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Salt resistance of metakaolin and asphalt waste dust-based geopolymer

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Abstract

Conventional Portland cement concrete is prone to durability issues in environments with salt contamination, where corrosion, expansion, and cracking are common. Dissolved salts are particularly aggressive and act as direct agents of concrete deterioration. The study focuses on synthesizing geopolymer materials made of metakaolin (MK) and asphalt waste dust (AD) to evaluate their influence on salt resistance performance, aiming to develop a new type of construction material similar to cement-free concrete. Physical and chemical properties were examined using XRD, XRF, ICP, and FESEM techniques. In addition, compressive strength and weight loss after salt exposure were measured to evaluate the mechanical and durability performance of the geopolymer samples. Geopolymer samples were prepared with 10 wt%, 30 wt%, 50 wt%, and 70 wt% AD replacing MK. Specimens were cured for 2 day, 7 day, and 28 day, followed by immersion in salt solutions for 90 day. Results showed that up to 30 wt% AD and at least 70 wt% MK were appropriate ratios for geopolymer synthesis. Cement samples lost compressive strength and weight, whereas the compressive strength of the geopolymer increased after 90 day of salt immersion. Geopolymer with 10 wt% AD showed the best corrosion resistance, compressive strength, and maintained weight after 28 day of curing.

1. Introduction

Concrete is the main building material used in current construction due to its strength and durability. However, its lifespan can be greatly reduced when exposed to aggressive chemicals, especially salts such as chlorides and sulfates [1–4]. These corrosive agents directly damage concrete, causing problems such as corrosion, swelling, and cracks in the material [5–7]. Sulfate ions can enter the concrete and react with the calcium-rich components of Portland cement (PC), forming expansive products such as ettringite and gypsum, which create high internal stress [8,9]. At the same time, chloride ions speed up steel corrosion, causing the concrete to expand and crack (spalling) [10,11]. This widespread problem leads to frequent and costly repairs, creating a strong need for salt-resistant materials that can maintain their durability in aggressive environments over the long term [12,13]. Geopolymer technology has emerged as a highly promising, eco-friendly alternative to traditional cement, offering both durability and sustainability benefits [14–19]. Geopolymers are inorganic aluminosilicate binders made by activating suitable source materials with alkaline solutions [20–22]. Using geopolymer technology provides clear environmental advantages: it produces much lower CO_2 emissions than conventional Portland cement and creates an important opportunity to reuse industrial waste as raw materials [23–26]. In addition to geopolymer systems, conventional cement-based materials have incorporated various industrial wastes as partial replacements for natural aggregates and cement to improve resource efficiency and reduce environmental impacts. For example, granite industry waste has been investigated as a sand replacement material, with compressive strength reported to be comparable to that of conventional concrete [27]. Similarly, the combined use of high-volume fly ash, plastic waste, and graphene nanoplatelets has been explored in cement-based systems to enhance sustainability, with mechanical properties and water absorption behavior found to vary depending on mixture design and replacement levels [28]. These studies reflect the increasing focus on sustainable waste utilization in cement-based materials. However, the performance of waste-derived geopolymer systems under aggressive salt exposure remains insufficiently understood. In this context, geopolymers are generally considered to be more resistant to sulfate and chemical attack than Portland cement (PC).

Geopolymers are more durable than PC because of their unique structure. Hydration of PC produces a porous matrix made of calcium silicate hydrate (C–S–H) and calcium hydroxide ($Ca(OH)_2$) [29–31]. In contrast, geopolymerization creates a dense, three-dimensional, highly cross-linked amorphous network called poly(sialate-siloxo) (N–A–S–H or K–A–S–H) [32,33]. Importantly, several studies have indicated that geopolymer matrices contain little or no $Ca(OH)_2$, which reduces the availability of the main reactant for expansive sulfate salts, thereby providing higher natural resistance to sulfate and chemical attacks compared to conventional Portland cement-based concretes [34–37].

The performance of a geopolymer largely depends on the reactivity of its source materials, since the chemical composition and structure of these precursors strongly influence the properties of the final geopolymer [38]. In this study, metakaolin (MK) has received much attention as a key precursor providing significant advantages over

traditional PC systems. MK is a calcined kaolin clay with an amorphous structure, an ideal chemical composition, and high reactivity, all of which are essential for successful geopolymerization. This allows the formation of a uniform and strong binder with excellent mechanical properties [39,40]. This research leverages geopolymers' ability to reuse waste materials by adding asphalt waste dust (AD), a by-product of road construction with limited use, making it a useful component to improve resource efficiency and enhance the sustainability of the composite [41,42].

To encourage the use of eco-friendly materials in demanding conditions, this research combines the high-performance properties of MK with the benefits of reusing waste asphalt dust (AD). The main goal is to develop and evaluate the salt resistance of metakaolin–asphalt waste dust-based geopolymer and to explore its potential as a sustainable construction material. In particular, the developed material is intended for use as a sulfate-resistant construction material in structures exposed to sulfate-bearing environments, such as sulfate-rich soils and seawater, where enhanced chemical durability is required.

2. Experimental

2.1 Materials and sample preparation

Metakaolin (MK) from China, imported by Imerys Ceramics (Thailand) Co., Ltd., was mixed with asphalt waste dust (AD) obtained from an asphalt concrete plant in Khok Kruat Subdistrict, Mueang Nakhon Ratchasima District, Nakhon Ratchasima, Thailand. The mixtures were prepared at MK:AD weight ratios of 100:0, 90:10, 70:30, 50:50, and 30:70. The alkaline activator consisted of sodium silicate (Na_2SiO_3, Na_2O = 16.5 wt%, SiO_2 = 34.5 wt%, $SiO_2/Na_2O \approx 2.09$) and 15 M sodium hydroxide (NaOH, Qrec, New Zealand) solution mixed at a weight ratio of 2:1 [43]. The liquid-to-solid (L/S) ratio of the geopolymer paste was fixed at 0.8. For comparison, Ordinary Portland Cement (Type I, Siam City Cement Public Co., Ltd., Thailand) paste was prepared with a water-to-cement (W/C) ratio of 0.8.

The paste was cast into cubic molds (50 mm × 50 mm × 50 mm). For each formulation, three specimens were prepared for compressive strength testing, and the reported strength values represent the average of three specimens. The specimens were cured at 30°C for 2 day, 7 day, and 28 day. After curing, they were directly immersed in a 5 wt% magnesium sulfate ($MgSO_4 \cdot 7H_2O$, Loba Chemie™, India) solution for 90 day to evaluate sulfate resistance. The testing procedure was conducted with reference to ASTM C1012 [44]; but the specimens were directly immersed in the $MgSO_4$ solution without water curing.

2.2 Weight loss measurement

After curing for 2 day, 7 day, and 28 day, the specimens were weighed to obtain the initial mass (W_i). The specimens were then immersed in a 5 wt% $MgSO_4$ solution for 90 day under the same exposure conditions described in Section 2.1. After exposure, the specimens were removed from the solution. Loose and deteriorated surface particles were gently brushed off, and the surfaces were wiped with dry cloth to remove excess solution. The specimens were then weighed again to obtain the final mass (W_x) [45].

The percentage of weight loss (ΔW) was calculated using the following equation:

$$\Delta W(\%) = \frac{W_i - W_x}{W_i} \times 100 \quad (1)$$

Where W_i is the initial mass before exposure and W_x is the mass after exposure.

2.3 Sample preparation for ICP-OES analysis

The specimens were collected after immersion and compressive strength testing. To minimize the influence of Mg deposition at the exposed surface, only the interior portions of the fractured specimens were used for sampling. The collected material was oven-dried at 100°C for 24 h and subsequently ground and sieved to pass a 200-mesh sieve.

A representative 7 g portion of the powdered material was mixed with 50 mL of distilled water, followed by the addition of 5 mL of HCl (37%, ACI Labscan, Ireland). The suspension was stirred for 5 min to facilitate dissolution. The mixture was subsequently filtered through a 0.45 μm membrane filter to remove suspended solids. The filtrate was analyzed using an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES; Optima 8000, PerkinElmer, USA).

Calibration was performed using standard Mg solutions (0 to 20 $mg \cdot L^{-1}$) prepared from a certified reference standard. A linear calibration curve ($R^2 > 0.999$) was established prior to analysis. All measurements were conducted in triplicate.

2.4 Characterization techniques

The geopolymer composite was evaluated to assess its chemical composition, mechanical performance, chemical resistance, and microstructural stability under aggressive conditions. X-ray Fluorescence (EDXRF; XGT-5200, Horiba, Japan) analysis, summarized in Table 1, showed that metakaolin (MK) contained high amounts of SiO_2 (59 wt%) and Al_2O_3 (36 wt%), while asphalt waste dust (AD) contained a high amount of CaO (91 wt%) with minor amounts of SiO_2 and MgO. Although XRF results are expressed in oxide form, AD mainly consists of calcite ($CaCO_3$), as reported in our previous study [41]. Compressive strength was measured using a Universal Testing Machine capacity of 100 kN (UTM; MUL-125TTR/THAI) according to ASTM C109/C109M [46]. Magnesium ions (Mg^{2+}) were quantified by ICP-OES as described in Section 2.3 to evaluate sulfate resistance. X-ray Diffraction (XRD; D2 PHASER, Bruker, Germany) was used to investigate the crystalline or amorphous phases of the sample, while Field Emission Scanning Electron Microscopy (FESEM; JSM-7800F, JEOL, Japan) was used to examine the microstructure and morphology of the samples.

Table 1. Chemical compositions of MK and AD.

Raw materials	Chemical composition [wt%]						
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	K_2O	CaO	MgO
MK	59	36	2	2	1	-	-
AD	3	1	1	-	-	91	4

3. Results and discussion

3.1 Effect of $MgSO_4$ exposure on weight loss

Figure 1 shows that the geopolymer mixes with ratios of 100:0 and 90:10 had the lowest weight loss after exposure to $MgSO_4$, indicating that these mixes formed a stable structure resistant to sulfate attacks. In contrast, the 50:50 and 30:70 mixes showed the highest weight loss, probably because of incomplete geopolymerization caused by the lower silica and alumina content [47,48]. On the other hand, Portland cement shows significant deterioration due to Mg^{2+} replacing Ca^{2+} in its C–S–H structure, weakening the structure and making it more prone to sulfate attack, which results in higher weight loss. These observations are discussed in more detail in Section 3.3, where the sulfate resistance behavior of both Portland cement and geopolymer samples is analyzed.

The results also show that longer curing times greatly reduce weight loss in all geopolymer samples, highlighting the importance of curing in improving material durability. These findings suggest that higher MK content contributes to a more stable geopolymer network capable of resisting sulfate-induced degradation.

3.2 Effect of $MgSO_4$ exposure on compressive strength

Figure 2 presents the compressive strength results. The compressive strength of the geopolymer samples gradually increased with curing age, and only the 90:10 ratio showed compressive strength above the minimum requirement of 28 MPa at 28 day, according to ASTM C150/C150M-22 [49], comparable to Portland cement Type I. Among the geopolymer formulations, the mix containing at least 70% metakaolin exhibited the highest compressive strength. Meanwhile, higher asphalt dust (AD) content lowered the silica-to-alumina ratio in the mixture and reduced the polymerization reaction and consequently the overall strength [47,48].

After 90 day of $MgSO_4$ immersion, for samples cured for 28 day, the 100:0 and 90:10 mixes exhibited the highest compressive strengths of 29.16 and 37.59 MPa, respectively, while the 50:50 and 30:70 mixes showed lower values of around 5 MPa to 9 MPa. This behavior corresponds well with the weight loss trend, where mixes with higher asphalt dust content (50:50 and 30:70) exhibited greater deterioration and lower durability under $MgSO_4$ exposure.

In addition, a slight increase in compressive strength was observed in the 100:0 and 90:10 mixtures after $MgSO_4$ exposure compared to their 28 day values. This behavior may be related to continued geopolymerization after the initial curing stage. In mixtures with low AD content, metakaolin remains the main reactive precursor, allowing further formation of the aluminosilicate network during immersion. This continued reaction may lead to additional gel formation and a denser internal structure. At the same time, the presence of a small amount of AD may also contribute by acting as a filler, helping to reduce pores and improve compactness. The combined effect of continued gel formation and improved packing may explain the slight strength increase observed after sulfate exposure. This explanation is consistent with the SEM observations discussed in Section 3.4, where the 90:10 mixture exhibits a relatively dense and continuous matrix after 90 day of $MgSO_4$ exposure compared to mixtures with higher AD contents.

In comparison, Portland cement showed a clear decrease in compressive strength after soaking in $MgSO_4$ solution, confirming its lower resistance to sulfate attack. This reduction is attributed to the reaction of $MgSO_4$ with the cement structure, which decomposes calcium silicate hydrate (C–S–H) into magnesium silicate hydrate (M–S–H). The formation of M–S–H leads to surface corrosion and a consequent loss of compressive strength [8,13,16].

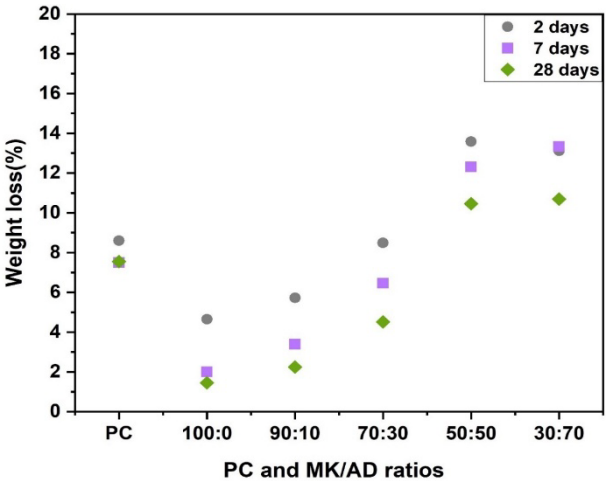

Figure 1. Weight loss (%) of PC and MK/AD-based geopolymer at various mix ratios after curing for 2 day, 7 day, and 28 day, followed by 90 day of $MgSO_4$ exposure.

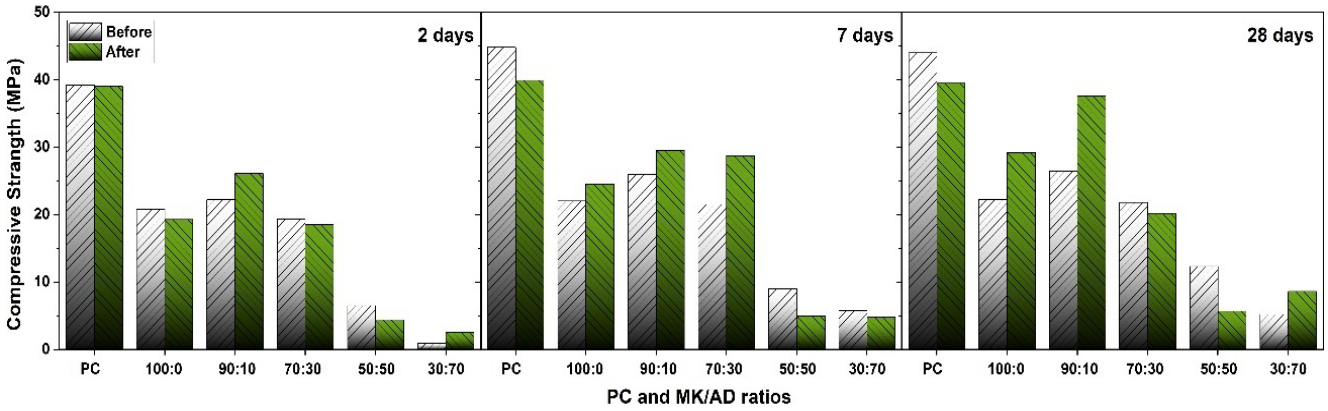

Figure 2. Compressive strength of PC and MK/AD-based geopolymer at various mix ratios after curing for 2 day, 7 day, and 28 day, before and after 90 day exposure to $MgSO_4$ solution.

3.3 Effect of sulfate attack

Figure 3 presents the differing behaviors of PC and geopolymer samples under sulfate attack, based on Mg^{2+} ion concentrations ($mg{\cdot}L^{-1}$) measured by ICP-OES after immersion in $MgSO_4$ solution for 2 day, 7 day, and 28 day. All geopolymer samples exhibited significantly higher Mg^{2+} concentrations compared to PC, which showed only 0.45 $mg{\cdot}L^{-1}$, 0.62 $mg{\cdot}L^{-1}$, and 1.5 $mg{\cdot}L^{-1}$ at 2 day, 7 day, and 28 day, respectively. Among the geopolymers, the Mg^{2+} concentration generally increased with higher MK content, reaching a maximum of 10.63 $mg{\cdot}L^{-1}$ after 28 days for the 90:10 MK: AD ratio. These results indicate that Mg^{2+} ions are consumed in reactions within the PC sample, leading to more severe corrosion, whereas in geopolymers, Mg^{2+} does not significantly interact with the geopolymer structure.

The deterioration of PC occurs through several destructive reactions with $MgSO_4$, as illustrated in Equation (2–6) [50].

$$Ca(OH)_2 + MgSO_4 + 2H_2O \rightarrow CaSO_4{\cdot}2H_2O + Mg(OH)_2 \quad (2)$$

$$3(CaSO_4{\cdot}2H_2O) + 3CaO{\cdot}Al_2O_3{\cdot}12H_2O + 14H_2O \rightarrow 3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O + Ca(OH)_2 \quad (3)$$

$$3(MgSO_4{\cdot}7H_2O) + 3(3CaO{\cdot}Al_2O_3{\cdot}12H_2O + 12H_2O \rightarrow 3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O + 3Mg(OH)_2 \quad (4)$$

$$x(CaO{\cdot}ySiO_2{\cdot}zH_2O) + xMgSO_4{\cdot}(3x + 0.5y - z)\,H_2O \rightarrow xCaSO_4{\cdot}2H_2O + xMg(OH)_2 + 0.5ySiO_2{\cdot}nH_2O \quad (5)$$

$$4Mg(OH)_2 + SiO_2{\cdot}nH_2O \rightarrow 4MgO{\cdot}SiO_2{\cdot}8.5H_2O + (n - 4.5){\cdot}H_2O \quad (6)$$

1. Attack on calcium hydroxide $Ca(OH)_2$ reacts with $MgSO_4$ to form gypsum ($CaSO_4{\cdot}2H_2O$) and brucite ($Mg(OH)_2$) in Equation (2).
2. Ettringite formation: The gypsum continuously reacts with calcium–aluminate hydrates (C–A–H) to form ettringite ($C_6AS_3H_{32}$) and regenerate $Ca(OH)_2$ as shown in Equation (3). Similarly, magnesium sulfate can also react directly with C–A–H to produce ettringite as shown in Equation (4).
3. Attack on C–S–H (Decalcification): Calcium silicate hydrate (C–S–H), the primary binder in PC, reacts with $MgSO_4$ to produce gypsum, brucite, and silica gel as shown in Equation (5). The resulting gypsum can further react with calcium aluminate hydrate (C–A–H) to form additional ettringite, as described in Equation (3).
4. Formation of M–S–H: Finally, brucite reacts with silica gel to form magnesium silicate hydrate (M–S–H), a weak and non-cementitious phase as shown in Equation (6).

Therefore, the low Mg^{2+} concentration in PC solutions reflects high corrosion activity and the formation of M–S–H within the cement structure. In contrast, the geopolymer samples exhibited strong sulfate resistance and maintained their geopolymerization network. This behavior is attributed to the absence of $Ca(OH)_2$ in geopolymers, which prevents the formation of M–S–H [34–37]. Among the geopolymer mixes, the 90:10 sample cured for 28 day showed the highest residual Mg^{2+} concentration, indicating high structural stability and low reactivity under sulfate exposure. This suggests that the incorporation of 10 wt% AD contributed to improved matrix compactness, likely through a filler effect that enhanced particle packing. As a result, the geopolymer network remained stable under sulfate exposure. This behavior consistent with the compressive strength and weight loss results, where the 90:10 mixture also exhibited improved strength retention and lower mass change after sulfate exposure.

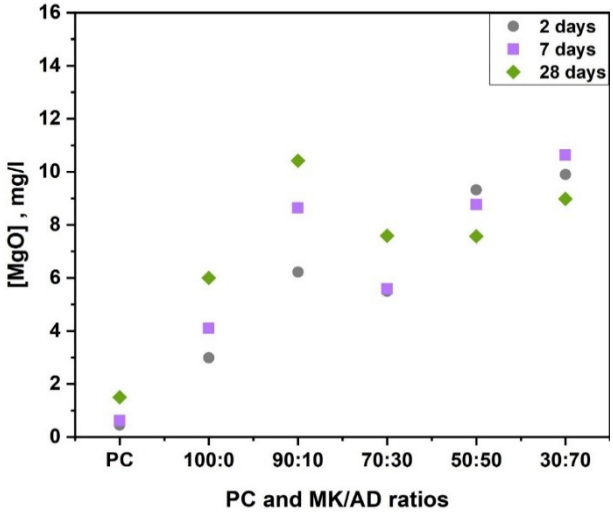

Figure 3. Sulfate resistance of PC and MK/AD-based geopolymer at various mix ratios after curing for 2 day, 7 day, and 28 day, followed by 90 day of $MgSO_4$ exposure.

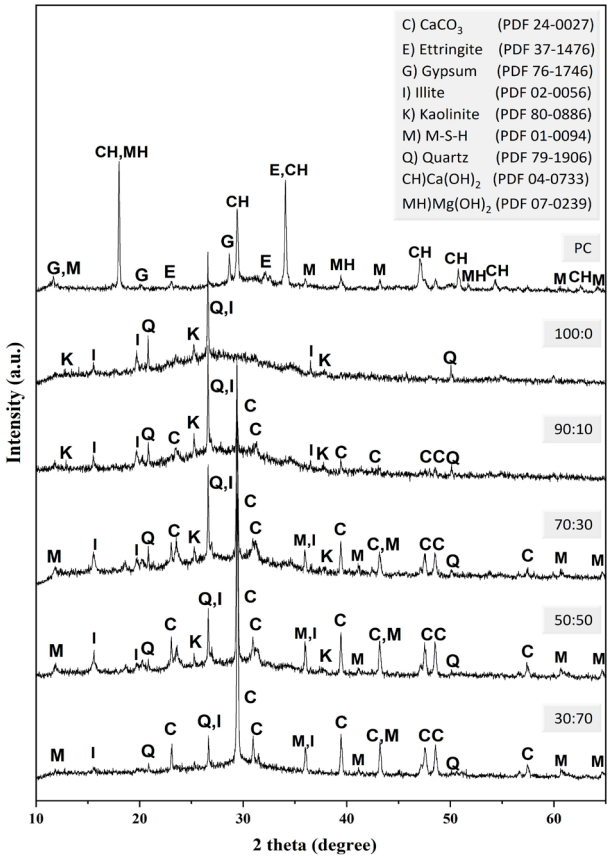

Figure 4. XRD analysis of PC and MK/AD-based geopolymer at different mix ratios after 28 day of curing, following 90 day exposure to $MgSO_4$ solution.

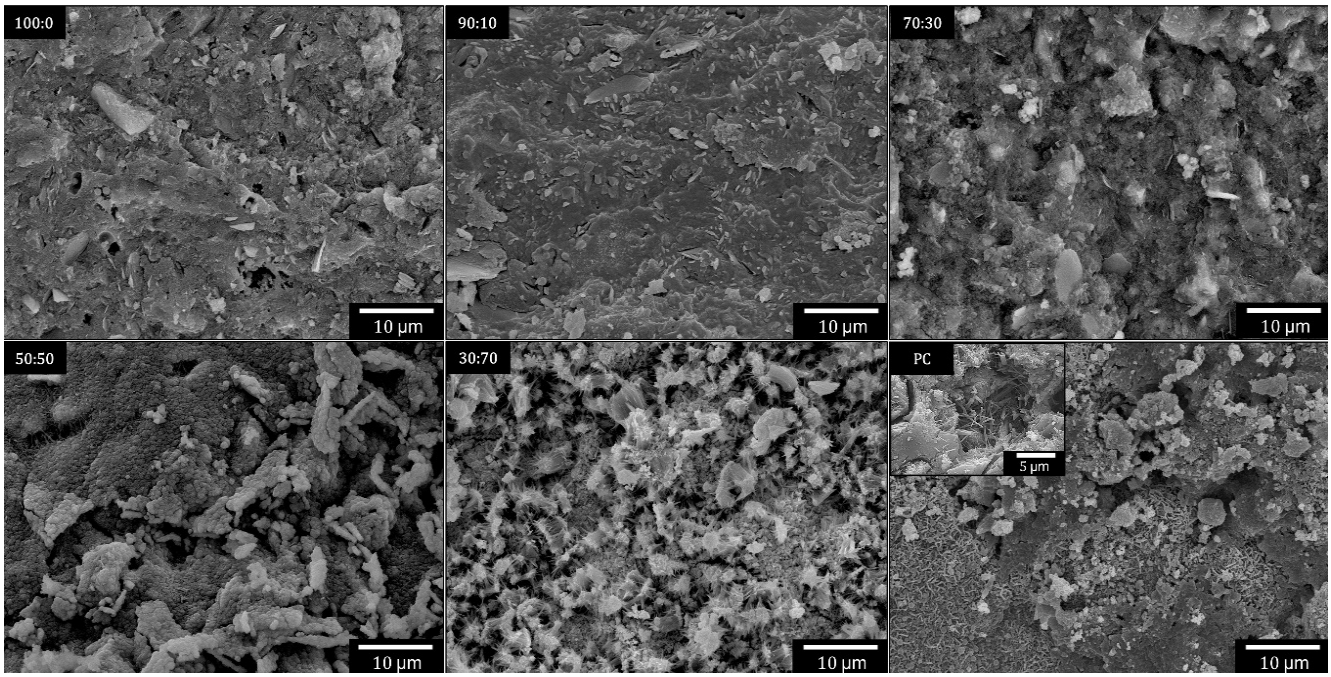

Figure 5. SEM images of PC and MK/AD-based geopolymer at various mix ratios after 28 day of curing and 90 days in $MgSO_4$ solution.

3.4 Effect of $MgSO_4$ exposure on phase transformation

Figure 4 presents the XRD analysis, showing the differences in phase transformation between PC and geopolymer samples under sulfate exposure. The XRD patterns of PC revealed the formation of undesirable phases, including gypsum, ettringite, magnesium sulfate, magnesium hydroxide, and magnesium silicate hydrate (M–S–H), which results from the transformation of C–S–H. In contrast, M–S–H formation in geopolymer samples occurred only when the Metakaolin (MK) content was below 70%, as a result of minor leaching of silica and alumina by the NaOH activator, allowing limited interaction with magnesium ions [51]. Importantly, the 100:0 and 90:10 geopolymer mixes showed no M–S–H formation, demonstrating their superior phase stability and strong resistance to sulfate attack.

Figure 5 presents the SEM images, which are discussed together with the XRD results to understand phase changes and material stability. After $MgSO_4$ exposure, the PC samples showed the formation of crystalline features and a less compact matrix, indicating significant microstructural alteration. Point-EDS analysis at selected areas showed the presence of Ca–Al–S. The calculated Ca/S and Ca/Al ratios (5.13 and 4.72, respectively) suggest the presence of ettringite-like phases, although the values are higher than the theoretical stoichiometric ratios, likely due to the influence of surrounding matrix Ca. In addition, Mg–Si rich regions were identified, with an Mg/Si ratio of approximately 1.5, indicating M–S–H–like gel formation. These reaction products are associated with reduced stability in the PC samples.

Regarding the geopolymer samples, the mixtures with MK:AD ratios of 100:0 and 90:10 showed a more compact structure, indicating better stability after sulfate exposure. The addition of 10% AD appears to reduce porosity, likely due to a filling effect that improves particle packing. However, when the AD content increases further, the MK content decreases. This lowers the available Al–Si needed for geopolymerization. As a result, gel formation becomes less complete, leading to a weaker and less compact structure. This is consistent with the lower compressive strength observed at higher AD contents.

Overall, the 90:10 MK:AD ratio identified in this study can be considered optimal under the specific materials and test conditions applied. Its application to other precursor sources, activator systems, or exposure environments may require further investigation. Nevertheless, the results demonstrate that AD can be effectively utilized as a supplementary waste material without compromising durability, provided that its content is properly controlled to maintain sufficient geopolymer gel formation.

4. Conclusions

Metakaolin (MK) and asphalt waste dust (AD) as precursors for geopolymers were prepared to evaluate their performance in salt-environmental resistance. Meanwhile, PC showed significant weight loss and decreased compressive strength due to Mg^{2+} ions replacing Ca^{2+} in C–S–H, leading to severe corrosion. Geopolymers with MK: AD ratios of 100:0 and 90:10 exhibited low weight loss, increased strength, and maintained their structure, with longer curing times further enhancing performance. The geopolymer with 90:10 (MK: AD) ratios showed the best stability under $MgSO_4$ salt exposure. XRD and SEM analyses confirmed M–S–H formation in PC samples, whereas geopolymers with 90% MK and above showed none of the M–S–H phase. These results indicate that geopolymers, made of metakaolin and asphalt waste dust, provide a sustainable and durable alternative to PC with superior resistance to magnesium sulfate salt attack.

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