: Flow rate for DK modified AAS mortars

Results lead to the conclusion that increasing the DK content leads to an adverse effect by decreasing the flow rate, whatever the ratio of Na(OH): GW used or any additional water content used. In addition, increasing the MK content leads to an adverse effect by decreasing the flow rate, whatever the MK content used. This may be attributed to the increase in the surface area of fine particles in both DK and MK, which reduces the flow rate and increases the cohesion of the mortar.

Also, for DK modified mortars, as the ratio of GW increases in the alkaline activator solution that leads to an adverse effect by decreasing the flow rate, whatever the DK content used or the additional water content used. This may be attributed to the increase in the amount of solids and the reduction in the amount of water in the Na(OH) solution, which reduce the flow rate and increase the cohesion of the mortar. Furthermore, regardless of the Na(OH):GW ratio or DK content used. Increasing the content of the additive water leads to an increase in flow rate. The consistency of DK and MK modified mortars is between stiff and plastic, as shown in Figures 9 (a, and b).

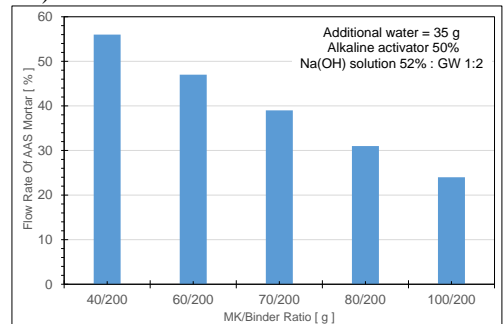


Fig. 8: Flow rate of MK modified AAS mortar



a) DK AAS mortar b) MK ASS mortar
Fig. 9: Flow Rate shapes

4.2 Initial setting time of Fresh Mortars:

Figure 10 represents the initial setting time of DK modified AAS mortar for four Na(OH):GW ratios studied, namely 1:0, 1:1, 1:2, and 1:3. Figures 10 (a), 10 (b), 10 (c), and 10 (d) show the initial setting time of fresh mortar versus DK content, with vertical axes representing the initial setting time and horizontal axes representing the DK content for four Na(OH):GW ratios, namely 1:0, 1:1, 1:2, and 1:3.

Figure 11 represents the initial setting time of fresh mortar after partial replacement of AAS mortar with MK. That was done for the optimum

alkaline activator ratio between Na(OH) concentration of 52% and sodium silicate ratio of 1:2 at additional water content of 35 gm of water versus MK content from 40 to 100 gm, where vertical axes represent the initial setting time and horizontal axes represent the MK content for alkaline activator 1:2. It is worth noting that the additional water content of 35 g and the 1:2 ratio of Na(OH) to GW have resulted in the best compressive strength of MK mortars modified for AAS.

Results lead to the conclusion that increasing the DK content leads to an adverse effect by decreasing the initial setting time, whatever the ratio of Na(OH):GW used or any additional water content used. In addition, increasing the MK content leads to an adverse effect by decreasing the initial setting time, whatever the MK content used.

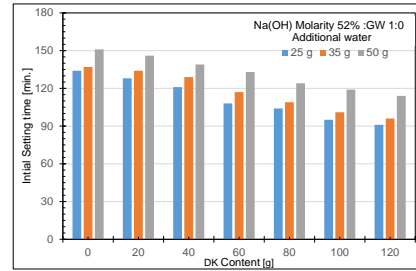
This may be attributed to the increase in the surface area of fine particles in DK and MK, which reduces the free water and increases the cohesion of the mortar.

Also, for DK-modified mortars, as the ratio of GW increases in the alkaline activator solution, that leads to an adverse effect by decreasing the initial setting time, whatever the DK content used or the additional water content used. This may be attributed to the increase in the amount of solids and the reduction in the amount of water in the Na(OH) solution, which reduce the initial setting time and increase the cohesion of the mortar.

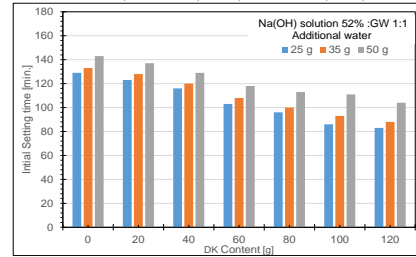
In addition, as the content of the additive water increases, that leads to an increase in the initial setting time, whatever the ratio of Na(OH):GW or DK content used. . Table 8 shows the initial setting time limits and classification according to the Egyptian code of practise [22], whereas the initial setting time of DK and MK modified mortars tends to be between the Egyptian code of practice's minimum requirements of N32.5 and R32.5 grades and N42.5 and R42.5 grades

Table 8 Initial setting time reference [22]

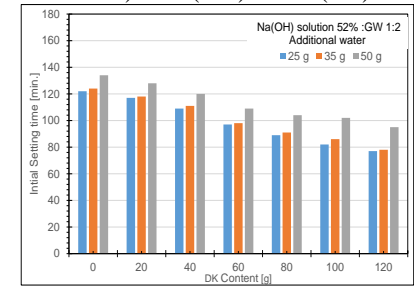
Cement grade	Initial setting time
N32.5 & R 32.5	≥ 75 minutes
N42.5 & R 42.5	≥ 60 minutes
N52.5 & R52.5	≥ 45 minutes



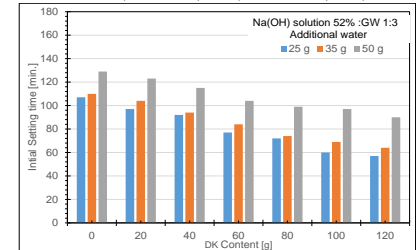
a) Na(OH) : GW (1:0)



b) Na(OH) : GW (1:1)



c) Na(OH) : GW (1:2)



d) Na(OH) : GW (1:3)

Fig. 10: Initial setting time for DKmodified AAS mortars

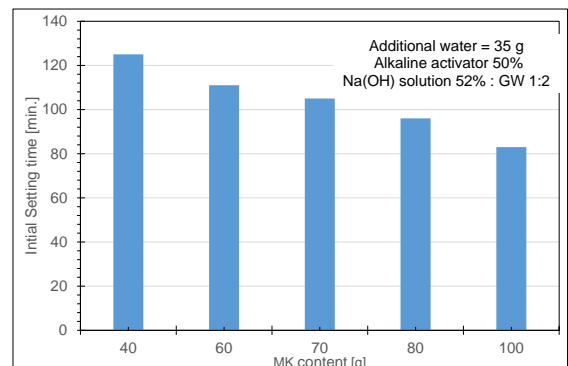


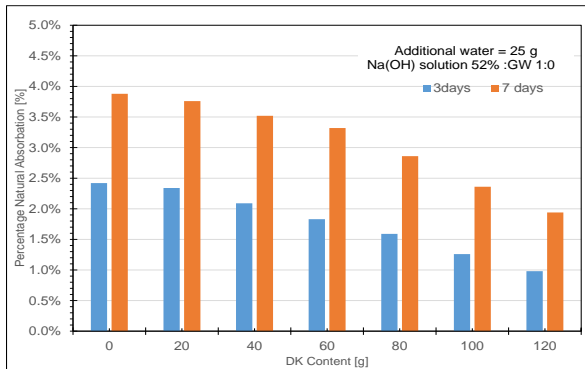
Fig. 11: Initial setting time at Additional water 35 g

4.3 Percentage of natural absorption:

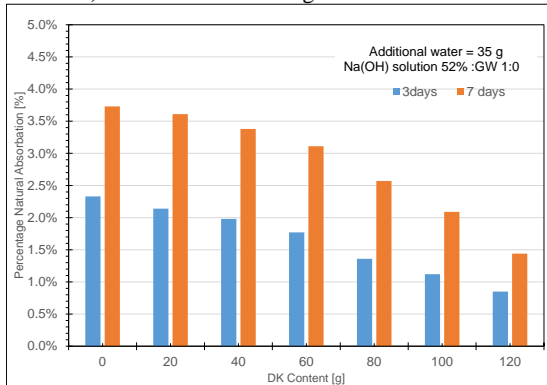
Figures 12, 13, 14, and 15 show the percentage of natural absorption of hardened mortar versus DK content ranging from 0 to 120 gm, with vertical axes representing the percentage of natural water absorption and horizontal axes representing the DK content for the four Na(OH):GW ratios studied, namely 1:0, 1:1, 1:2, and 1:3. for varying amounts of extra water ranging from 25 to 50 gm.

Figure 16 represents the percentage of natural absorption of hardened mortar from partial

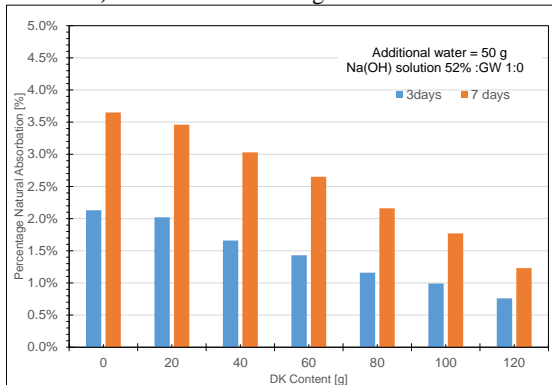
replacement of AAS mortar with MK for the optimum alkaline activator ratio between Na(OH) concentration (52%), sodium silicate (1:2), and additional water content of 35 gm versus MK content from 40 to 100 gm, where the vertical axes represent the initial setting time and the horizontal axes represent the MK content for alkaline activator 1:2. It is worth noting that the additional water content of 35 gm and the 1:2 ratio of Na(OH) to GW have resulted in the best compressive strength of MK mortars modified for AAS..



a) Additional water 25 g

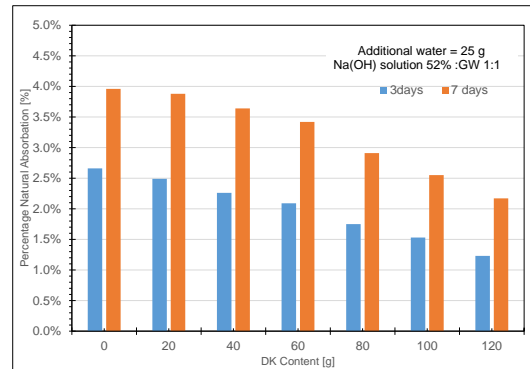


b) Additional water 35 g

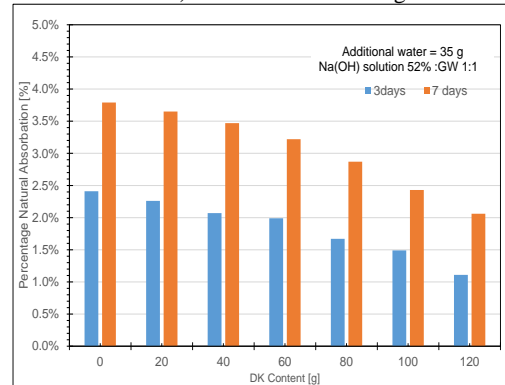


c) Additional water 50

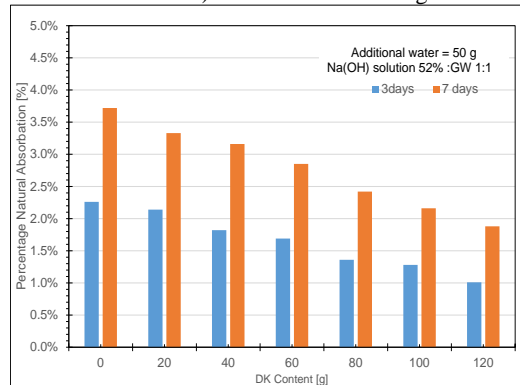
Fig. 12: Percentage of natural water absorption of Na(OH):GW (1:0) Na(OH):GW (1:1)



a) Additional water 25 g

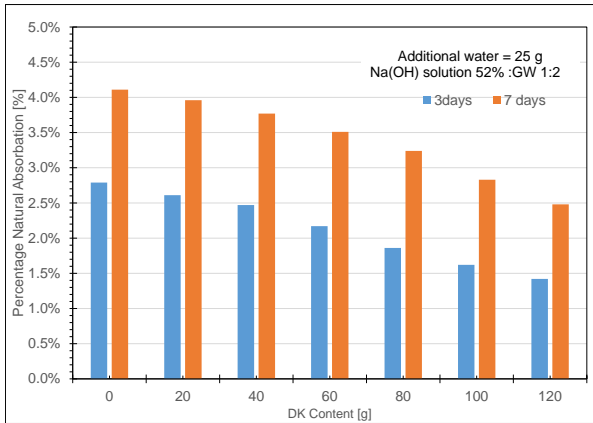


b) Additional water 35 g

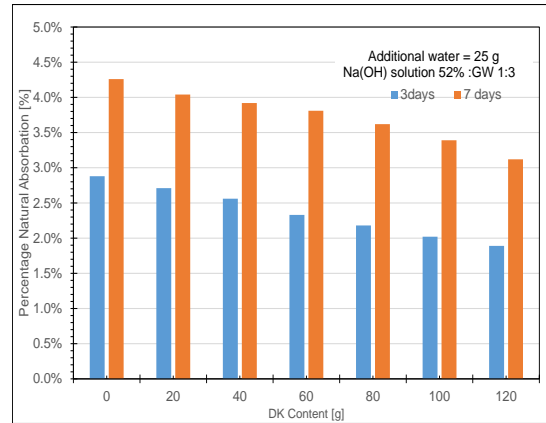


c) Additional water 50

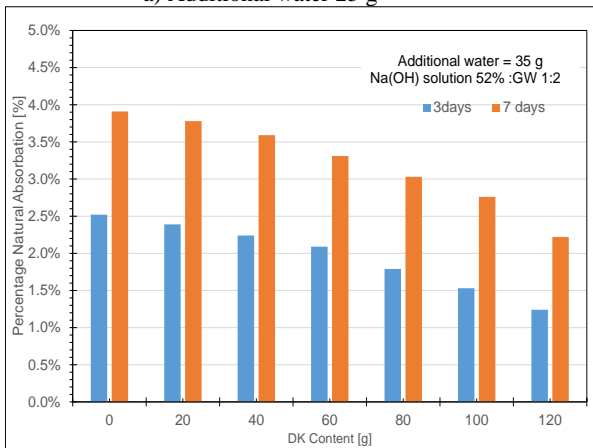
Fig. 13: Percentage of natural water absorption of



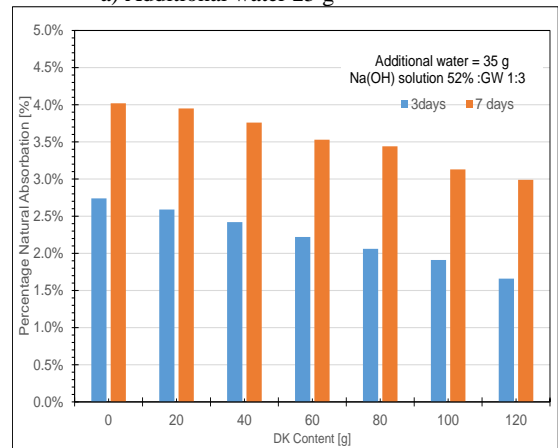
a) Additional water 25 g



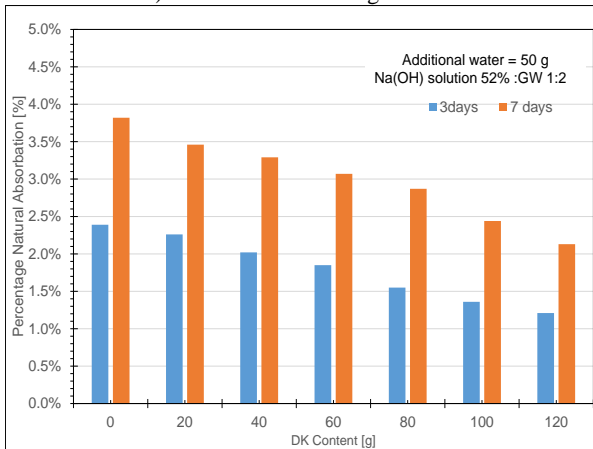
a) Additional water 25 g



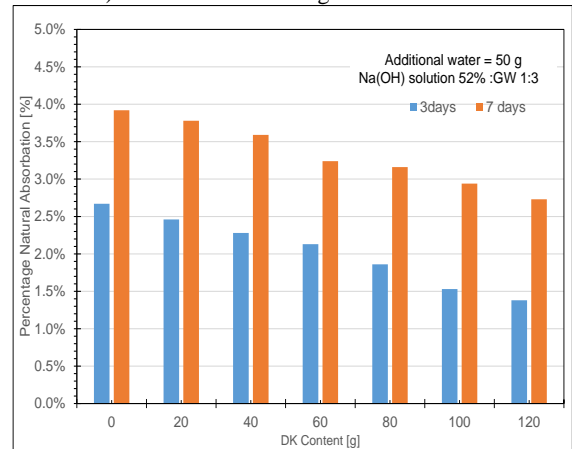
b) Additional water 35 g



b) Additional water 35 g



c) Additional water 50



c) Additional water 50

Fig. 14: Percentage of natural water absorption of Na(OH):GW (1:2) Na(OH):GW (1:3)

Fig. 15: Percentage of natural water absorption of Na(OH):GW (1:2) Na(OH):GW (1:3)

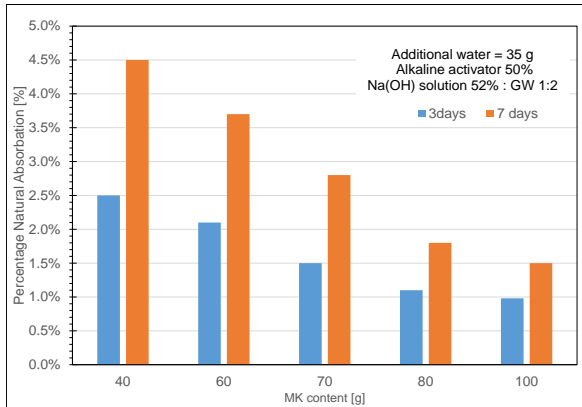


Fig. 16: Natural absorption at additional water 35g

Results leads to a conclusion that increasing the DK and MK content leads to an adverse effect by decreasing percentage of natural water absorption whatever ratio of Na(OH): GW alkaline activator solution used or any additional water content used. This may be attributed to the increase in the surface area of fine particles in DK and MK that reduces the free water and increases cohesion of the mortar.

Also, for DK modified mortars, as the content of the additive water increases that leads to a decrease in the percentage of natural water absorption whatever ratio of Na(OH): GW alkaline activator solution used or the DK content used. This may be attributed to the increase in the water-to-binder ratio resulting in most of the internal voids in the AAS mortar are saturated with water.

Moreover, also, as the ratio of GW increases in the alkaline activator solution, that leads to an increase in the percentage of natural water absorption, whatever the DK content used or the additional water content used. This could be explained by an increase in the amount of solids and a decrease in the amount of water in the Na(OH) solution alkaline activator. This may have increased the water absorption due to the decrease in the water-to-binder ratio and the partial saturation of mortar voids.

4.4 Ultimate compressive strength:

Figures 17, 18, 19, and 20 represent ambient and wet-cured compressive strengths of hardened mortar for 3 and 7 days versus DK content for additional water contents of 25, 35, and 50 gm, respectively, where vertical axes represent compressive strengths and horizontal axes represent DK content. Table 10 represents the coefficients of variation for ultimate compressive strength calculated for DK modified AAS mixtures. The calculation of standard deviations are carried out for each mixture and are plotted on the

ultimate compressive strength figures

Figures 21 (a), (b), and (c) represent the 7-day ambient cured compressive strength of hardened mortar versus MK content for additional water contents of 25, 35, and 50 gm, respectively, where the vertical axes represent compressive strength and the horizontal axes represent the MK content. Table 11 represents the coefficients of variation for ultimate compressive strength calculated for MK modified AAS mixtures. The calculation of standard deviations are carried out for each mixture and are plotted on the ultimate compressive strength figures

The experimental results lead to the conclusion that as the DK content increases, it leads to an increase in the compressive strength until a DK content of 80 gm represents 40% of the slag content, whatever the ratio of Na(OH) to GW alkaline activator solution or the amount of additive water used. Increasing the DK content beyond that would lead to a reduction in the compressive strength. In addition, increasing the MK content leads to an increase in the compressive strength until a MK content of 60 gm represents 30% of the slag content, whatever the ratio between Na(OH) and GW used or the amount of additive water used.. Increasing the MK content beyond that lead to a reduction in the compressive strength. This may be attributed to the optimum content for DK and MK. Also, for DK modified mortars, increasing the GW ratio in the activator lead to decrease in the compressive strength.

The highest compressive strengths at all ages were recorded at ratio of Na(OH):GW alkaline activator solution 1:0 regardless of the DK content or the amount of the additive water. On the other hand, for MK modified mortars Increasing the ratio of GW in the alkaline activator lead to increase in the compressive strength till the ratio between Na(OH) solution and GW reach to 1:2 then the compressive strength begin to dropped. The highest compressive strengths were recorded at the ratio between Na(OH) solution and GW 1:2 regardless of the MK content or the amount of the additive water. That refers to the optimum ratio of alkaline activation of the binder for DK and MK.

The effect of the amount of additive water on DK and MK modified mortars was typical for different ratios between Na(OH) solution and GW; the increase in additive water content to 35 gm led to an increase in the compressive strength, and beyond that content, the compressive strength reduced when the additive water increased.. This may suggest the optimum additional water-to-binder content was between 12.5% and 17.5% of the slag content.

The curing regime at ambient temperature increases the compressive strength over the wet curing regime, which had an adverse effect on gaining the compressive strength in samples regardless of the DK content or ratio of Na(OH) in the GW alkaline activator solution or the amount of additive water used.

In addition, for MK modified mortars, the curing regime at ambient temperature increases the compressive strength over the wet curing regime that had an adverse effect on gaining the compressive strength in samples, whatever the MK content. This could be attributed to the development of alkali activation at room temperature for DK and MK modified mortars without the need for wet curing. Table 9 represents the compressive strength limits for

different cement grades according to the Egyptian code of practise [22]. The compressive strengths of

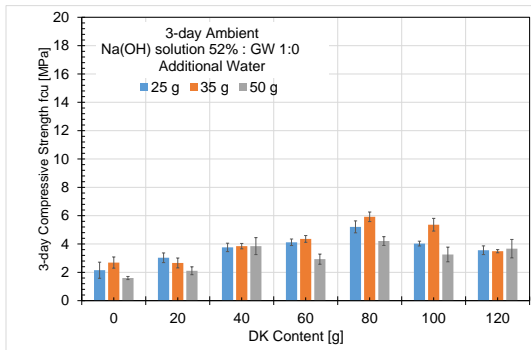
DK and MK modified mortars tend to be between the minimum requirements of N32.5, R 32.5, and N42.5 grades according to the Egyptian code of practise and the mode of failure as shown in Figure 22.

Table 9 Compressive strength reference [22]

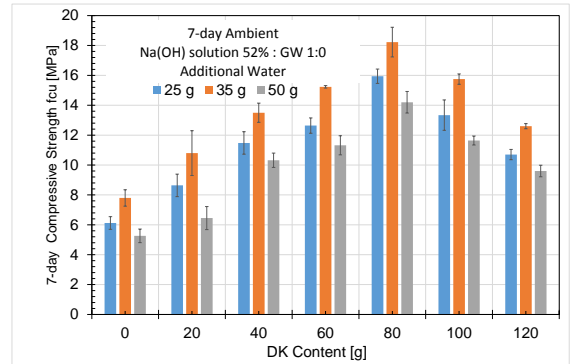
Cement grade	Early compressive strength (MPa)	
	3 days	7 days
N32.5	-	≥ 16
R32.5	≥ 10	-
N42.5	≥ 10	-
R42.5	≥ 20	-
N52.5	≥ 20	-
R52.5	≥ 30	-

Table 10 Coefficients of variation for ultimate compressive strength of DK mortar mixtures

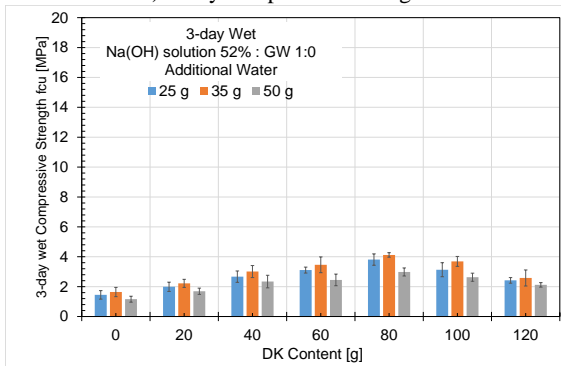
Na(OH):GW	Age	Curing Regime	Additional water			Curing Regime	Additional water		
			25 gm	35 gm	50 gm		25 gm	35gm	50 gm
1:0	3-day	Ambient	11.55	8.37	14.05	Wet	12.69	13.33	12.87
	7-day		5.72	5.03	5.92		6.17	6.81	6.89
1:1	3-day		11.42	12.76	14.98		14.61	13.18	14.93
	7-day		7.45	5.55	6.20		5.04	6.78	5.60
1:2	3-day		14.90	15.03	14.20		12.95	14.89	14.41
	7-day		5.50	3.43	5.80		7.70	8.37	14.38
1:3	3-day		14.60	14.65	11.50		13.96	14.54	14.23
	7-day		14.83	11.20	13.38		11.72	9.15	14.44



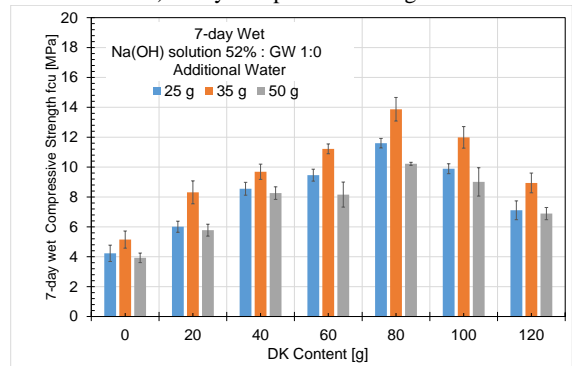
a) 3-day compressive strength



c) 7-day compressive strength

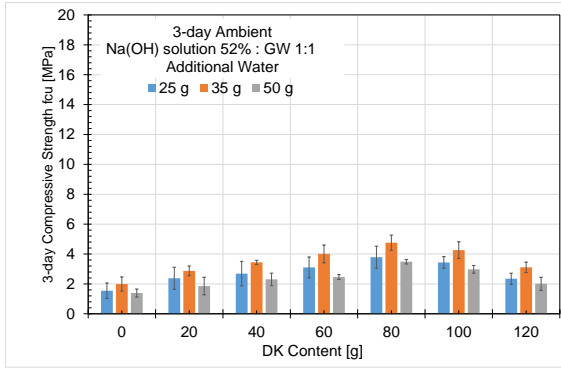


b) 3-day wet compressive strength

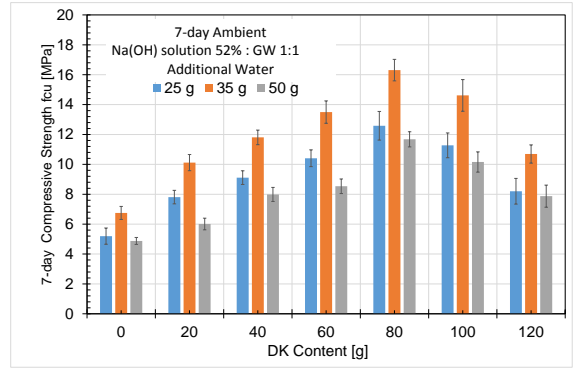


d) 7-day wet compressive strength

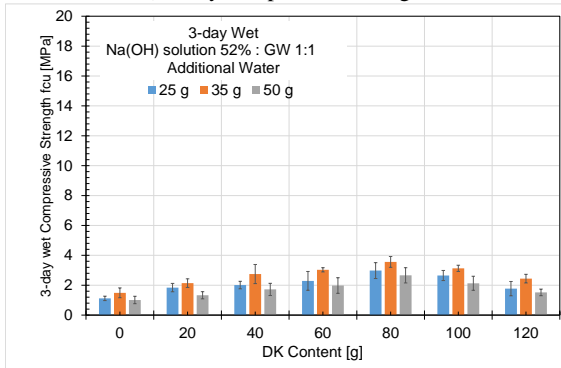
Fig.17: Compressive strength of alkali activated slag mortar MPa of Na(OH) :GW (1:0)



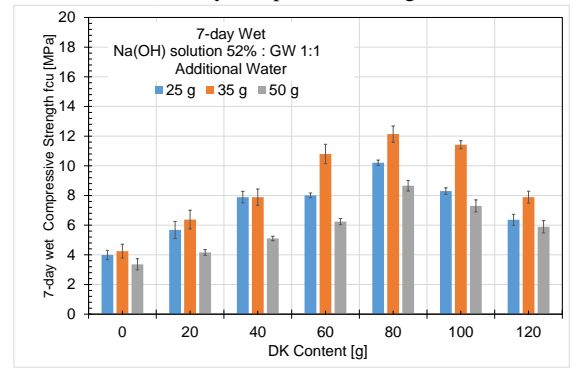
a) 3-day compressive strength



c) 7-day compressive strength

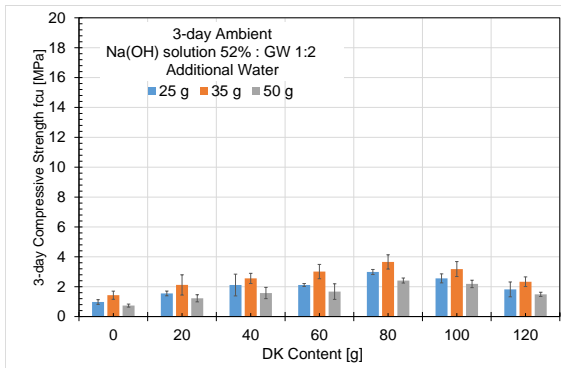


b) 3-day wet compressive strength

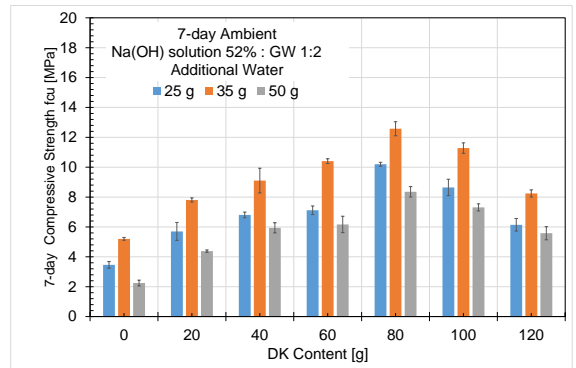


d) 7-day wet compressive strength

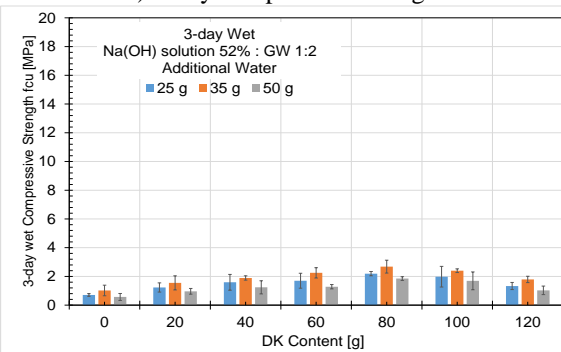
Fig.18: Compressive strength of alkali activated slag mortar MPa of Na(OH) :GW (1:1)



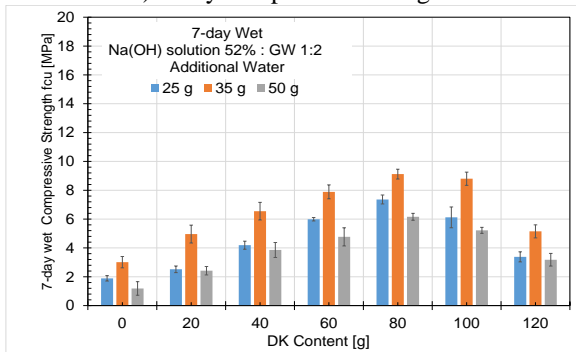
a) 3-day compressive strength



c) 7-day compressive strength



b) 3-day wet compressive strength



d) 7-day wet compressive strength

Fig. 19: Compressive strength of alkali activated slag mortar MPa of Na(OH) :GW (1:2)

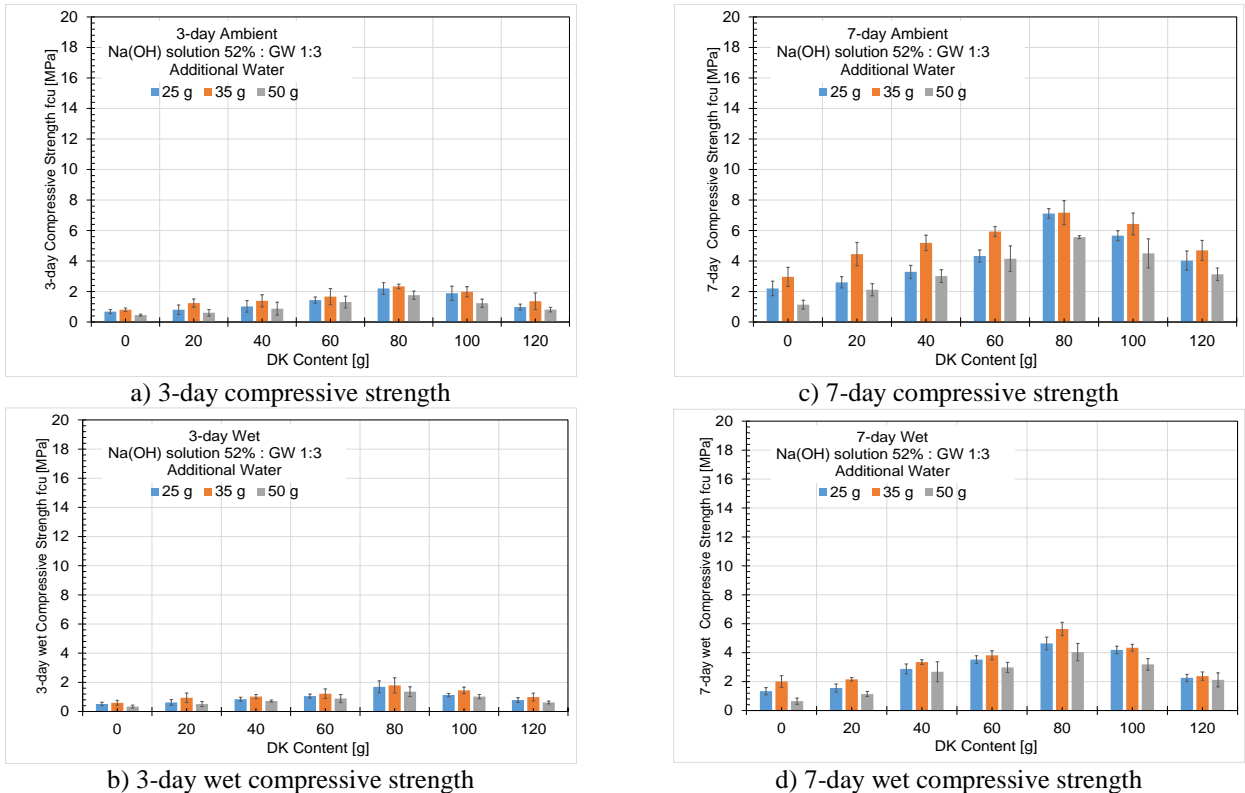


Fig. 20: Compressive strength of alkali activated slag mortar MPa of Na(OH) :GW (1:3)

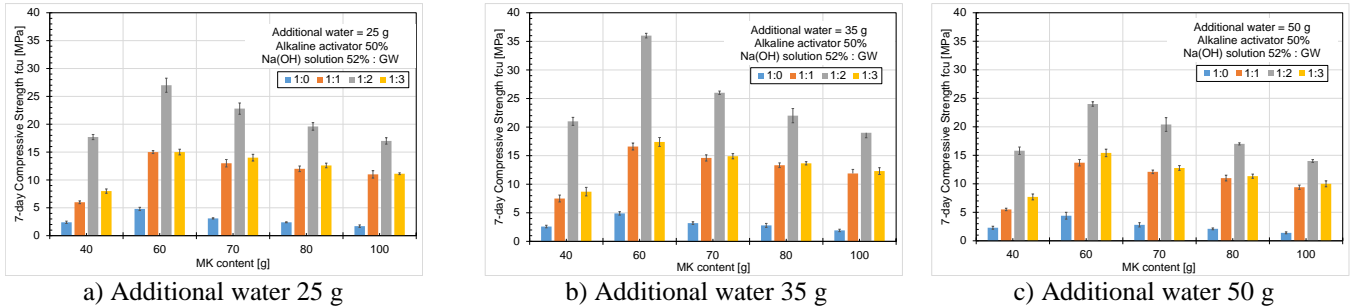


Fig. 21: 7-day compressive strength of modified MK mortar (MPa)

Table 11 Coefficients of variation for ultimate compressive strength of MK mortar mixtures

Na(OH):GW	Age	Curing Regime	Additional water		
			25 gm	35 gm	50 gm
1:0	7-day	Ambient	6.53	9.74	11.57
			3.84	4.85	3.78
			3.73	3.17	2.93
			3.45	4.67	4.65
1:1	7-day	Ambient	6.53	9.74	11.57
			3.84	4.85	3.78
			3.73	3.17	2.93
			3.45	4.67	4.65
1:2	7-day	Ambient	6.53	9.74	11.57
			3.84	4.85	3.78
			3.73	3.17	2.93
			3.45	4.67	4.65
1:3	7-day	Ambient	6.53	9.74	11.57
			3.84	4.85	3.78
			3.73	3.17	2.93
			3.45	4.67	4.65



Fig. 22: Failure mode of compressive strength test

5. Evidence of alkaline activation:

The results of the Energy dispersive X-ray (EDX) analysis of the study are given in Figure 23 and table 12. Using qualitative and quantitative EDX, the chemical composition of modified DK mortar is investigated. The physical and mechanical properties of alkaline activator mortar were a function of successful $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratios. Qualitative EDX analysis confirms the presence of major elements Si, Al, Na, and O, which are the main components that promote alkaline activation synthesis and the formation of geopolymer mortar. The scanning electron microscopy (SEM) images and EDX analyses for the studied mixtures are shown in Figures 24 (a, b, c, and d).

The micrograph for the optimum blend shows the formation of a microporous matrix with the distribution of both CSH and geopolymer within the matrix Figures 24 (a, and b); adding more DK up to 25% results in the formation of an intensified homogenised geopolymer structure with growth of the amorphous (N-A-S-H) gel and the binding gels (C-A-S-H), which has an advanced impact on the micro-structural characteristics forming a. Adding more DK results in an increased dissimilarity with large spaces between its components, reducing the effectiveness of the reactions in Figure 24 (c, and d) as interpreted from the FTIR and DTG.

The cementitious products bind slag and DK particles, then fill up the pore spaces and form a dense matrix. These images show the morphology of particles in the alkaline activation process. It was noticed that the optimum sample showed a satisfactory compressive strength of 18.4 MPa due to the suitability of the ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3$, which is sufficient to form the cementitious activation product with the added Na(OH). This composite achieved activation structures with a high compressive strength

of 18.4 MPa.

The SEM images verify the presence of strong alkaline activation in which the ratios of Si/Al and Na /Al noticed from EDX analysis are matched to that of the initial precursor mixtures.

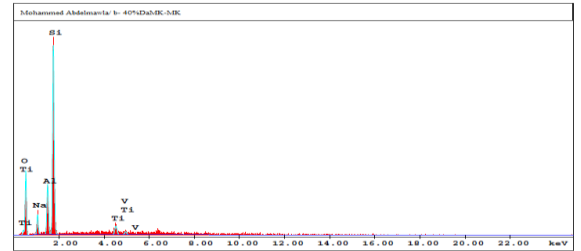


Fig. 23: EDX spectra microscope for the optimum mix

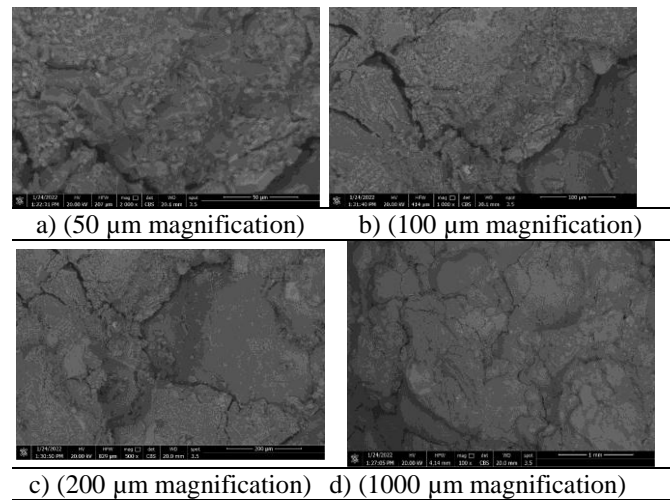


Fig. 24: SEM image of optimum sample

Table 12 EDX materials amount %

Element	O	Na	Al	Si	Ti	V
Amount (%)	50	4.7	7.3	35	2.2	0.46

6. Conclusion:

1-The optimum DK mortar mixture was obtained at Na(OH): GW ratio of 1:0 and with an alkali content of 100 gm representing 50% of the binder content, a DK content of 80 gm representing a 40% partial replacement of the slag content, and an additive water content of 35 gm that represents 17.5% of the slag content

2- The optimum MK mortar mixture was obtained at a Na(OH):GW ratio of 1:2, with an alkali content of 100 gm representing 50% of the binder content, a MK content of 60 gm representing a 30% partial replacement of the slag content, and an additive water

content of 35 gm representing 17.5% of the slag content.

3- Increasing the DK and MK content has the same effect on the consistency of the mortar mixtures. The maximum flow rate always occurs at the lowest DK and MK contents, while the minimum flow rate always occurs at the highest DK and MK contents, whatever the ratio between Na(OH) and GW or the additive water content used.

4- Increasing the DK and MK content has the same effect on the initial setting time of the mortar mixtures. The maximum initial setting time always occurs at the lowest DK and MK contents, while the minimum initial setting time always occurs at the highest DK and MK contents, whatever the ratio between Na(OH) and GW or the additive water content used.

5- Increasing the DK and MK content has the same effect on the percentage of natural water absorption of the mortar mixtures. The maximum percentage of natural water absorption always occurs at the lowest DK and MK contents, while the minimum percentage of natural water absorption at the highest DK and MK contents, whatever the ratio between Na(OH) and GW or the additive water content used.

6- Increasing the DK content leads to an increase in the compressive strength until a DK content of 80 gm, and beyond that, a reduction in the compressive strength is noticed. This may set the optimum partial replacement of DK content at 40% of the slag content.

7- Increasing the MK content leads to an increase in the compressive strength until a MK content reaches 60 gm, and beyond that, a reduction in the compressive strength is noticed. This may set the optimum partial replacement of MK content at 30% of the slag content. This finding coincides with the percentage reported in the literature.

8- The results of the experimental work show that DK behaves similarly to MK in most of the AAS mortar physical and hardened properties. Furthermore, the evidence of alkaline activation opens the door to conducting additional studies on DK modified mortar under various tests to evaluate the effect of the partial replacement of DK material on the durability of AAS mortar.

References:

- [1] Hassan S.K., "Production and Properties of Alkali Activated Slag Modified Using DK and MK", M.Sc. thesis, Sustainable Design in Construction STDC Program, Post Graduate Studies, Faculty of Engineering, The British University in Egypt, 167 pp., 2023.
- [2] Hassan B. "Provenance of sedimentary kaolin deposits in Egypt: Evidences from the Pb, Sr and Nd isotopes", Journal of African Earth Sciences, volume 100, August 2014.
- [3] Marco V., Matteo S., Mehdi C., and Seyed G., "A comparison between alkali-activated materials and Portland cement-based composites incorporating recycled tire rubber", Journal of Cleaner Production, Volume 333, 20 January 2022.
- [4] Luz M.M., Silvio D., and Marsiol G., "A study of a hybrid binder based on alkali-activated ceramic tile wastes and Portland cement", Sustainable and Nonconventional Construction Materials using Inorganic Bonded Fiber Composites, 2017, Pages 291-311..
- [5] Abdel Ghany M.T., El Sayed H.A., and Abdel Moied S., "Geopolymer synthesis by the alkali-activation of blast furnace steel slag and its fire-resistance", HBRC Journal, Volume 14, Issue 2, August 2018 Pages 159-164.
- [6] Beatryz C. M. , Leonardo G. P. , Carlos M. F. V. ,Markssuel M., Afonso R.G. A., and Jose M. F., "Application of eco-friendly alternative activators in alkali-activated materials", Journal of Building Engineering, Volume 35, March 2021.
- [7] Jordi P., Francisco A., Julia R., Maria M. M., and Maria V. B., "Applicaton of alkali-activated industrial waste", Woodhead Publishing Series in Civil and Structural Engineering, 2019 ,Pages 357-424.
- [8] Flora F., Mariano A. Z., and Cristoforo D., "Design guidelines for structural and non-structural applications", Woodhead Publishing Series in Civil and Structural Engineering, 2022, Pages 509-527,.
- [9] Apriany S., Takumu A., Yuko O., and Kenji K., "Effect of silica fume on efflorescence formation and alkali leaching of alkali-activated slag", Journal of Cleaner Production, Volume 315, 15 September 2021.
- [10] Shamsad A., Ashraf A. B., Abbas A. S. ,Hammad R. K., Ali H.A., and Mohammed M., "Effects of key factors on the compressive strength of metakaolin and limestone powder-based alkali-activated concrete mixtures", Case Studies in Construction Materials, Volume 16, June 2022.
- [11] Alaa M. R., "Alkali-activated metakaolin: A short guide for civil Engineer - An overview. Construction and Building Materials", Construction and Building Materials, Volume 41, April 2013, Pages 751-765.
- [12] Wei Z., Xiao Y., Tao Y., and Zuhua Z. "The degradation mechanisms of alkali-activated fly ash/slag blend cements exposed to sulphuric acid ", Construction Build. Materials, volume 186, August 2018, pages 1177-1187.
- [13] Lei G., Terry B., and phillip V., "Sulfuric acid exposure of conventional concrete and alkali-activated concrete: Assessment of test methodologies", Construction And Building. Materials, volume 197, January 2019, pages 681-692
- [14] Ayoub D., Farhad A., and Neda G. P., "Effects of initial SiO₂/Al₂O₃ molar ratio and slag on fly ash-based ambient cured geopolymer properties.", Construction and Building Materials Volume 293, July 2021.
- [15] Bayrak A., Tareq A., and Qais H., "Effect of discontinuous curing and ambient temperature on the compressive strength development of fly ash based Geopolymer concrete". MATEC Web of Conferences, volume 162, 2018.
- [16] Paulo H.R. B., Nemkumar B. ,Himad A. A., and Wander L. V., "Performance of blended metakaolin/blast furnace slag alkali-activated mortars", Cement and Concrete Composites, Volume 71, August 2016, Pages 42-52
- [17] Mostafa N.Y., El-Hemaly S.A.S, Al-Wakeel E.I., EL-Korashy S.A., and Brown P.W., "Characterization and evaluation of the pozzolanic activity of Egyptian industrial by-products: I: Silica fume and dealuminated kaolin", Cement and Concrete Research. , volume (3), March 2001, pages 467-474.

- [18] ASTM C1437-20, "Standard Test Method for Hydraulic Cement Mortar", ASTM International, West Conshohocken, PA, 2020.
- [19] ASTM C807-21, "Standard Test Method for Time of Setting of Hydraulic Cement Mortar by Modified Vicat Needle", ASTM International, West Conshohocken, PA, 2021.
- [20] ASTM C109-20, "Standard Test Method for Compressive Strength of Hydraulic Cement Mortar (Using 2-in or [50-mm] Cube Specimens) ", ASTM International, West Conshohocken, PA, 2020.
- [21] ASTM C128-15, "Standard Test Method for Compressive Strength for Relative Density (Specific Gravity) and Absorption of Fine Aggregate", ASTM International, West Conshohocken, PA, 2015.
- [22] Egyptian Code of Practice 203, "Basics of Design and regulations of Construction of Reinforced Concrete Structures: Appendix III, Guide for Testing of Concrete Materials", Egyptian Ministry of Housing, Egypt, 2018.