Resistance of Alkali-Activated Blended Volcanic Ash-MSWI-FA Mortar in Sulphuric Acid and Artificial Seawater

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Abstract

This paper investigates the resistance of alkali-activated binder from volcanic ash (VA) + Municipal Solid Waste Incinerator Fly Ash (MSWI-FA) system exposed into sulphuric acid solution and artificial seawater at 56 days. The effect of these aggressive media on the physical, structural, mechanical and microstructural properties of alkali-activated mortars was discussed. The degradation was studied using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The decrease of compressive strength after 56 days was 9.10%, 22.08%, 32.5% in artificial seawater, 2% H_2SO_4 and 4% H_2SO_4, respectively. The loss of strength in both media is primarily due to the fact that the water molecules present in these media penetrated into the pore cavities between binders and aggregates, making them weak. In an acidic medium, it is also due to the decalcification and depolymerization reactions whereas in seawater medium, it is attributed to the movement of content ions and probable formation of Friedel's salt. The results disclose that VA + MSWI-FA based alkali activated materials resist better in seawater than in sulphuric acid solution. Synergistic use of volcanic ash and MSWI-FA for construction materials through alkaline activation looks like the upcoming trend to valorize these wastes.

1 Introduction

The alkali-activated material is a class of an amorphous inorganic polymer material derived from the alkaline attack of a low or high calcium aluminosilicate powder or matrix. The product formed a structure of zeolitic and amorphous type [1, 2]. This eco-material is an alternative potential to Portland cement because its production is energy-efficient and releases little greenhouse gas [3]. The negative charge carry by alumina in its structure can be balancing by alkali cations or heavy metal cations, thus contributes to the immobilization of some toxic waste via solidification/stabilization [4–6]. A wide range

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Highlights • VA+MSWI-FA based alkali activated materials resist better in seawater than in sulphuric acid solution
• The loss of strength in acid medium is due to the decalcification and depolymerization reactions
• The calcium sulphate is the new mineral forms in acidic medium

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of raw materials is used in the synthesis of this green generation of binder this including: clay, calcined clay, laterites, fly ashes, slag, red mud, pozzolan, etc. [3, 7, 8].

The major challenge of alkaline activation technology in recent years focuses on the valorization of local materials in this field of research. In Cameroon, volcanic ash is one of the most abundant materials available for the synthesis of geopolymer cement [9, 10]. One of the limits of its use is the low reactivity in alkaline medium at room temperature [11], thus requiring activation or the addition of a supplementary cementitious material which may be activated aluminosilicates and toxic or non-toxic industrial waste [12]. Tome et al. [13] recently used MSWI-FA as a source of CaO in order to improve the reactivity of low reactive volcanic ash. The authors pointed out that 10 wt% of MSWI-FA is sufficient to generate geopolymer with moderate mechanical properties. The microstructure analysis of geopolymers also revealed the coexistence of the C-S-H and N-A-S-H. The products obtained are prospective to be used in several areas of construction where higher compressive strength is not required.

Any material once exposed in nature undergoes attacks from aggressive agents. The impact of the attack strongly depends on the structure of the material [14, 15]. Djobo et al. [16] already investigated the behaviour of volcanic ash based geopolymers in sulphuric acid solution medium. However, to the best of our knowledge, a system where C-S-H and N-A-S-H gels provided from a mixture of MSWI-FA with volcanic ash has not yet been studied for durability in acid and saline environments. The acid attack can originate from the rain, soils, animal husbandry and industrial process and inorganic salts, sulphate, chorine attack from seawater. The acid attack leads to depolymerization by the substitution of Al by H and the formation of new crystalline phases such as gypsum in some acid [17]. The corrosion, when used in sea, can be due to the reaction of these ions (Na^+, K^+, Mg^{2+}, Ca^{2+}, Cl^-, CO_3^-) with the constituents of cement or the leaching of some constituent of cement such as calcium phases. As for the alkali-activated materials and particularly the volcanic ash-based alkali-activated materials, the literature reveals that they have good resistance in aggressive media [18]. But this resistance can be influenced by the modification of the bulk composition of cement. For example, additives rich in calcium phases can cause a coexistence of the geopolymer gel (N-A-S-H) and C-S-H [19]. C-S-H is easily attackable in the acidic medium compared to geopolymer gel [20]. However, limited investigations of the C-S-H gel from alkali-activated matrix having the same behaviour with C-S-H from Portland cement have been conducted. The decrease of Ca/Si molar ratio in the case of Portland cement is more prominent than in the case of geopolymer concrete [17, 21].

This work consists of studying the resistance of alkali-activated blended volcanic ash-MSWI to corrosive medium (sulphuric acid solution and seawater). It will contribute firstly to valorize the local materials for designing eco-friendly materials. Secondly, to understanding the structural and microstructural transformation of the system where there is the co-existence of N-A-S-H and C-S-H in two media i.e. acid and saline. The effects of these aggressive media on the physical, structural, mechanical and microstructural properties of alkali-activated mortars will be discussed.

2 Materials and Methods

2.1 Materials Used VA and MSWI-FA

The volcanic ash (VA) used in this work was collected from one of Cameroon's reserves of pozzolana located in Loum, Littoral region and the MSWI-FA from the incineration plant located in the same region. These materials were already characterized by Tome et al. [6, 13]. Volcanic ash mainly contains SiO_2, Al_2O_3 and Fe_2O_3 at 75.98% and in MSWI-FA is composed of SiO_2 and CaO making 36.46%, followed by Cl and SO_3. Based on the chemical composition of MSWI-FA, this solid precursor is hazardous due to its high heavy metals content. But our work on leachability of MSWI-FA-volcanic ash based geopolymer [6] showed that the products are appropriated for the environment. The major mineral phases in volcanic ash include: Anorthite (Na ($AlSi_3O_8$), PDF#01–073-6461), Feldspar ($NaAlSi_3O_8$, PDF#01–083-6911), Nepheline ($K_{0.48}Na_{3.48}(Al_{0.99}Si_{0.01}O_4)$, PDF#01–073-6265), Maghemite (Fe_2O_3, PDF#00–001-0551) and Diopside ($Ca(Mg,Fe,Al)(Si,Al)_2O_6$, PDF#01–071-0994). The MSWI-FA is mostly composed of glassy phases and Anhydrite ($CaSO_4$, PDF#00–003-0368), Bassanite ($CaSO_4.0.5H_2O$, PDF #00–041-0224), Gypsum ($CaSO_4.2H_2O$ PDF#04–009-3817), Calcite ($CaCO_3$, PDF#00–005-0586), Quartz (SiO_2, PDF# 00–029-0085) and Halite (NaCl, PDF#00–005-0628) as the crystalline phases. The laser particle size analyzer (Mastersizer 2000, Malvern, UK) was used to measure the size distribution of particles (volume and number) of MSWI-FA powders and VA. Both materials had comparable median diameters of 11.52 μm and 11.14 μm for MSWI-FA and VA, respectively. The BET surface area of MSW-FA is 7.4 m^2/g and for volcanic ash is 3.2 m^2/g. The specific gravity and the maximum particle size of the used sand were 2.46 and 1.26 mm, respectively.

The activated solution was prepared using sodium hydroxide and sodium silicate solutions as alkaline solution. Analytical grade sodium hydroxide in flake from (98% purity) was used to prepare a 10 M NaOH solution. After 24 h, the NaOH solution was blended with commercial water glass supplied by SDFCL-India (With 28.7 wt.% SiO_2, 8.9 wt.% Na_2O and 62.4 wt.% H_2O; density 1.37 g/mL) to reach the desired composition of alkaline activator solution (silicate modulus =1.2).

Sulphuric acid solution (98 wt.%) was diluted with tap water to obtain two acid solutions with a concentration of 2 wt.% and 4 wt.%. The artificial seawater (pH = 7.5 ± 0.5) was prepared following M1942 procedure [22] by dissolving NaCl (24.56 g), KCl (0.67 g), $CaCl_2.2H_2O$ (1.35 g), $MgCl_2.6$ H_2O (6.29 g) and $NaCO_3$ (0.18 g) in tap water (1 L). The solutions were used after minimum 24 h. The quality of water can also affect the mechanical properties of cementitious materials [1–3]. Therefore, in this study, distilled water was used for the characterization tests and tap water for moulding the samples and preparing the activators and chemical solutions [4, 5].

2.2 VA-MSWI-FA Matrix-Based Alkali-Activated Mortars Procedure

The alkali-activated mortars were obtained by first mixing fine powders (90 wt.% VA + 10 wt.% MSWI-FA) during 5 min in order to obtain homogeneous powder. Secondly, by a mix of this powder with sand for another 5 min in the mass ratio of sand/powder equal to 2. Finally, the mixing of powders with sand was followed by the addition of alkaline solution in a liquid to solid binder precursor mass ratio of 0.56 for 5 min. The above-mentioned mixtures were carried out via DIGI MORTAR MIXER (Made by Amil Ltd., India). The alkali-activated mortars obtained were then poured into 5 cm cubic moulds. After vibration for 2 min, the specimens were cured at room temperature (27 ± 3 °C and 65% RH), then demoulded after 24 h and kept in closed plastic at room temperature until the mechanical test was performed after 28 days. The quality of water can also affect the mechanical properties of cementitious materials [23–25].

2.3 Resistance Test

The test consisted of the immersion of samples aged for 28 days into aggressive media for 56 days. The solution was renewed weekly in order to keep the pH constant. The weight loss and residual compressive strength were then tested after 7, 14, 28 and 56 days of immersion. The specimens were retrieved, cleaned softly with a cloth to remove loose particles and weighed. The percentage of weight loss of alkali-activated mortars was determined using the eq. (2.1):

$$\text{Weight loss } (\%) = \frac{w_i - w_t}{w_i} \times 100 \qquad (2.2)$$

Where the W_i = average initial mass of three dry unexposed specimens and W_t = average mass of three dry specimens after exposure of t days. The specimens before and after exposure were dried at 105 °C for 24 h. The change of compressive strengths of mortars was also evaluated on specimens before

and after immersion in aggressive media. The compressive strength of samples was tested in an Automatic Compression Testing Machine (AIMIL COMPTEST 2000, India). The triplet test was used for each specimen.

2.4 Characterization Test

The residual of alkali-activated mortar specimens after compressive strength test aged for 28 days and exposed in aggressive media for 56 days were dried, crushed, and sieved at 100 μm and their powders were subjected to a Bruker X-ray diffractometer (D8 Discovery, US) using radiation Cu Kα. The rotation speed was 2° /min and the scan angle is 10° to 70° was used. The different chemical bonds of unexposed and exposed powders were determined through a Fourier transform infrared spectroscopy (Nicolet 5700 FTIR, Thermo Electron Corporation) in reflectance mode in the range of 400–4000 cm^{-1}.

The morphological and phase composition of broken samples from compressive strength of unexposed and exposed at 56 days of samples were examined through HITACHI S-3400 N Scanning Electron Microscope equipped with Energy Dispersive X-ray Spectrometry analysis (EDS), operating at 15.0 kV. A DYNAVAC sputter coater was used in coating the fractured specimens with carbon.

3 Results and Discussions

3.1 Visual Appearance

The appearance of mortar specimens exposed in 2 and 4 wt.% sulphuric acid solution and seawater after 56 days is depicted in Fig. 1. The samples exposed in seawater do not show perceptible degradation. However, the white layer is formed on its surfaces. This displays that aggression is only structural. The low degradation is observed in the samples immersed in sulphuric acid solution showing that the degradation is both physical and structural. This deterioration emerges with the increase in the concentration of acid.

### 3.2 Phases Evolution and Microstructure

3.2.1 FTIR Analysis

The FTIR spectra of alkali-activated mortars before and after immersion in aggressive media for 56 days are shown in Fig. 2. The spectra of alkali-activated mortars present the band of H-O bonds at 3500 and 1450 cm^{-1}, the band of carbonate at 1650 cm^{-1} and those of aluminosilicates (Si-O-T) at 1000 and 500 cm^{-1}. In the view of these spectra, the samples immersed

Fig. 1 Appearance of mortar specimens exposed in 2% and 4% sulphuric acid solution and artificial seawater after 56 days
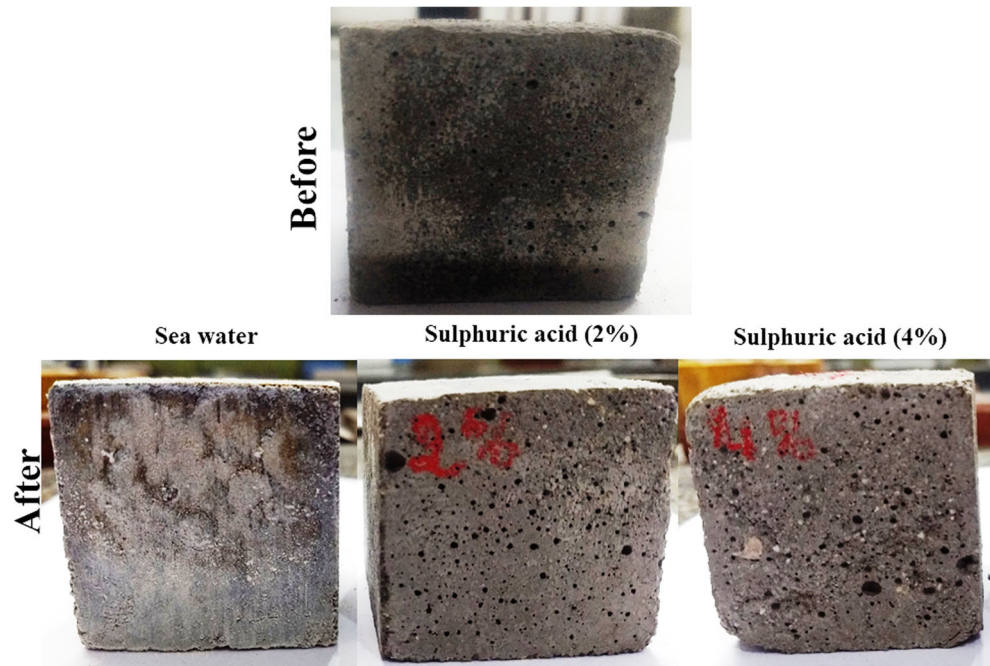

in seawater present all these aforementioned bands practically in the same position. This observation reveals that no significant structural changes take place when the geopolymer binder is immersed in seawater. Whereas the samples immersed in acid media present relevant some differences: (i) The shift of O-H band from 1471 cm^{-1} to 1623 cm^{-1} and 1619 cm^{-1} are observed for 2% and 4%, respectively. (ii) The shift of Si-O-T bonds from 970 cm^{-1} to 1124 cm^{-1} and 1122 cm^{-1} in 2% and

4%, respectively, and (iii) The appearance of S-O bond at 673 cm^{-1}. Indeed, when the geopolymer binder is attacked by acid, the neutralization reaction that wants to take part conduct to the depolymerization phenomenon. It results in the rupture of some aluminosilicate bonds to form -Si-OH and -Al-OH bonds [26]. A similar observation has been done during the dissolution of some glass phases (nepheline, jadeite and albite). The authors concluded that the depolymerization of the aluminosilicate system is due to the protonation of terminal Al hydroxyl sites within the hydrated layer of the structure which increases with lower pH [27].
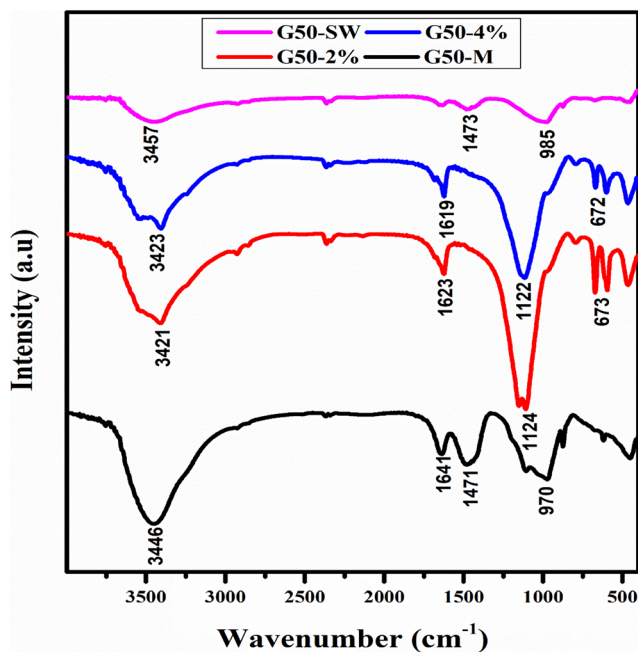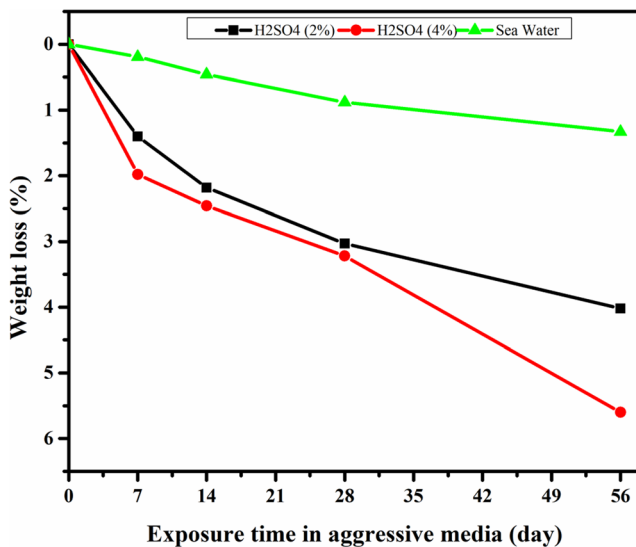

Fig. 2 FTIR spectra of alkali-activated mortars before and after immersed in aggressive media for 56 days
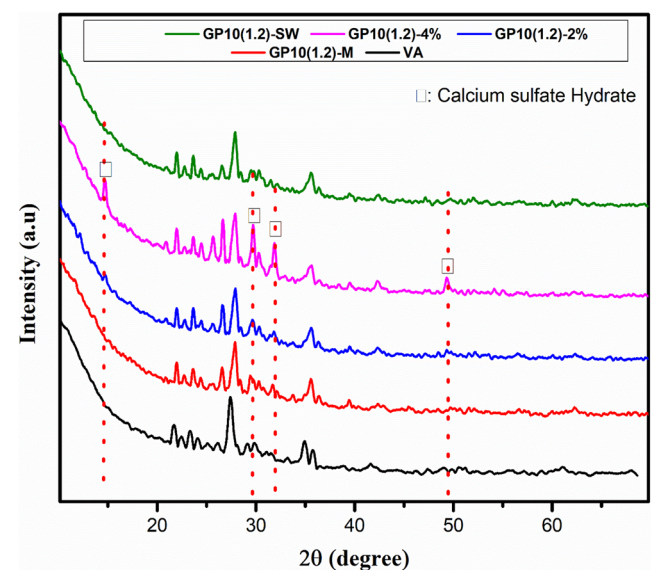
Fig. 3 XRD diffractograms of volcanic ash (VA), alkali-activated mortars before and after immersed in aggressive media for 56 days
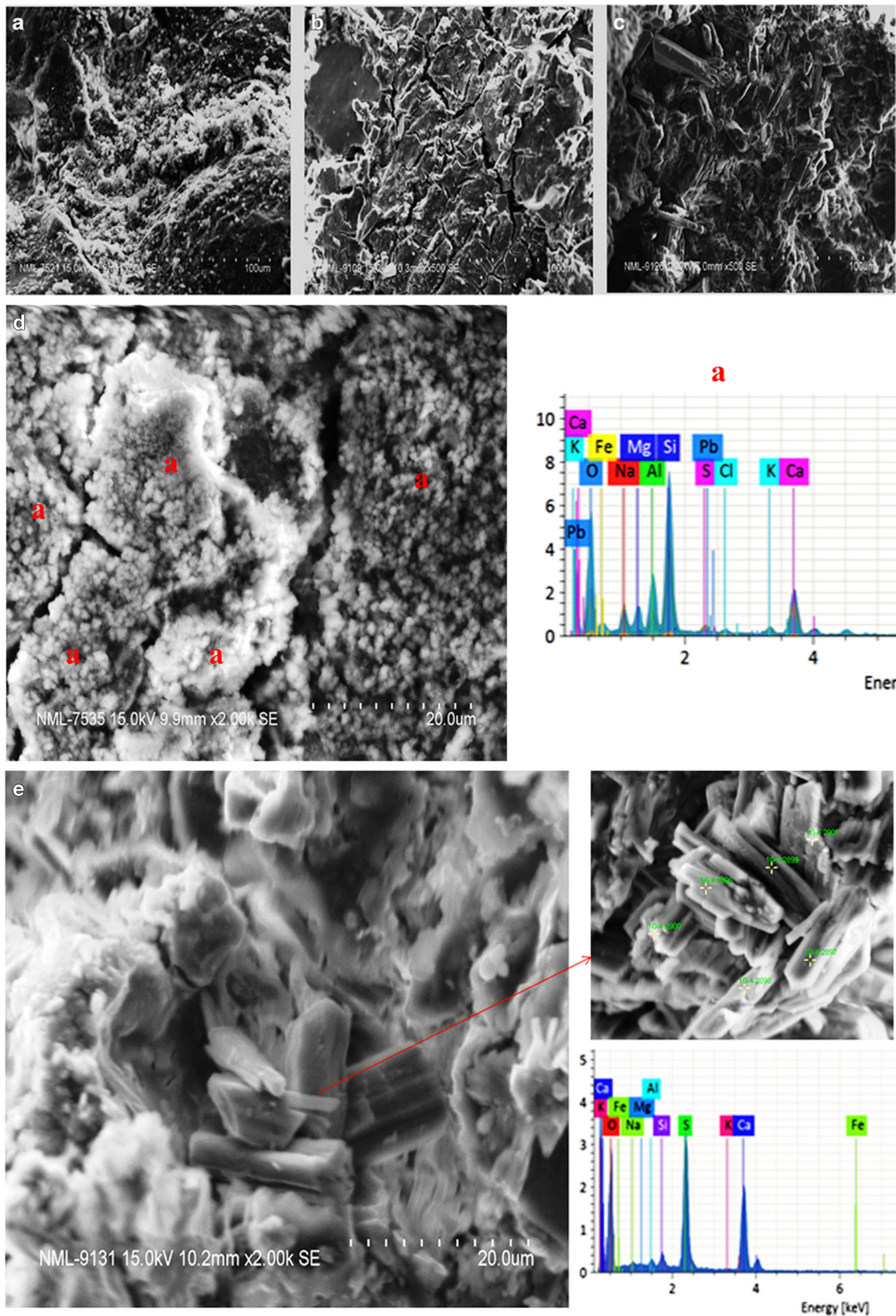
Fig. 4 a-c. Micrographs of the AAM before immersed (**a**), after immersed in seawater (**b**) and acid (**c**) for 56 days. **d.** Micrograph and EDS of the AAM after exposure to seawater for 56 days. **e.** Micrograph and EDS of the AAM after exposure to sulphuric acid solution for 56 days

Fig. 5 Variation of weight loss of alkali-activated mortars immersed in aggressive media with the time

3.2.2 XRD Analysis

The diffractograms of AAM before and after immersion in aggressive media for 56 days are presented in Fig. 3. As was previously observed in FTIR spectrum (Fig. 2), no prominence differences are observed in the diffractogram of mortars before and after exposure in seawater. This result confirms the assumption that the seawater reacts with the specimens at their surface and does not affect the chemical bonds of the system. Yet, the appearance of new peaks at 2θ equal to 14.5, and 49° is observed on the diffractograms of alkali-activated materials after immersion into sulphuric acid solution. This change characterizes the formation of a new phase namely calcium sulphate hydrate (PDF#03–0053). The intensity of these peaks increases with the concentration of the solution. Indeed, in acidic medium, the
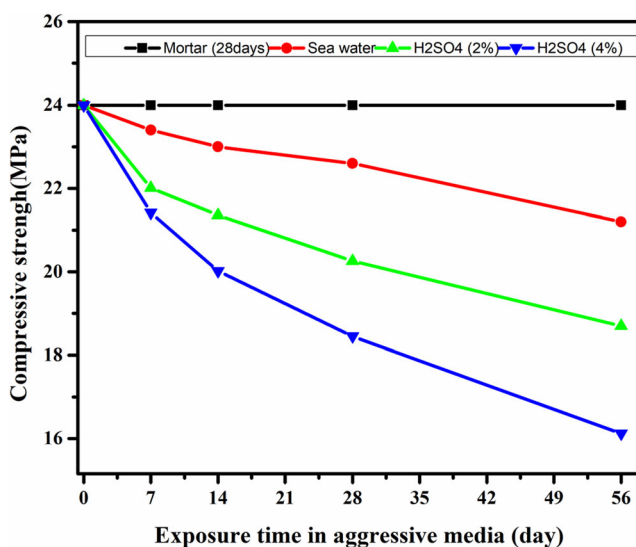
Fig. 6 Compressive strength change of alkali-activated mortars immersed in aggressive media

C-S-H gel generally undergoes a decalcification. The released Ca^{2+} ions precipitate with the sulphate ions to form the solid $CaSO_4$. On the view of these results, we can conclude that the products resulting from the depolymerization of the geopolymer binder do not form any crystalline phases.

3.2.3 SEM/EDS

The micrographs feature of alkali-activated mortars before and after immersed in aggressive media for 56 days are depicted in Figs. 4(a-e). It is seen that the alkali-activated mortars present dense microstructure with the coexistence of alkali-activated gel and C-(N-A-)S-H gel (Fig. 4). Despite the low reactivity of volcanic ash in alkaline medium, its mix design with low fraction of MWSI-FA (10 wt.%) enhances the reactivity of the solid powder. The sand particles are embedded by the matrix of AAM. Some unreacted and/or undissolved particles are observed at the surface of their structure. These particles are those which cannot take part to the polymerization process such as heavy metals, quartz sand, etc. It is well observed that the samples immersed in seawater present the microcracks on their surface and the one in acid are surrounded by the formation of new phases and the intermittence of gels (Figs. 4b and c). These facts show that the seawater improves the cracks into the matrix of alkali-activated mortar and the acid destroys the alkali-activated matrices. In the view of the composition of seawater, water is prominent by alkali and chlorine ions. When the sample is immersed in seawater, the infiltration of seawater provides these species throughout the matrix and the deep of infiltration increases with the time until the equilibrium of infiltration is achieved. After the equilibrium, the matrix is saturated of these aforementioned species. It is noticed that the alkali-activated mortars have the potentiality to leach out these species from the matrix. This diffusion may cause the formation of cavities or cracks (observed at low magnification Fig. 4d) throughout the matrix resulting in low strength when the samples are immersed in seawater. In an acid medium, the destruction of the matrix could likely be due to the aforementioned depolymerization resulting in the breaking down of Si-O-Al bonds [28]. The presence of crystalline calcium sulphate (Fig. 4e) is consistent with the XRD analysis, where only the calcium sulphate was found throughout the matrix.

3.3 Weight Loss

The percentage of weight loss with exposure time in aggressive media is showed in Fig. 5. The alkali-activated mortars in aggressive media have a low mass loss value, less than 6%. The mass loss in seawater is very low (less than 2%). This observation agrees with the visual appearance, which showed that seawater is less corrosive than sulphuric acid solution. The mass loss in the acid environment could be attributed to the probable exchange of cations (Na^+, K^+, Ca^{2+}, etc.) in the

structure by protons H$^+$ [29]. It is also due to the physical deterioration that resulted in the loss of debris detached from the sample as observed in Fig. 1. Concerning the mass loss in seawater, this is due to the leaching of alkaline ion, chlorine and the unreacted particles which are not embedded in the alkali-activated system.

3.4 Changes in Compressive Strength

The compressive strengths of mortars as a function of exposure time in aggressive media are presented in Fig. 6. The decrease after 56 days was 9.10%, 22.08%, 32.5% for specimens immersed in seawater, 2 wt.% and 4 wt.% H$_2$SO$_4$, respectively. The drop is observed in both acidic environments and seawater. The loss of strength in both media is primarily due to the fact that due the water molecules present in these media penetrated into the pore cavities between binders (C-S-H and N-A-S-H) and aggregates, making them weak. In an acidic medium, this weakness of the cohesion between the fillers is accompanied by the decalcification and depolymerization reactions mentioned below, which also affect the strength of the materials [30]. The effect of these reactions increases with the time because the longer the samples remain in acid, more they are attacked by acid. The calcium sulphate formed is expansive and therefore creates cracks, which has the direct effect on reducing compressive strength. The loss of mechanical strength by samples immersed in seawater is not predominantly due to depolymerization, but rather to the movement of ions it contains. Also, it is well known that generally, the infiltration of water molecules inside the pore cavities of AAM affect the strength of bonds that resulting the weakness of its structure [4, 16]. The penetration of chloride into the matrix through the pores forms the Friedel's salt, which is mainly results from the interaction between chloride and calcium sulfoaluminate [31]. The lack of detection of its crystalline phases in the XRD analysis is probably due to their amount being below the limit of detection. However, this low quantity formed influences the mechanical properties by forming microcracks.

4 Conclusion

This work consisted to study the behaviour of alkali-activated mortars from VA + MSWI-FA system in aggressive media i.e. sulphuric acid solution and artificial seawater. The changes in structure, microstructure, weight, and compressive strength were investigated on samples before and after being immersed in both aggressive media. Based on the above results this alkali-activated material has the potentiality of resisting seawater, whereas some modifications of the structure have been noticed. The changes in the structure of samples immersed in seawater are materialized by the shift of Si-O-T and O-H bonds which confirms the degradation of aluminosilicate phases, mainly geopolymer gel. The appearance of S-O bond subject to the formation of sulphate phases. This fact was confirmed by the formation of calcium sulphate on XRD analysis which is the result of the precipitation of calcium ion leach after decalcification of C-S-H gel and the sulphate ion presents in acid. The microstructural analysis shows the prominence of microcrack and the formation of the crystalline phase in seawater and acid, respectively. The loss of strength in both media is primarily due to the fact that the water present in these media hydrolyzes the bonds between the binders and the aggregates, making them weak. In an acidic medium, it is also due to the decalcification and depolymerization reactions. In seawater medium, it is also attributed to the movement of ions it contains and the probable formation of Friedel's salt.

Synergistic use of volcanic ash and MSWI-FA for construction materials through alkaline activation looks like the upcoming trend to valorize these wastes. Supplementary investigations of durability such as carbonation and wet-dry cycles will be carried out to really control the use of these green inorganic polymer materials.

Availability of Data and Material All data generated or analysed during this study are included in this article.

Author Contributions Sylvain Tome: Conceptualization, Methodology, Investigation, writing - original draft, resources. Achile Nana: Writing - review & editing. Cyriaque Rodrique Kaze: Writing - review & editing. Thamer Salman Alomayri: Writing - review & editing. Jean Noel Y. Djobo: Writing - review & editing. Elie Kamseu: Writing - review & editing. Marie-Anne Etoh: Writing - review & editing. Jacques Etame: Writing - review & editing. Sanjay Kumar: Conceptualization, Methodology, Writing - review & editing.

Code Availability Not applicable.

Declarations

Conflict of Interest The authors declare that they have no conflict of interest.

Consent to Participate Not applicable.

Consent for Publication Not applicable.

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