Use of waste dealuminated metakaolin in alkali-activated cement mixes

Mostafa Elsebaei [1,2], Maria Mavroulidou [2]*, Amany Micheal[1], Maria Centeno [2], Rabee Shamass [3], Ottavia Rispoli [2]

[1] The British University in Egypt, El Sherouk City , Suez Desert Road , Cairo 11837, Egypt

[2] School of Engineering and Design, London South Bank University, 103 Borough Road, SE1 0AA, UK

[3] College of Engineering, Design and Physical Sciences, Brunel University of London, Kingston Lane, Middlesex UB8 3PH, UK

*corresponding author: Maria Mavroulidou
e-mail: mavroum@lsbu.ac.uk

Abstract This paper explores the feasibility of using dealuminated metakaolin (DMK), a hazardous by-product of the alum industry, in alkali-activated cement (AAC) mixes, as an outlet for this voluminous hazardous waste, alternative to landfilling. After reviewing global DMK waste management practices and emerging uses as a pozzolanic material, the study experimentally investigates geopolymer mortars made with 75% Ground Granulated Blastfurnace Slag (GGBS) and 25% DMK. Activated with various alkaline solutions across different silicate modulus values (Ms 0.2–1.2), the mortars were tested for compressive strength (CS). Some mixes achieved compressive strengths over 40 MPa after just 7 days of curing, demonstrating the potential of DMK in structural concrete applications. Ongoing research aims to elucidate the interaction between DMK and GGBS, towards further optimization of the AAC systems to enhance mechanical performance, durability, workability, and setting behaviour.

Keywords: hazardous waste management; aluminium sulphate industry; dealuminated metakaolin (DMK), Ground Granulated Blastfurnace Slag (GGBS); alkali-activated cements (AAC)

1. Introduction

This paper addresses the solid waste management of dealuminated metakaolin (DMK), by studying the feasibility of using DMK in alkali-activated cement (AAC) mixes. DMK, a hazardous by-product of the aluminium sulphate (alum) industry, is a very fine material of and high-silica content formed during acid leaching of metakaolin to produce alum. DMK is classified as hazardous due to its fine particle nature, which contributes to air pollution, and its low pH, which increases environmental risks when disposed in landfills (Farouk et al., 2023; Hassan et al., 2023). Alum is used in a wide range of applications including water treatment, paper manufacturing, and cloth dyes amongst other. Due to the wide use of alum for different applications, very large amounts of DMK arise annually worldwide. These are typically disposed of in landfills or, in some countries, are dumped in remote locations. Thus, if feasible, the production of good quality AAC with DMK would have the dual advantage of providing an outlet for this hazardous waste, while giving alternatives to Portland cement, whose production is linked to negative environmental impacts (high energy consumption and CO_2 emissions, and high consumption of non-renewable natural raw materials). AAC can provide an eco-friendly alternative option to Portland cement, as they can incorporate waste materials in the alkali-activated material system and have the potential of lower environmental impact in terms of raw materials used for their production.

DMK has been tested for pozzolanic effect and several studies demonstrated that partial replacement of Portland cement by up to 15% DMK shows around 20% higher compressive strength. Conversely, very few studies, performed only recently, have investigated DMK in AAC systems. These include Abdullah & Abdullah (2023), who produced geopolymer cement systems in combination with metakaolin (MK) and showed that the DMK-MK geopolymers could have an enhanced performance if appropriate ratios of DMK:MK are used. To the Authors' knowledge, only two recent studies (Al-kroom et al., 2024; Hassan et al., 2023) combined DMK with Ground Granulated Blastfurnace Slag (GGBS), a by-product of the pig iron industry, which is very widely used for high calcium alkali-activated cement systems. Both studies showed a high reduction in compressive strength when DMK was introduced in the AAC system.

From the above, it is clear that, considering the wide variety of alkaline activators and AAC system components, more research is required to conclude on the feasibility of DMK in AAC mixes. In line with this, this paper reports results of an experimental study focusing on the use of DMK and GGBS precursor mixes in AAC mortars, as part of a research project, focusing on the production of good quality AAC concrete incorporating DMK in the system. In this paper, the focus of the study is the effect of silicate modulus values (Ms) on the compressive strength of DMK-GGBS AAC mortars.

2. Materials and Methods

The GGBS used in this investigation had a specific gravity of 2.8, and a specific surface area of 4088 cm^2/g. DMK used was obtained from Aluminium Sulphate Co. Egypt

(S.A.E.); it is the same DMK as that used in Farouk et al., (2023) and Hassan et al. (2023), who reported a specific gravity of 2.1 and a specific surface area of 42000 cm^2/g. The chemical composition of GGBS and DMK was determined by XRF analysis, using a Rigaku Supermini200 Spectrometer apparatus (see Table 1). The sand used in the mortar mixes was desert sand sieved using sieve meshes between 0.3 mm and 1.18 mm to match sand grading for concrete according to the Egyptian code for the design of concrete ECP 203-2020, (Research Centre for Housing and Building, 2020). Two activators were used: sodium hydroxide (NaOH) and base liquid sodium silicate (Na_2SiO_3). Five mixes were prepared, one of them acting as a control mix with 100% GGBS while the remaining four had 75% GGBS and 25% DMK. All mixes had the same water content (43%), Na_2O content (12%) and sand ratio (2.75) but different silica moduli Ms (see Table 2).

The alkaline solution was prepared first and was left to cool down for 1h. It was then added to the mix of GGBS, DMK and sand and the mortar ingredients were mixed for 3 minutes. Triplicate 70.6 mm mortar cubes were cast for each mix and cured at ambient regime for 7 days, when they were tested for compressive strength (CS).

3. Results and Discussion

For brevity, the average compressive strength and the standard deviation (SD) of the results are shown in Table 2, together with the mix design of each mix. The control mix reached 55 MPa. Upon 25% GGBS replacement by DMK (mix M1), the strength (CS) dropped to 25.2 MPa corresponding to a reduction in CS of c.a. 55% compared to the control mix. This could be attributed to the high modulus of silicate (1.2), which was presumably further increased when DMK was introduced into the mix. Similarly, mix M2 with Ms=0.8 shows a 55% reduction in CS compared to the control mix. As Ms further reduces, CS starts increasing, so that mix M3 (Ms=0.4) achieves a strength of 36.6 MPa, whereas for Ms=0.2 (mix M4) an average CS of 43 MPa is achieved. Although this corresponds to a 22% drop compared to the control mix, this 7-day strength is adequate for structural purposes. It should be noted that although better in terms of CS, mixes with lower Ms (i.e., mix M3 and M4) were setting very fast (within 10-15min) compared to mixes with higher Ms (M1, and M2). The rapid setting thus remains an issue for practical purposes and requires further investigation.

Table 2. Mix design and respective CS results

Mix	W/C	Na_2O	Ms	CS (MPa)	SD
Control	43%	12%	1.2	55.1	3.9
M1	43%	12%	1.2	25.2	2
M2	43%	12%	0.8	24.0	1.7
M3	43%	12%	0.4	36.6	5.8
M4	43%	12%	0.2	43.0	4.3

4. Conclusion

This study showed that using appropriate silica moduli, combinations of GGBS with DMK precursors in AAC can exceed 40 MPa already within 7 days of casting, which is an adequate compressive strength for building materials. This finding demonstrates the potential of DMK for structural applications, which can provide a useful outlet for this voluminous hazardous material, alternative to landfilling. Ongoing research and extensive material analysis aims to elucidate the interactions between DMK and GGBS, towards further optimisation of the AAC systems thus further enhancing the resulting concrete and mortar mechanical performance and durability, while addressing workability, and setting behaviour.

Table 1. XRF Results for GGBS and DMK used in this study

Compound	SiO_2	CaO	Al_2O_3	MgO	Na_2O	SO_3	K_2O	TiO_2	MnO	Fe_2O_3	L.O.I
GGBS	18.4	56.5	10.1	4.3	0.5	2.2	1.1	1.5	0.8	2.5	1
DMK	77.1	0.6	7.8	---	---	3.4	0.1	5.1	---	0.9	4.7

References

Abdullah, N. A., & Abdullah, H. (2023). Compressive strength, setting time, and flowability of OPC mortar mixtures modified with a composite of Nano carbon and partially de-aluminated metakaolin. *Journal of Silicate Based and Composite Materials*, *75*(3), 118–126. https://doi.org/10.14382/epitoanyag-jsbcm.2023.17

Al-kroom, H., Rashad, A. M., Alghamdi, H., Abadel, A. A., Elrahman, M. A., Abdel-Gawwad, H. A., & Arif, M. A. (2024). Application of pretreated dealuminated kaolin as a modifier agent for alkali-activated slag cement. *Case Studies in Construction Materials*, *20*(November 2023), e02858. https://doi.org/10.1016/j.cscm.2024.e02858

Farouk, K. M., Helmy, A. I., Morsy, K. M., & Abdullah, N. A. (2023). The Usage Of Di-Aluminated Meta-Kaolin As A Mineral Admixture In Concrete. *International Journal of Engineering Research & Technology*, *12*(10).

Hassan, S., Khalaf, M. A., Abdullah, N. A., & Helmy, A. I. I. (2023). Production and Properties Of Alkali Activated Slag Modified Using DK & MK. *The Egyptian International Journal of Engineering Sciences and Technology*, *44*(1), 1–17.

Research Center for Housing and Building (2020). Egyptian code for design and construction of reinforced concrete buildings (ECP-203), Giza, Egypt

Zidan, I., Khalaf, M. A., & Helmy, A. I. I. (2023). Properties of alkali-activated slag mortar and prediction of its compressive strength. *Ain Shams Engineering Journal*, *14*(11), 102536.