

Effects of sodium oxide content on the durability of alkali-activated mortar utilizing botswana copper mine tailings and fly ash

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Abstract

This investigation aims to provide experimental data on the performance of alkali-activated mortar made of copper mine tailings (CMT) and fly ash (FA) exposed to acid-sulphate attacks and elevated temperature environments as a measure of durability. FA was used as a replacement material, substituting 20 to 40% of the CMT by mass, and sodium hydroxide was the alkaline activator, which was added in terms of Na₂O content by mass of the total binder at 5%, 10%, and 15%. The durability performance of alkali-activated mortar was evaluated against 5 % and 10% concentrations of sulphuric and hydrochloric acids, and magnesium and sodium sulphates up to 180 days of exposure, as well as elevated temperature environment. The specimens were first visually examined, and weight change was measured before being exposed to an elevated temperature environment and the residual compressive strength was measured. It was observed that Na₂O content and elevated temperature environment influence the residual compressive strength of alkali-activated mortar. Increasing the Na₂O content for all CMT-FA-based mortar samples performed well in sulphates and acids mediums in terms of durability, but with a slight reduction in its durability performance in terms of residual compressive strength. However, these effects were more profound in samples exposed to acids, particularly those with lower FA replacement levels and sodium oxide content. For the high-temperature exposure, the residual compressive strength of all CMT-FA samples was much higher than the initial values. The findings also revealed that the partial replacement of CMT by FA significantly improved the residual compressive strength in terms of the durability performance of the alkali-activated mortar

Keywords: Copper mine tailings, Fly ash, Alkali activation, Acid and Sulphate resistances, Elevated temperature.

1. INTRODUCTION

Durability is a critical necessity for application and the viability of construction materials for the overall performance in aggressive environments during its design existence period. Structures made of concrete are known to be badly damaged by sulfate attacks, and high temperatures are a major hazard as well. The main reason for the expansion and degradation of concrete in hardened cement is when C₃A reacts with sulfate ions in the presence of calcium hydroxide to form ettringite and gypsum [1]. Cement production involves several steps, that is a high energy-intensive process that emits one ton of greenhouse gases for each ton of cement produced and requires the equivalent of a barrel of oil per ton [2], which has a negative influence on the environment, as well as the future of mankind [3-4].

Alkali-activated cementitious binders can provide a sustainable and environmentally friendly alternative to conventional ordinary Portland cement (OPC) [5-6], that provides a reduction in CO₂ emissions of up to 80% when

compared to OPC, which is primarily based on chemically activating industrial wastes such as granulated ground blast furnace slag (GGBFS), fly ash (FA), and metakaolin as precursor materials reach in aluminosilicate [7-8], to produce a nanoparticle base gel [9-10]. The binder's characteristics can be greatly altered by the mineralogy and particle morphology of the precursor materials adopted [11-12], it possesses better durability with high early strength and has no hazardous alkali-aggregate reaction in alkali-activated cementitious materials [13]. Alkali-activated binders with high amorphous aluminosilicate content are primarily composed of fly ash or metakaolin and those with a high slag content are composed of a calcium silicate hydrate (C-S-H) gel with Al and alkali substitution [14-15]. Alkali-activated concrete has improved mechanical qualities and greater durability in harsh settings than normal cement concrete [16-18]. Cementitious materials activated with alkali were generally stable in the presence of sulphuric and

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hydrochloric acid, with weight loss ranging from 5 to 8 percent, while Portland-based cement was destroyed, and the calcium-alumina cement lost weight by 30 to 60 percent [19]. Alkali-activated cementitious materials have been reported to be acid resistant because they rely on alumina-silicate rather than calcium silicate hydrate. Numerous studies on alkali-activated concrete have been conducted, but there is still no agreement on the effect of various parameters on the properties of alkali-activated materials. For alkali-activated concrete, the most essential parameters are the aluminosilicate supply, curing conditions, type and concentration of alkaline activator, and alkali activator to base ratio [20-22]. NaOH and Na₂SiO₃ are frequently utilized to activate when making alkali-activated concrete [23]. Several investigations have shown that alkaline-activated mortar properties are influenced by the Na₂SiO₃ concentration rather than NaOH and its molarity [24]. These properties influenced may be attributed to the use of more sodium silicate, which led to the production of more silica gel and contributed significantly to the high strength. An increase in Na₂SiO₃/NaOH content, according to [25], leads to better performance of cementitious materials. Studies have proven that Na₂SiO₃ promotes the reaction process, the reaction product resulting in more Si and higher mechanical strength [26]. When it comes to innovative cementitious materials, the strength and durability of novel blend regimes are still not fully understood due to the variation in experimental data from prior studies [27-29]. Much research has been conducted on alkali-activated mortar, however, there is limited research to quantify the double effect of sodium oxide (Na₂O) content and elevated temperature environment on the durability properties of alkali-activated mortar. The research aims to provide experimental data on the effect of NaOH in terms of Na₂O content by mass of the total binder on the

durability performance of alkali-activated mortar paste containing waste materials such as Botswana copper mine tailing and fly ash. The durability performances of alkali-activated mortar were measured in terms of sodium and magnesium sulfate resistance, sulphuric and hydrochloric acid resistance, and elevated temperature environment. The evaluations were done through visual observation, change in weight after exposure, and residual compressive strength in an elevated temperature environment.

2. Materials and experimental procedures

2.1 Materials

Copper mine tailings (CMT) from BCL- Selebi Phikwe copper mine and ASTM Class F [30] low-calcium fly ash from Morupule coal power station, Botswana were used as the base materials for making alkali-activated mortar pastes. Figure 1 shows the grading curve for the base materials (CMT and FA). The particle size analysis was conducted using a Malvern mastersizer which presents the curved volume density against phi units. Table 1 shows the chemical compositions of CMT and FA. To activate the base materials, commercial grade sodium hydroxide (NaOH) flakes with 98% purity were purchased in bulk from a local supplier and used as an alkaline activator. The sodium hydroxide was measured in terms of its sodium oxide content in the following series (5 %, 10% and 15% Na₂O content) by mass of the total binder. To avoid the impact of unknown contaminants, the sodium hydroxide pellets were firstly dissolved in distilled water to the required molarity and then stored at room temperature of 23 ± 2°C with a relative humidity of 65% for 18 hours before use.

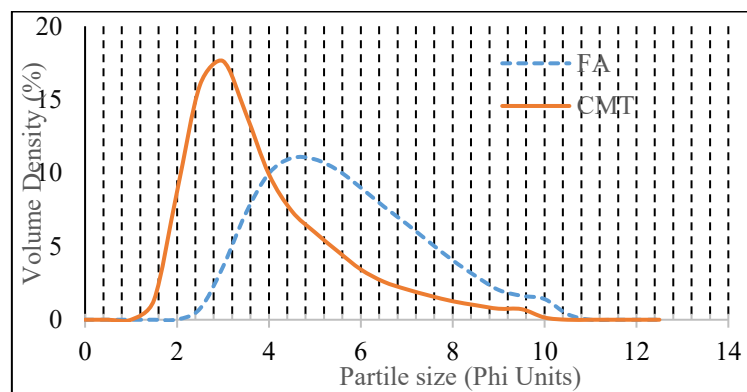


Figure 1: Particle size distribution for CMT and FA

Table1: Chemical composition of copper mine tailings and fly ash

Material	Oxides								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	SO ₃	MnO
CMT (%)	48.48	28.67	4.33	4.06	7.25	1.9	3.26		2.05
FA (%)	31.72	28.69	6.83	1.17	20.91	1.59	1.32	7.55	0.22

2.2 Methods and preparation of alkali activated mortar paste specimens

All mixtures have a weight ratio of 1.5, 2.33, and 4 for the base materials. A sodium hydroxide solution to the base material ratio of 1.19, 1.14, and 1.06 was used to make alkali-activated mortar pastes. The extra water demand of mortar mixtures was calculated using a standard flow table and a flow diameter of 110 ± 5 mm, as specified by ASTM C230 [31]. A Hobart mixer was used to dry mix the base materials for roughly 10 minutes before being used. The alkali-activated mortar pastes were then reworked with the addition of an alkaline solution. For another 10 minutes, the based materials and alkali solution components of the combination were combined. This was followed by the normal processes of casting using a cube mold of $50 \times 50 \times 50$ and compacting cement mortar. The specimens were covered with plastic sheets to prevent moisture loss and cured for 12 hours in the molds at room temperature. All mortar specimens were placed in an oven at the temperature of 80 ± 2 °C, demolded after 36 hours, and continued curing in an oven for 28 days.

2.2.1 Acid resistance

Three specimens of each mortar mixture were submerged in each of the 5% and 10% sulphuric acid (H_2SO_4), and 5% and 10% hydrochloric acid (HCl) for a period of 20 weeks following the ASTM C-267 [32] standard. Acid resistance was measured on mortar samples in terms of the weight gained and weight loss. The solution was changed every week to maintain its concentration. Relative weight loss and weight gain were estimated as a percentage of the initial weight of specimens to determine its weight loss and weight gained.

2.2.2 Sulphate resistance

Alkali-activated mortar pastes were immersed for 20 weeks in a plastic tank containing 5% and 10% of each sodium and magnesium sulphates solution [32]. These solutions were made with tap water and renewed every

week. Change in weight of specimens was assessed after selected periods of exposure up to 20 weeks. Specimens were removed from the sulphate solutions and cleaned before the measurement of their weight loss and weight gain. Average values were calculated from the measurements of three replicates.

2.2.3 Residual compressive strength at elevated temperature

The residual compressive strength was measured on the specimens according to ASTM C109 [33]. Three specimens of each mixture were placed in an oven at an elevated temperature after the exposure to acids and sulphates attacks. The specimens were tested after exposure to an elevated temperature to determine the average residual compressive strength.

3. Results and Discussions

3.1 Visual appearance

The visual appearances of the alkali-activated mortar paste specimens after varied exposures (water, acids, and sulphates), are illustrated in Figure 2. The mix proportions of alkali-activated mortar pastes are presented in Table 2. Specimens exposed to water for 20 weeks indicated no difference in appearance from what was observed before exposure. Following a 20-week soak in sulphuric acid solution, the alkali-activated mortar paste specimens are shown in Figure 2a-b. The surface of the test specimens subjected to 5% and 10% sulphuric acid was seen to withstand the attack. As illustrated in Figure 2d-e, the specimens subjected to hydrochloric acid solution resembled those in H_2SO_4 solution, with a rough surface and apparent void. After 20 weeks of contact with sodium and magnesium sulphate solutions, there was no substantial damage to the surface of mortar pastes specimens. The surface however got softened and rough as the examination proceeded. In a similar work, Bhutta et al. [34] discovered no appreciable damage to the surface of test specimens after one year of exposure to a 5 % sodium sulphate solution.

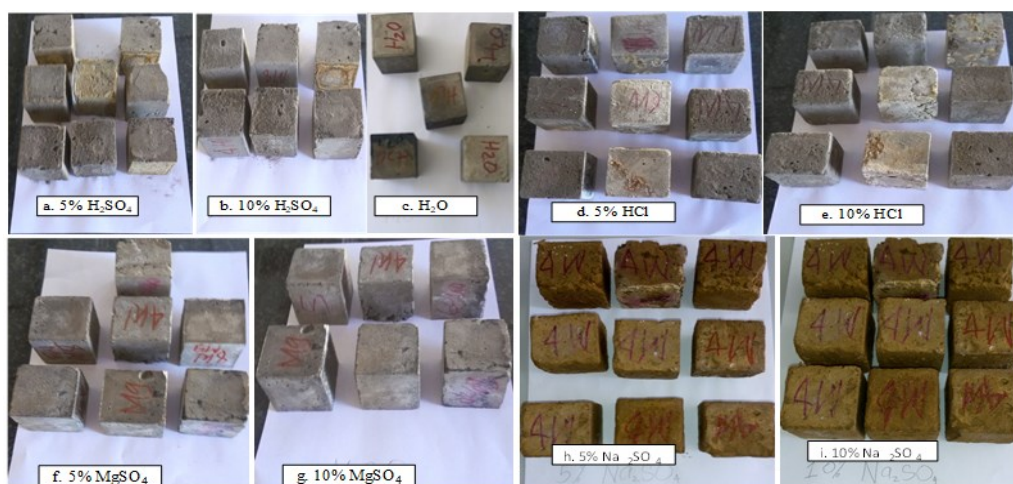


Figure 2: Visual appearance of alkali-activated mortar paste specimens after 20 weeks exposure in: a) 5% H_2SO_4 , b) 10% H_2SO_4 , c) H_2O , d) 5% HCl, e) 10% HCl, f) 5% $MgSO_4$, g) 10% $MgSO_4$, h) 5% Na_2SO_4 and I) 10% Na_2SO_4

Table 2: Mix Proportion

Mix ID	CMT		FA		W/B ratio	Water Added g	NaOH g	Na ₂ O content	
	%	g	%	g				%	g
CMT	100	968.75	0		0.2	757.5	250	5	193.75
CMT80-FA20	80	775	20	193.75					
CMT70-FA30	70	678.13	30	290.63					
CMT60-FA40	60	581.25	40	387.5					
CMT	100	968.75	0		0.2	740	500	10	387.5
CMT80-FA20	80	775	20	193.75					
CMT70-FA30	70	678.13	30	290.63					
CMT60-FA40	60	581.25	40	387.5					
CMT	100	968.75	0		0.2	722.5	750	15	581.25
CMT80-FA20	80	775	20	193.75					
CMT70-FA30	70	678.13	30	290.63					
CMT60-FA40	60	581.25	40	387.5					

3.2 Change in weight

The weight changes of CMT-FA alkali-activated mortar specimens in sulphate solution are presented in Figures 3 and 4. The weight of all specimens gradually increased within the exposure period for up to 20 weeks in sulphate solution due to absorption of the exposed liquid. The rapid increase in weight occurred for all specimens up to 4 weeks of exposure. A maximum increase in weight was observed in CMT60-FA40 specimens and the least gained in weight occurred in CMT specimens. After 20 weeks, CMT60-FA40 specimens in 10 % Na₂SO₄ solution gradually gained weight of 7.53%, 6.73% and 6.34% for the Na₂O content 5 %, 10 % and 15 % (see Figures 3). CMT specimens in 10 % Na₂SO₄ solution gained the weight of 3.97%, 3.56% and 2.94% for same Na₂O content. CMT60-FA40 mortar specimens in the 10 % MgSO₄ solution gained weight of 6.68 %, 6.41 % and 6.09 % for Na₂O content of 5 %, 10 % and 15 % (see Figure 4). The results showed that CMT60-FA40-based alkali-activated mortar paste specimens had gained weight in the first four weeks of exposure to 6.43 % in the 10 % sodium

sulphate solution while 5.86 % in the 10 % magnesium sulphate solution for 5 % Na₂O content. The increase in weight might be due to white deposits within the surface pores, which were flaky, or needle-like during the early stages of exposure. Irrespective of the solution used, an increase in the Na₂O content, decreased the weight gained. A large gain in weight was observed in solutions with Na₂SO₄ than those with MgSO₄. For the same Na₂O content, varying the content of Na₂SO₄ or MgSO₄ from 5 % and 10 % did not have a noticeable effect on the weight gained. Weight change is the most important criterion to investigate the effect of specimens after being exposed to the acid solution. Mortar specimens immersed in 5 % acid solution, gradually gained weight. The gradual increase in weight occurred for all specimens up to 20 weeks of exposure. CMT60-FA40 specimens in 5 % HCl and H₂SO₄ solutions gradually gained the weight of 6.68 %, 5.41 % and 4.98 % and to 6.12 %, 5.45 % and 5.23 % while CMT mortar specimens gained weight to 3.94 %, 3.72 % and 3.25 % and to 3.75 %, 3.35 % and 2.75 % after 20 weeks of exposure for the Na₂O content 5 %, 10 % and 15 % respectively.

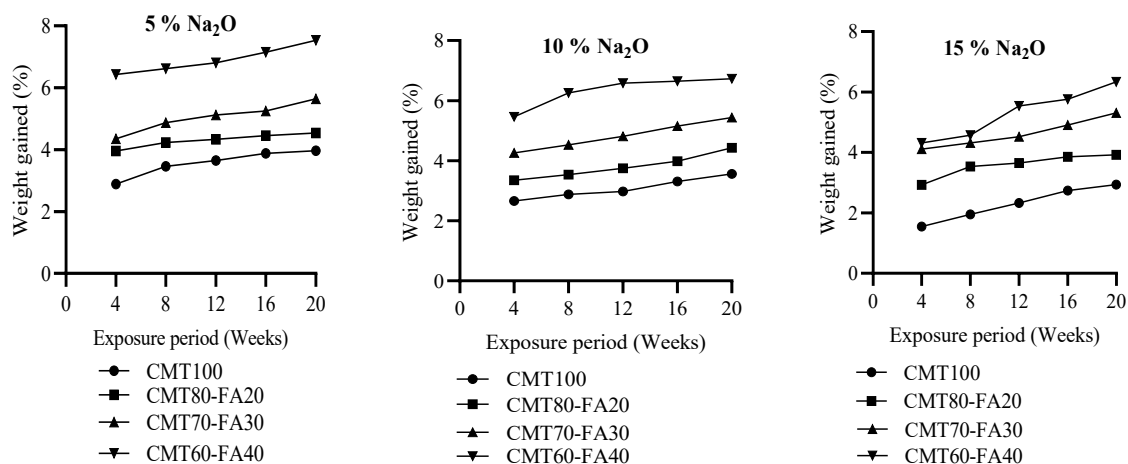


Figure 3: Effect of Na₂O content on exposure period on weight gained of alkali activated mortar pastes exposed to 10 % Na₂SO₄ solution

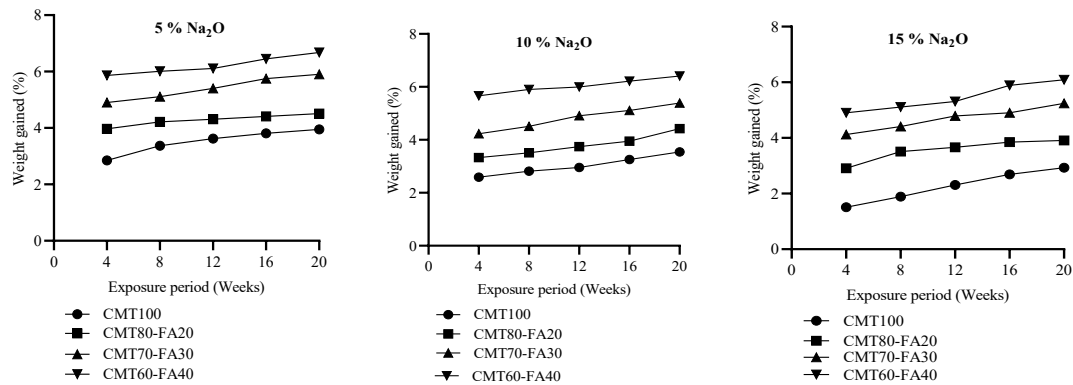


Figure 4: Effect of Na₂O content on exposure period on weight gained of alkali activated mortar pastes exposed to 10 % MgSO₄ solution

Figures 5 and 6 present the effect of Na₂O content and exposure period on the weight loss of alkali-activated mortars exposed to 10 % HCl and H₂SO₄ solutions. Sudden weight loss was observed for CMT specimens in 10 % acid solutions for up to 20 weeks. Weight loss for CMT in 10 % HCl solution was recorded as 7.75 %, 6.78 % and 5.54% (see Figure 6) and CMT mortar specimens in H₂SO₄ solution weight loss was recorded in the order of 7.53 %, 6.73 %, and 5.65 % with the same Na₂O contents (see Figure 6). CMT-FA alkali-activated mortar specimens with the highest Na₂O content had the minimum weight loss after 20 weeks. As the Na₂O content increased, weight loss also decreased correspondingly in acid solution. Alkali-activated cement was found to have

a very low weight loss of 5 – 8 % by [19]. When samples were immersed in 5 % sulphuric acid and hydrochloric acid solutions. In contrast, Portland cement was destroyed in the same environment. In this study, minimum weight losses were attained in CMT60-FA40 specimens while the maximum weight loss was attained in CMT specimens. Pacheco-Torgal and Jalali indicated an average weight loss of just 2.6 % after being attacked by (sulphuric, hydrochloric, and nitric) acids for 28 days, while Portland cement concretes weight loss is more than twice that value of alkali-activated concretes [35]. In this study, weight loss was observed in the 10 % acid solutions at a marginal weight loss.

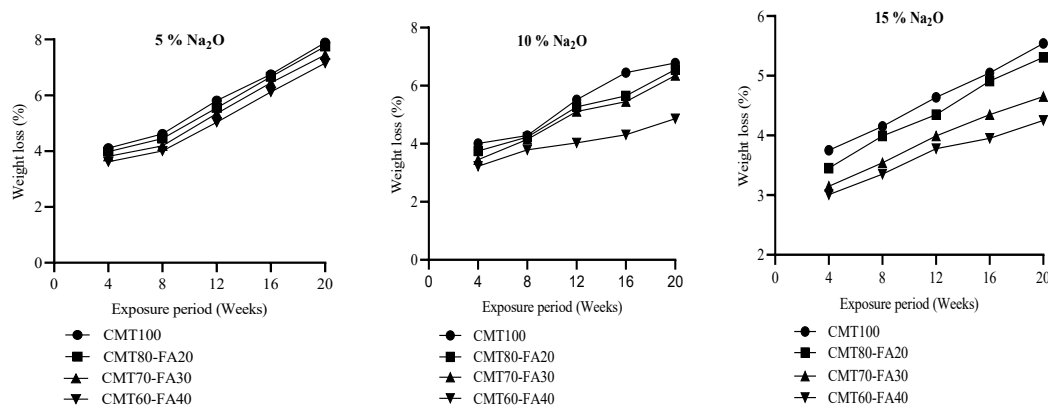


Figure 5: Effect of Na₂O content on exposure period on weight loss of alkali activated mortar pastes exposed to 10 % HCl solution

3.3 Residual compressive strength at elevated temperature

Figures 7 to 10 present the residual compressive strength of alkali-activated mortar pastes exposure to elevated temperature after sulphates and acids attacks. The trend in residual compressive strength increases for all mortar specimens to a temperature of 200 °C, then gradually decreased up to 600 °C as the CMT replacement by FA increases. The specimens with 5 % Na₂O content were observed to have the least increase in residual compressive strength while the 15 % Na₂O content was observed to have the highest increase in residual compressive strength. The CMT specimens were the least increase in residual compressive strength while CMT60-FA40 specimens had the highest residual compressive

strength. As the CMT replacement by FA and Na₂O content increases, residual compressive strength increases. Mortar paste specimens in 10 % sulphates concentration are presented in Figures 11 and 12. It is observed that all specimens in sulphates perform well in terms of residual compressive strength. Before exposure of specimens to sulphates and acids, CMT and CMT60-FA40 specimens 28 days' compressive strength were recorded as 12.9, 14.9, and 17 MPa, and 18.9, 35.5, and 54.6 MPa for 5 %, 10 %, and 15 % Na₂O content, respectively. After the exposure to sodium sulphate and elevated temperature, residual compressive strength for CMT specimens gradually increased to 16.9, 18.9, and 21.1 MPa while CMT60-FA40 specimens increase to 23.5, 39.3, and 57.5 MPa. for 5 %, 10 % and 15 % Na₂O

content, respectively. Specimens introduced to an elevated temperature treatment from magnesium sulphate solution had an increase in residual compressive strength for CMT specimens of 17.6, 19.6, and 21.8 MPa while CMT60-FA40 specimens increased to 24.2, 40, and 58.2

MPa for 5 %, 10 % and 15 % Na₂O content, respectively. Mortar pastes specimens in the MgSO₄ solution perform better in terms of residual compressive strength than the specimens in the Na₂SO₄ solution.

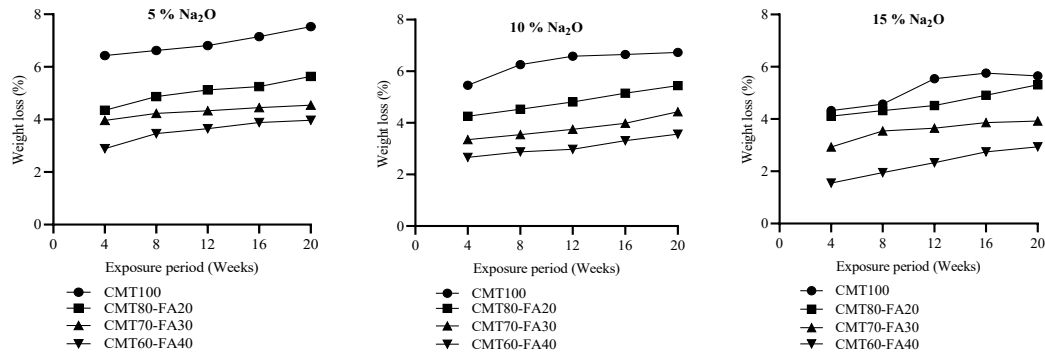


Figure 6: Effect of Na₂O content on exposure period on weight loss of alkali activated mortar pastes exposed to 10 % H₂SO₄ solution

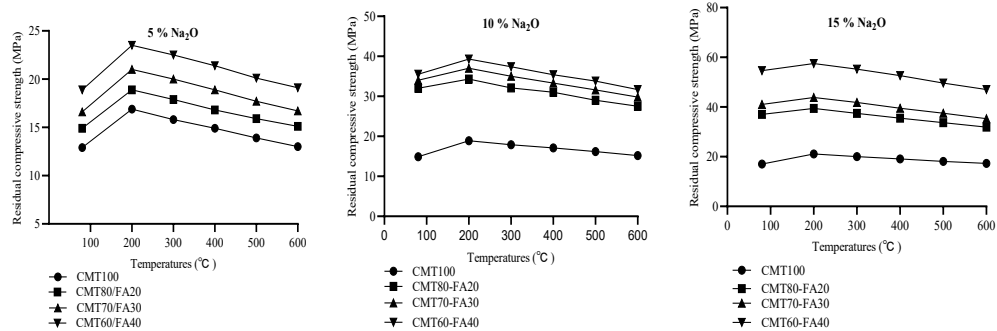


Figure 7: Effect of Na₂O content and elevated temperature on residual compressive strength of alkali-activated mortar pastes after 20 weeks of exposure to 10 % Na₂SO₄ solution

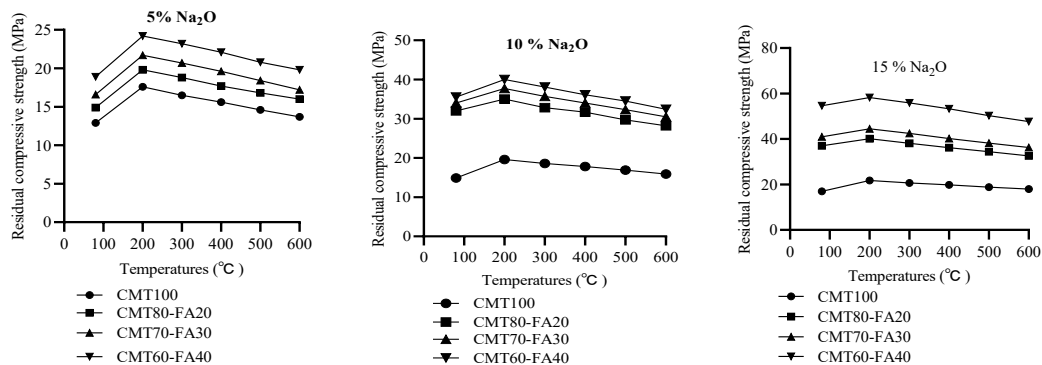


Figure 8: Effect of Na₂O content and elevated temperature on residual compressive strength of alkali-activated mortar pastes after 20 weeks of exposure to 10 % MgSO₄ solution

Alkali-activated mortar specimens in 10 % acids solutions are presented in Figures 9 and 10. After the specimen's exposure to HCl and elevated temperature treatment led to an increase in residual compressive strength to 16.3, 18.2, and 20.9 MPa for CMT specimens (see Fig. 13) while the CMT60-FA40 specimen's residual compressive strength increased to 23.9, 40 and 56.3 MPa (see Fig. 14) for 5 %, 10 % and 15 % Na₂O content respectively. Specimens introduced to an elevated temperature treatment from H₂SO₄ acid solution had an increase in residual compressive strength for CMT specimens of 16.7, 18.7, and 21 MPa while CMT60-FA40 specimen's

residual compressive strength increased to 23.3, 39.1 and 55.3 MPa for same Na₂O content. Mortar pastes specimens in the H₂SO₄ solution perform better in terms of residual compressive strength than the specimens in the HCl solution

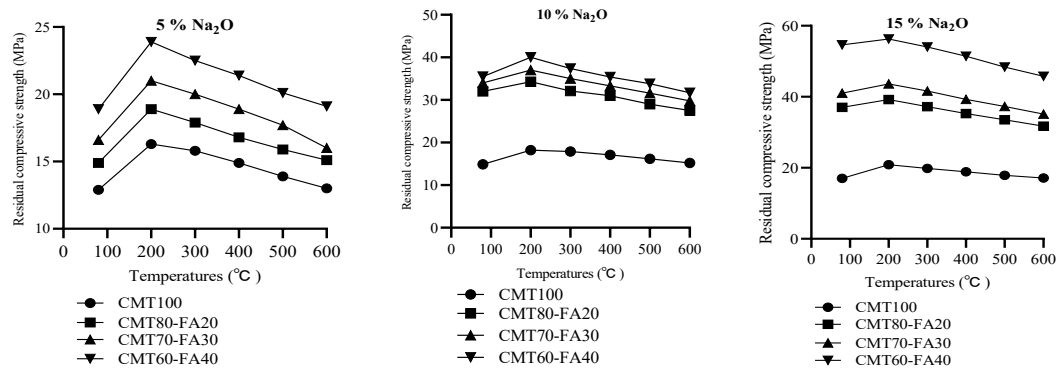


Figure 9: Effect of Na₂O content and elevated temperature on residual compressive strength of alkali-activated mortar pastes after 20 weeks of exposure to 10 % HCl solution

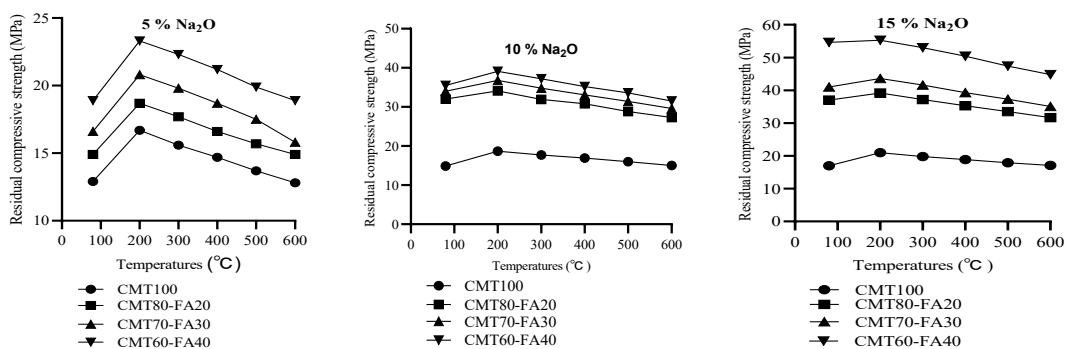


Figure 10: Effect of Na₂O content and elevated temperature on residual compressive strength of alkali-activated mortar pastes after 20 weeks of exposure to 10 % H₂SO₄ solution

4. Conclusion

Alkali-activated mortar specimens prepared with pozzolanic base materials utilising sodium hydroxide as the alkaline activator, which was added in terms of Na₂O content by mass of the total binder ranging from 5%, 10%, and 15% showed mortar pastes resisted the attack when exposed to acids and sulphates, as well as to elevated temperature environment. Based on findings during the present study, the following conclusions are drawn.

1. All mortar specimens exposed to sodium and magnesium sulphate solution for up to 20 weeks had no damage on the surface. The CMT60-FA40 specimens best performance in different sulphate and acid solutions.
2. Alkali-activated mortar specimens gained weight during exposure to sodium and magnesium sulphates solutions as well as in 5 % acid solutions. A maximum increase in weight was observed in CMT60-FA40 specimens and the least gain in weight occurred in CMT specimens. The pattern of weight gain is almost similar for three different Na₂O content.
3. The weight loss was observed to be gradually decreased with an increase in Na₂O content for all the specimens immersed in sulphates and acids. The maximum weight loss was obtained for CMT-based mortar specimens in 10 %

sulphuric and hydrochloric acid solutions after 20 weeks.

4. The inclusion of FA in the CMT-based alkali-activated mixture is found to be suitable for alkali-activated mortar under heat curing conditions for better durability performance.
5. When compared sulphate to acid immersion, acid was found to be aggressive in 10 % solution indicating higher weight loss.
6. Alkali-activated mortar pastes specimens from the MgSO₄ solution and elevated temperature environment perform better in terms of residual compressive strength than the specimens in the Na₂SO₄ solution. Mortar pastes specimens from the H₂SO₄ solution and elevated temperature perform better in terms of residual compressive strength than the specimens in the HCl solution.
7. A maximum increase in residual compressive strength for all specimens was observed at 200 °C, followed by a gradual decrease up to 600 °C. CMT60-FA40 better perform in all exposure conditions while CMT specimens were the least performing samples.

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