# Mechanical Behavior and Durability of Perlite Based Mortar Exposed to Sodium Sulfate Attack

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The objective of this study is to gather additional on the impact of perlite on both the mechanical properties and resistance to chemical attacks of materials. Various combinations ,incorporating perlite in the form of cement additions, were examined. Five different substitution rates of 10 %, 15 %, 20 %, 25 %,and 30 % of cement with powder perlite were utilized for comparison with plain cement. The findings indicate that the interaction between lime,silica,and alumina in powder perlite results in the formation of several hydrates, such as calcium silicate hydrate, hydrated calcium aluminates,and hydrated gelhenite.Our research focuses on the development of natural perlite from maghnia as a potentially pozzolanic supplementary cementing material. In evaluating the pozzolanicity of this material, it was observed that external aggressions, such as chlorides, $CO_2$, and chemical attacks, can compromise the physical and mechanical properties of concrete,affecting its long-term durability. However,natural pozzolans like pelite exhibit a positive influence on the durability of mortars against sulphate attacks. Test results revealed specimens with no apparent degradation, indicating that sulphate ions in the solution did not adversely affect perlite-based mortars. There is a growing preference for mineral additives with lower production costs than cement. Consequently, the optimal mixture was determined to be one containing 25 % perlite. However, cement pastes and mortars incorporating up to 20 % perlite demonstrated satisfactory physical and mechanical properties, comparable to materials without perlite. The investigation into the perlite materials used in conjunction with cement suggests the potential for sustainable concrete. Experimental results indicate that natural perlite powder from Maghnia can be considered a good pozzolanic material, suitable as a mineral admixture in cement production.

*Keywords:* mortar, cement, perlite, mechanical properties, attack, durability, sodium sulfate, chemical.

## 1. INTRODUCTION

The incorporation of perlite into cement or concrete systems offers numerous advantages to concrete properties. These include improvements in workability, reduction in the heat of hydration, high thermal conductivity, and enhanced strength. However, it is noteworthy that many volcanic materials, particularly natural perlite, may slow down the rate of strength development. Reference [1] has reported that blended Portland cements containing 10 %, 20 %, and 30 % Santorini Earth, a natural pozzolan from Greece, exhibited similar or higher compressive strength compared to the reference Portland cement [2].

The investigation of the physical phenomena associated with the flow of mortar holds significant importance in the realm of rheology. The objective of this approach is to elucidate and analyze the macroscopic rheological properties of fresh mortar. In the behavior of constituent phases, perlite, a siliceous volcanic glass, is noteworthy for its substantial volume expansion under the influence of heat. When heated beyond 870 °C, its volume increases four to twenty times the initial volume [3]. Perlite finds applications in diverse fields such as building materials, agriculture, medicine, and the chemical industry [4]. Its non-hazardous nature and fire resistance further contribute to its increased utilization [5]. The primary aim of this study is to provide additional data on the effects of perlite on mechanical properties and durability. A substitution rate of perlite powder (PP) between 10 % and 20 % is anticipated to enhance mechanical performance over time, densify the transfer matrices of mortars, and ensure resistance to carbonation [6]. Conversely, the advantageous properties of perlite, such as extremely high water retention and low thermal conductivity, broaden its applications. Consequently, its production has increased. The qualities of lightness and insulation make perlite particularly suitable for concrete in roof decks [7, 8]. Expanded perlite has been employed as an aggregate replacement [9]. The incorporation of mineral additives has become a crucial technique in improving concrete properties such as fluidity, strength, and durability. These additives significantly impact the rheology of cement materials in their fresh state, directly influencing the development of strength and durability in hardened materials. However, to fully leverage these benefits and select the optimal mix design, understanding the characteristics of these new components (mineral additives) and their effects on concrete properties is essential. Consequently, the design of concrete is highly ontingent on the environment it will face during its lifespan. To reduce total cement consumption in the concrete

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industry, numerous studies have explored the application of cementitious materials like fly ash, blast furnace slag, and silica fume to cement concrete [10]. Sulfate attack stands out as a significant concern jeopardizing the durability of concrete. Various sulfate sources, such as magnesium sulfate and sodium sulfate, are present in ground or surface waters, as well as natural soils. Structures and components that come into contact with groundwaters, soils, or wastewater are susceptible to potential damage from magnesium and sodium sulfate attack [11]. The origin and control of sulfate attack phenomena remain imperfectly understood. Sulfate attack manifests through the precipitation of secondary sulfate products, resulting in substantial expansion and chemo-mechanical deterioration, including changes in porosity, transport properties, cracks, and loss of strength and cohesion. The consequences can lead to the deterioration of cementitious materials, with the severity varying over time based on the nature, content, and concentration of sulfates in contact, as well as the type of cement used and the water/cement ratio. Sulfate ions, integral to the attack, may either be of internal origin within the concrete or external, emanating from the surroundings. Generally, internally sourced sulfate ions arise from constituents of the concrete, such as cement, aggregates, and mixing water. The degradation of concrete under this type of attack is evidenced by the phenomenon of expansion, closely tied to the crystallization of secondary ettringite. Research on the degradation of cementitious materials due to internal sulfate attacks has shown that the development of secondary ettringite, known for its expansive characteristics, results in swelling. It is crucial to differentiate this form of ettringite from primary ettringite, which emerges in the early phases of Portland cement hydration through the interaction of gypsum within the initial few minutes of water-cement contact. Primary ettringite is non-detrimental and plays a vital role in regulating cement setting [12]. Incorporating pozzolanic materials like natural pozzolan and perlite offers several advantages. This not only aids in decreasing environmental pollution but also significantly improves the durability of concrete. When these materials are introduced to concrete, they typically modify the existing pores in the concrete, thereby reducing water permeability [13]. Therefore, the use of natural pozzolan and perlite affects both the initiation and propagation time of advances in steel corrosion. A cement replacement of 20 % and 30 % of pozzolana and perlite respectively has a negative effect on corrosion in the aggressive medium. Values of Ecorr are low. This means that the sulphate ions did not affect the corrosion activity in the concrete samples throughout the immersion period (365 days), the results obtained show that 10 % and 20 % of natural pozzolan improve the mechanical properties, and it is noted that 10 % of the replacement of the cement by pozzolan and 10 % of the perlite led to a reduction of the speed of NaCl corrosion [14].

It was observed that the mixture with 2 % NS and 15 % replacement of OPC with perlite could increase the electrical resistivity to 35.2 kΩ·cm and reduce the migration coefficient to $4.1 \times 10^{-12}$ m$^2$/s at 91 days. Moreover, this mixture decreased the water absorption at 28 days to the extent of 43 % in comparison with the control mixture [15].

## 2. MATERIALS AND METHODS

### 2.1. Cement

The cement employed in all the tests was CPA-CEM-I from the farge cement plant, in accordance with the Algerian Standard for cement, specifically CEM-I 42, Ordinary Portland cement (OPC), conforming to the requirements specified in NA 442-2008 [16]. The physical and chemical properties and mineralogical composition of this cement are shown in Table 1 and Table 2. Mortars, intended for the creation of test specimens with dimensions of $(5 \times 5 \times 5)$ cm³, were mixed and prepared using ordinary Portland cement , following the procedures outlined in the NF EN 196-1 standard . The ratios of cement to sand and water to cement were consistently maintained at 1:3 and 1:2, respectively, for all specimens. Each solution yielded twelve test tubes, comprising three test tubes for each mortar mixture. These test tubes were demolded after a 24-hour curing period and then submerged in lime-saturated water for a subsequent 28-day period, maintaining a constant temperature of $20 \pm 2$°C. This curing process precedes subjecting the specimens to sulfate attack. Upon completion of the curing period, the test pieces underwent weighing to establish their initial weight before the chemical attack. Subsequently, they were immersed in solutions containing 5 % Na$_2$SO$_4$, simulating a sulfate attack. This comprehensive procedure aligns with established norms, ensuring a systematic approach to evaluating the resistance of the mortar to the deleterious effects of sulfates. The initial curing in lime-saturated water allows the mortar to develop strength before exposure to the chemical challenge, facilitating a thorough assessment of its performance under sulfate attack conditions.

**Table 1.** The physical properties and mineralogical composition of the cement

| Physical properties | | |
|---|---|---|
| Powder | (PER) | Unit |
| Specific surface area | 3308 | cm$^2$/gr |
| Volume mass apparent | 1.02 | gr/cm$^3$ |
| Density | 3.12 | - |
| Mineralogical composition (perlite) | | |
| C$_2$S | 25.69 | % |
| C$_3$S | 47.15 | % |
| C$_3$A | 2.41 | % |
| C$_4$AF | 15.12 | % |

**Table 2.** Elemental chemical composition of cement, %

| CaO | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | SO$_3$ | K$_2$O | Na$_2$O | MgO | IR[1] | PAF[2] |
|---|---|---|---|---|---|---|---|---|---|
| 63.89 | 21.4 | 4.59 | 5.52 | 2.72 | 0.41 | 0.13 | 1.37 | 0.22 | 2.47 |
| [1] IR: Insoluble residue; [2] PAF: Loss on ignition. | | | | | | | | | |

## 2.2. Perlite

Perlite is a siliceous volcanic rock [17]. The rock undergoes initial crushing and calibration through particle size analysis. It is extracted from the Boughrara Hammam deposit located in Tlemcen, Algeria. The industrial expansion of perlite is carried out by EFISOL in special furnaces, either fixed or rotary [17]. When subjected to heat, as depicted in Fig. 1, the perlite granules undergo expansion, resulting in the formation of numerous closed cells inside the grains. For all the tests, perlite is utilized in the form of a powder screened at 80 μm [18]. Perlite is a natural pozzolan abundant in several countries which are major producers of cement. This makes perlite attractive for producing sustainable concretes. Strength development and heat evolution of perlite-containing mixtures, and the influence of chemical and thermal activation on their early and later age properties were investigated using five activator chemicals and four curing temperature-duration combinations. Chemical activation could increase the 1-day, or 3-day compressive strengths of 25 % perlite mortars to above the cement-only control but was not very beneficial to 50 % perlite mixtures. Thermal activation could increase strength at ages up to 28 days, and even above the room-temperature control at 1 day. Perlite content affects the maximum value of the isothermal calorimetry heat evolution rate but not its time of occurrence. Chemical and thermal activation influence both the height of the rate peak and its timing, which are linearly correlated for activated or non-activated pastes with a given perlite content [19]. The chemical composition of perlite, % is presented in Table 3.

**Table 3.** Elemental chemical composition of perlite, %

| CaO | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | SO$_3$ | K$_2$O | Na$_2$O | MgO | PAF |
|-----|------|--------|--------|-----|-----|------|-----|-----|
| 2.38 | 72.81 | 13.68 | 2.15 | 0.01 | 3.64 | 0.97 | 0.05 | 4.20 |

### 2.2.1. Grinding operation

Before the grinding operation, the perlites are dried at 110 °C, crushed, and sieved through a 4.75 mm sieve. The purpose of sieving is to maintain uniformity between each grinding operation by using the same sizes of power supply. The grinding operation was carried out using a laboratory grinder with a length of 450 mm and a diameter of 420 mm. The grinder's speed was set at 30 rpm. The crusher load consisted of spherical and cyclops steel balls. The total weight of the load (98 kg) and the quantity of material to be ground (7 kg) were kept constant throughout the grinding operations. During the grinding operation, samples of approximately 100 g were taken at regular intervals to determine certain properties such as density, Blaine fineness, and the quantity of material retained on an 80 μm sieve. The grindability of materials was studied by obtaining 'grinding fineness-time' relationships for perlite and cements made with them. Fineness was determined by measuring Blaine fineness and the amount of material retained on an 80 μm sieve after wet sieving.

### 2.2.2. X-ray diffraction (XRD) of perlite

The mineral analysis of natural perlite from the Hammam Boughrara deposit using X-ray diffraction (XRD) is presented in Fig. 2, a schematic representation of the XRD of perlite.
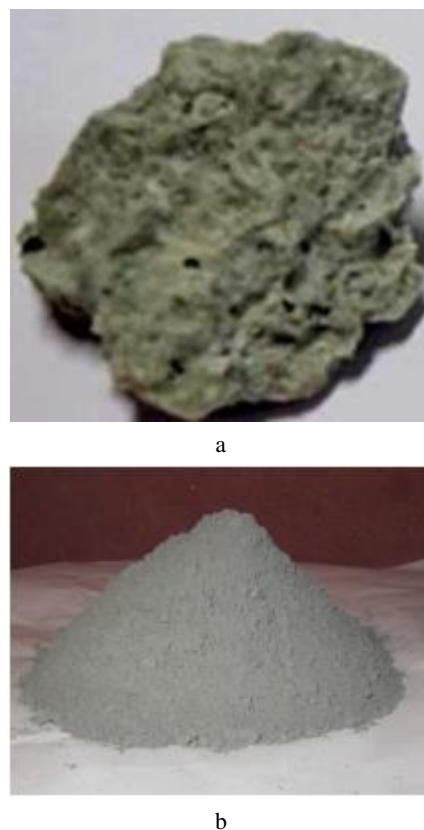


**Fig. 1.** Photographs: a – slags of expanded perlite; b – powder of the Boughrara hammam perlite after grinding, D < 80
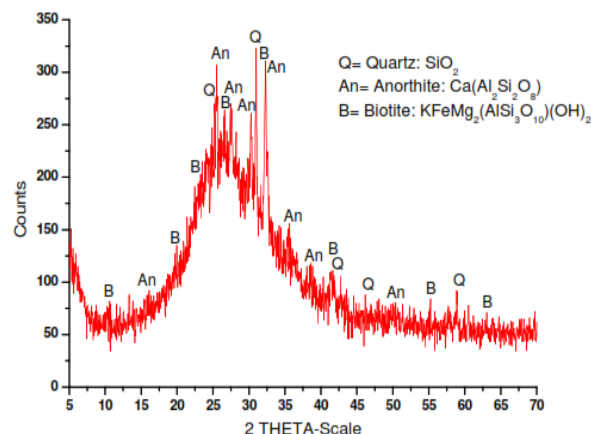


**Fig. 2.** A schematic representation of the XRD of perlite

The diagram indicates that natural perlite comprises minerals common to volcanic substances. X-ray diffraction (XRD) analysis reveals a significant presence of quartz, anorthite, and biotite. No hydraulic minerals were identified in this material, but the detachment of the diffractogram baseline suggests the presence of a potentially reactive amorphous phase.

## 2.3. Sand

The sand utilized for mortar mixes is standardized sand certified in accordance with EN 196-1 ISO679 (ISO standardized sand) is a natural, siliceous sand, especially in its finest fractions. It is clean, with grains that are generally

isometric and rounded in shape. The sand is dried, screened, and prepared in a modern workshop, offering full guarantees of quality and regularity. It is packaged in polyethylene bags, each containing $1.350 \pm 5$ g. The particle size composition, as determined by sieving, meets the standards' requirements.as shown in Fig. 3, Fig. 4 and Table 4.



**Fig. 3.** Standard sand EN 196-1

**Table 4.** Standardized sand particle size

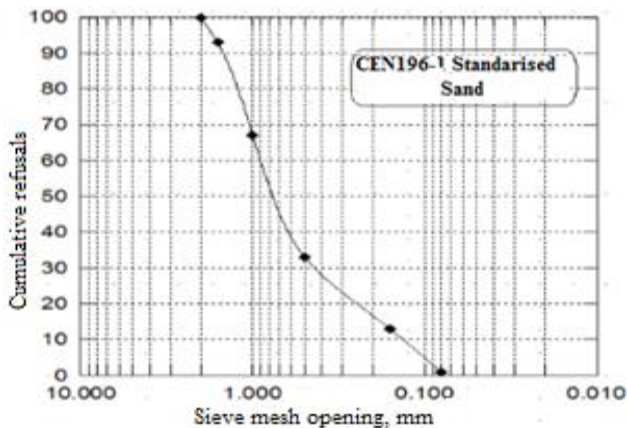| Sieve mesh opening, mm | Cumulative refusals, % |
|---|---|
| 0.08 | 99±1 |
| 0.16 | 87±5 |
| 0.50 | 67±5 |
| 1.00 | 33±5 |
| 1.60 | 7±5 |
| 2.00 | 0 |



**Fig. 4.** Grain size curve for standardized sand

## 2.4. Water for mixing

Drinking water is suitable for mortar spoilage. Chlorides, in small proportions, may slightly modify the setting and hardening of the cement. However, a high proportion can react with the cement, compromising the durability of the mortar. Such effects are detrimental to reinforcements, as their corrosion can cause the mortar to burst. NF EN206-1 (NF P 18-325) sets the maximum amount of chlorine ions [20]. The properties of water are presented in Table. 5

## 2.5. Formulation and preparation of pastes various

Binders were produced by substituting ordinary Portland cement C with different proportions of natural perlite NP (10, 15, 20, 25, and 30 % of the cement weight), Table 6 outlines the different composite binders, Table 7. The formulation and preparation of pastes were conducted in accordance with the requirements of ASTM C305 – 20, 15 Jul 2020 [21]. The binder and water were previously weighed on a precision balance with a $\pm 0.01$ g accuracy. The mixture was then blended in a vertical shaft mixer with a 5-liter capacity.

**Table 5.** The chemical composition analysis of the mixing water.

| Compound | Symbol | Content, mg/l |
|---|---|---|
| Chlorures | Cl | 128 |
| Sulfates | $SO_4$ | 192.12 |
| Magnésium | Mg | 52 |
| Calcium | Ca | 88 |
| Dioxyde de carbone | $CO_2$ | 2.31 |
| Bicarbonates | $CO_3H$ | 137 |
| Matières organiques | | 0.18 |
| PH = 7.91 | | |

**Table 6.** Proportions of mixtures of different mortars

| Designation of the mortar | Perlite, % | W/L | Spread, mm |
|---|---|---|---|
| M0 | 0 | 0.5 | 117 |
| M10 | 10 | 0.5 | 112 |
| M15 | 15 | 0.5 | 100 |
| M20 | 20 | 0.5 | 98 |
| M25 | 25 | 0.5 | 96 |
| M30 | 30 | 0.5 | 93 |

**Table 7.** The different composite binders

| Mortars | Binder | Cement | Natural perlite |
|---|---|---|---|
| M0 | 100%C+0%NP | 500 | 0 |
| M10 | 90%C+10%NP | 450 | 50 |
| M15 | 85%C+15%NP | 425 | 75 |
| M20 | 80%C+20%NP | 400 | 100 |
| M25 | 75%C+25%NP | 375 | 125 |
| M30 | 70%C+30%NP | 350 | 150 |

### 2.5.1. Workability of fresh mortars

The workability of the fresh mortars was assessed using the shaking table method in accordance with ASTM C 1437 standard [5]. A consistent water-to-lime (W/L) ratio of 0.5 was maintained for the preparation of all paste mixtures, and the corresponding results are detailed in mortars tend to decrease with an increasing rate of cement substitution by perlite. the table is providing details about the mortar mixtures and workability.

The results of spreading tests Fig. 5, indicate that the water quantity needs to be increased for mortars incorporating natural perlite to achieve a spreading value within ± 5 % of that observed in cement mortar without any addition. The water demand to mortars tends to rise with an increase in the substitution rate of cement with natural perlite.

### 2.5.2. Warm-up temperature

The control mortar, M0, reached its highest temperature of 31.2°C after 17.10 hours of mixing. For mortars M10, M15, M20, M25, and M30, the maximum temperatures were 29.30, 28.20, 27.40, 26.55, and 26.50 °C, respectively, achieved after 18.20, 18.30, 18.80,

18.85, and 18.90 hours of mixing, Table 8. Consequently, the increase in the substitution of cement with natural perlite powder led to a decrease in the heat released. This behavior can be explained by the lower reactivity of natural perlite compared to pure cement.
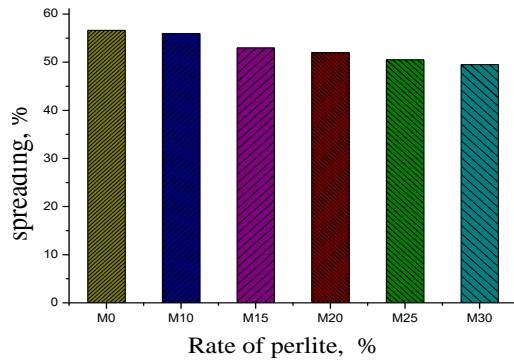


**Fig. 5.** The spreading of the mortar based on the Perlite rate

**Table 8.** Maximum temperatures attained during the hydration of mortars

| Mortars | Maximum temperature achieved, °C | Time to reach maximum temperature, h | Rate of maximum temperature decrease, % |
|---------|------|------|------|
| M0 | 31.20 | 17.10 | – |
| M10 | 29.30 | 18.20 | 6.10 |
| M15 | 28.20 | 18.30 | 9.61 |
| M20 | 27.40 | 18.80 | 12.17 |
| M25 | 26.55 | 18.85 | 14.90 |
| M30 | 26.50 | 18.90 | 15.10 |

# 3. RESULTS AND DISCUSSION

## 3.1. Setting times

The results for the start and end of setting of all pastes are given in Table 9.

**Table 9.** Setting times of various cement pastes

| Cements | Beginning of the take | End of the take |
|---------|------|------|
| C0 | 103 | 197 |
| C10 | 99 | 178 |
| C15 | 88 | 165 |
| C20 | 85 | 155 |
| C25 | 79 | 145 |
| C30 | 74 | 138 |

According to ASTM C191-21 [22], the setting time should be a minimum of 45 minutes and a maximum of 420 minutes. Fig. 6, illustrates that all recorded setting times for cements containing natural perlite exceed the 45-minute minimum requirement and are within the 420-minute maximum limit. This confirms that all mixes containing natural perlite meet the specified standards.

The results, depicted in Fig. 6, clearly indicate that the setting times (both start and finish) accelerate with the addition of natural perlite. The natural perlite used in this study has a high $Al_2O_3$ content (13.68 %), and its inclusion in the cement increases the amount of aluminate compounds in the mix. According to Bresson [23], increasing the C3A content of cement from 3 % to 11 % decreases the setting time by half.
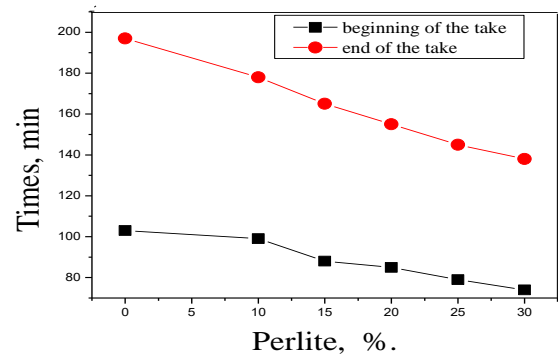


**Fig. 6.** Variation in setting time as a function of perlite rate

Additionally, accelerated setting in pastes incorporating natural perlite can be associated with an increased formation of calcium sulphate hydrate (tri- and monosulpho aluminate).

## 3.1.2. Mortar formulation and preparation

In accordance with ASTM C109/C109M20 [24], the material proportions for each mortar are one part cement and 2.75 parts sand by mass. For mortar prepared from Ordinary Portland Cement without the addition of perlite, a water-cement mass ratio of 0.485 is used. The water-cement ratios used in mortars prepared from Portland cement with varying percentages of perlite are adjusted to attain a spread within ±5% of that observed in the cement mortar without perlite. These ratios will be determined through workability tests conducted on the spreading Table 10, details regarding the compositions of each mortar mix. Table 11.

**Table 10.** Mortar spreading results

| Mortars | Perlite, % | S/L | W/L | Spreading | Spreading, % |
|---------|------|------|------|------|------|
| M0 | 0 | 2.75 | 0.5 | 15.65 | 56.65 |
| M10 | 10 | 2.75 | 0.5 | 15.60 | 56.00 |
| M15 | 15 | 2.75 | 0.5 | 15.30 | 53.00 |
| M20 | 20 | 2.75 | 0.5 | 15.20 | 52.00 |
| M25 | 25 | 2.75 | 0.5 | 15.05 | 50.50 |
| M30 | 30 | 2.75 | 0.5 | 14.95 | 49.50 |

**Table 11.** Proportions of the different mortar mixtures

| Source code | Cement, g | Natural perlite, g | Sand, g |
|---------|------|------|------|
| M0 | 491 | 0 | 1350 |
| M10 | 441.90 | 49.10 | 1350 |
| M15 | 417.35 | 73.65 | 1350 |
| M20 | 392.80 | 98.20 | 1350 |
| M25 | 368.50 | 122.75 | 1350 |
| M30 | 343.70 | 147.30 | 1350 |

Proportions of the different mortar mixtures. However, the compressive strength test was conducted on cubic mortar specimens ($50 \times 50 \times 50$ mm) following the ASTM C109/C109M20 [24] standard. After 24 hours, the specimens were demolded and immersed in water saturated with lime at a temperature of $23 \pm 2$ °C until the specified test age. The crushing of the test piece occurs once it is removed from its storage environment; it is wiped with a dry cloth. Subsequently, it is placed in the crushing cell of

the hydraulic press, applying a loading rate with a relative movement speed between the upper and lower plate corresponding to a loading speed on the specimen of 1000 N/s.

## 3.2. Compressive strength

Table 12 presents the resistance results depicted in Fig. 7, which illustrates the influence of perlite substitution percentages on the compressive strength of mortars at different time intervals. Upon analyzing Fig. 7, it becomes evident that the compressive strengths of mortars, produced with different perlite substitution rates, consistently remain lower than those of the control mortar at all stages of development. Initially, increasing the dosage of perlite negatively affects the compressive strength.

**Table 12.** Results of compressive strength

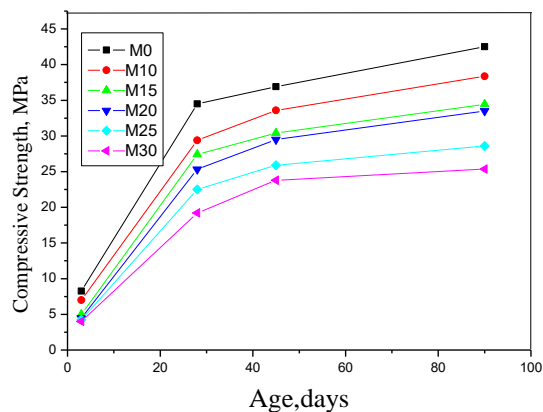| Source code | Age, days | | | |
|---|---|---|---|---|
| | 3 | 28 | 45 | 90 |
| M0 | 8.25 | 34.5 | 36.90 | 42.5 |
| M10 | 7.00 | 29.40 | 33.60 | 38.35 |
| M15 | 5.00 | 27.40 | 30.40 | 34.45 |
| M20 | 4.50 | 25.33 | 26.50 | 30.50 |
| M25 | 4.25 | 22.50 | 25.90 | 28.60 |
| M30 | 4.00 | 19.20 | 20.80 | 25.35 |



a



b
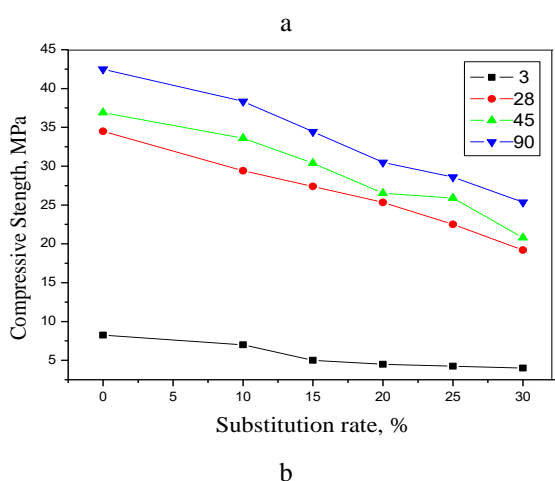
**Fig.7.** a – evolution of the compressive strength of different mortars; b – variation in the strength of perlite mortars as a function of the substitution rate

Indeed, the difference in strength widens from 15 % to 52 % at 3 days compared to the control, for perlite dosages ranging from 10 % to 30 %. However, this discrepancy tends to decrease over time. It diminishes from 43 % to 44 % at 28 days and from 9 % to 44 % at 45 days for the same substitution rates (10 – 30 %), highlighting the positive long-term effect of the perlite substitution. Additionally, it is observed that the perlite level indeed influences resistance, with an increase in perlite correlating to a proportional decrease in resistance.

### 3.2.1. Results of compressive strength in the solution of 5 % $Na_2SO_4$

The resistance of various mortars to acid attack was assessed following the guidelines of ASTM C267, 2001. Cubes of different mortar compositions, measuring $50 \times 50 \times 50$ mm, were employed to evaluate their resistance to acid exposure. Following a 28-day curing period in lime-saturated water, the cubes were immersed in a $Na_2SO_4$ solution at a controlled temperature of $20 \pm 2$ °C for 13 weeks. This extended exposure period allowed for a comprehensive evaluation of the mortars' durability under acid attack conditions. These results distinctly show that the difference in strength between the control mortar and the mortars incorporating various levels of perlite continuously decreases over time Table .13.

**Table 13.** Results of compressive stength in the solution of 5 % $Na_2SO_4$

| Source code | Age, days | | | |
|---|---|---|---|---|
| | 3 | 28 | 45 | 90 |
| M0 | 7.5 | 29.35 | 30.5 | 38.4 |
| M10 | 7.2 | 28.30 | 32.7 | 37.9 |
| M15 | 5.30 | 26.40 | 29.45 | 35.65 |
| M20 | 4.90 | 27.45 | 30.5 | 31.50 |
| M25 | 4.57 | 26.6.9 | 29.50 | 32..35 |
| M30 | 4.40 | 23.55 | 24.60 | 28.45 |

When comparing the strength of mortars incorporating natural perlite with mortar without perlite at each age, it is observed that the strength values decrease with age for the mortar without perlite. The strength values decrease as the rate of replacement of cement by perlite increases, particularly at the seventh age, highlighting a notable impact on the seventh day of hardening. However, it is important to note that this difference diminishes over time, likely due to the pozzolanic reaction of the natural perlite with portlandite resulting from the hydration of the silicate phases of the clinker ($C_3S$ and $C_2S$). Ghrici et al. [25] demonstrated that the resulting secondary C-S-H gel from the pozzolanic reaction of natural pozzolan, after deposition in the pores, leads to an increase in the compressive strength of mortars, particularly in the long term. Moreover, the ASTM C1157/C1157M-20a [26] standard for hydraulic binders requires minimum strengths of 20 and 30 MPa at 3 and 7 days, respectively. In this study, the compressive strength values of all mortars incorporating natural perlite exceed 20 and 30 MPa at 3 and 7 days, satisfying the minimum strength requirements specified in this standard. The workability of pozzolanic pastes decreases as the rate of substitution of cement by natural pozzolan increases. Additionally, pozzolanic pastes require more water to achieve normal consistency,

impacting the cement hydration process and increasing the setting and finishing times in proportion to the percentage of pozzolan [27]. As shown in Fig. 8, it can be observed that at an early age (3 days), all mortars exhibit strength gains. However, by 28 days, all mortars experience an increase in strength. The strength gains at 3 days can be attributed mainly to the filler effect of natural perlite, which enhances the compactness of the mortars.
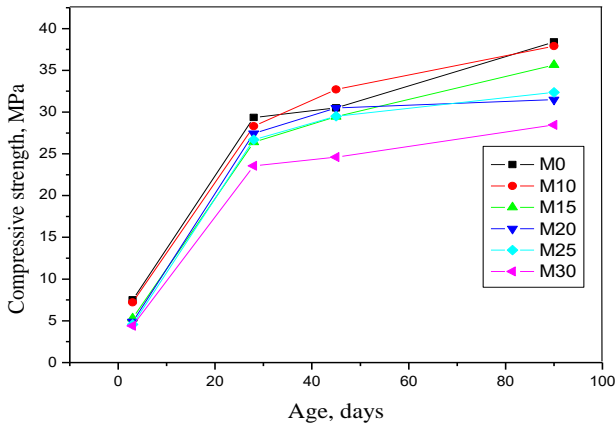


**Fig. 8.** Compressive strength of concrete in the solution of 5 % $Na_2SO_4$

The strength losses observed on the 28th day in the reference mortar are the result of the dilution effect of the cement. This dilution effect leads to less intense hydration of the binder, resulting in the formation of small quantities of C-S-H hydrates. From 28 days onward, the dilution effect of the cement is offset by the pozzolanic reaction of natural perlite with the portlandite produced during cement hydration. This explains the strength gains observed, with the increase in strength being directly proportional to the percentage of natural perlite in the cement. This gain in strength reflects the pozzolanic activity of natural perlite, which develops with age, contributing to increased strength. Similar observations were made by Ghrici. Cement mortars incorporating 10 %, 20 %, and 30 % natural pozzolan exhibit increasing strength losses from 2 to 7 days of age, with the exception of the 10 % dosage, which shows a gain at 7 days. However, from 28 days onward, all mortars experience strength gains that continue to increase with age. It can be concluded that natural perlite is among the mineral additions that can enhance the microstructure and mechanical properties of composite cement. The reaction mechanism can be divided into a physical effect, which is predominant at a very young age, and a chemical effect, which is predominant in the long term. The physical effect involves ultrafine particles filling the voids between cement grains, creating a denser microstructure in the cement mortar. The chemical effect results from the active silica of natural perlite consuming the portlandite formed during cement hydration to produce secondary C-S-H, filling the material's porosity and contributing to its increased strength.

### 3.3. Sodium sulphate attack

The variations in the length of the mortar samples immersed in the sodium sulphate solution for 12 weeks are shown in Fig. 9.

Cubes of different mortars (50 × 50 × 50 mm) were used to assess the resistance to sodium sulfate attack. After 28 days of curing in lime-saturated water, the cubes were immersed in a 5 % sodium sulfate ($Na_2SO_4$) solution at $20 \pm 2$ °C for 12 weeks. Samples were taken each week from the solution, rinsed with tap water, and then weighed. The samples were then placed back into freshly prepared solutions asshownin Fig. 10.
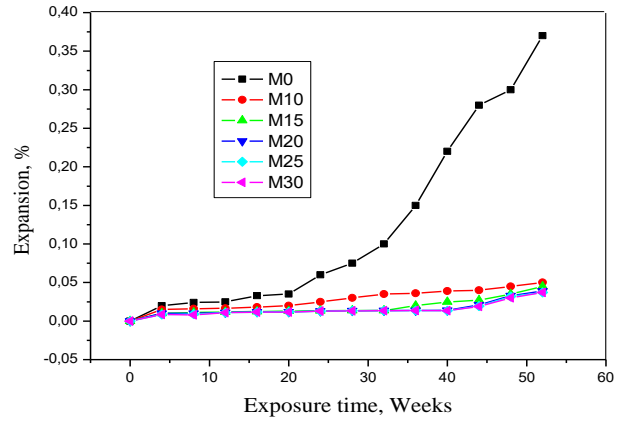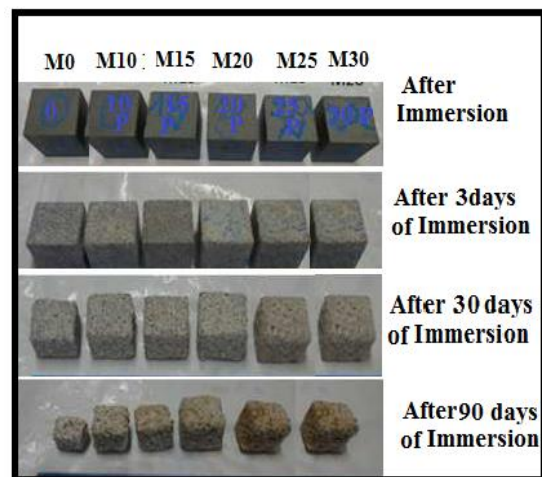


**Fig. 9.** Evolution of the expansion of different mortars immersed in a 5 % solution of $Na_2SO_4$ solution



a



b

**Fig. 10.** a – visual inspection of mortar before and after etching with 5 % $Na_2SO_4$; b – visual inspection of mortar after etching with 5 % $Na_2SO_4$
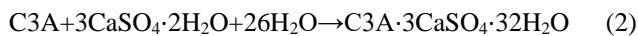
After an apparent stabilization period of around 9 weeks, the expansion of the M0 mortar increased more

rapidly than those incorporating natural perlite. After 12 weeks of exposure, the expansion value of the M0 mortar is five to nine times greater than that of the M10, M15, M20, M25, and M30 mortars. Additionally, increasing the percentage of natural perlite in the cement reduces the expansion of the mortar. According to ASTM C1157 /c1157-20 [26], a mortar made with moderately sulfate-resistant cement must have an expansion of less than 0.10 % after 180 days in a 5 % $Na_2SO_4$ solution. Furthermore, a mortar made with cement with high sulfate resistance must have an expansion of less than 0.05 % after 180 days and less than 0.10 % at 1 year. In our study, it is noteworthy that all mortars incorporating natural perlite met the requirements of the ASTM standard C1157/c1157M-20 [26] particularly at replacement levels above 10 %, concerning cement with high resistance to sulfates. The progressive degradation and dissolution of the cement matrix at a young age are attributed to the decomposition of calcium hydroxide and, consequently, the formation of a quantity of gypsum which dissolves according to the following reaction:
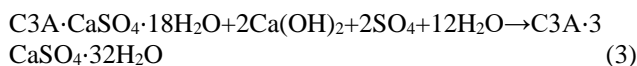
$$Ca(OH)2+Na_2SO_4+2H_2O \rightarrow CaSO_4 \cdot 2H_2O+2NaOH \quad (1)$$
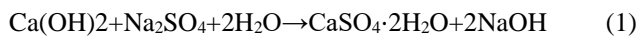
In this reaction, calcium hydroxide ($Ca(OH)_2$ reacts with sodium sulfate ($Na_2SO_4$) in the presence of water ($2H_2O$). The reaction results in the formation of secondary gypsum ($CaSO_4 \cdot 2H_2O$) and sodium hydroxide ($2NaOH$). The sodium hydroxide generated contributes to high alkalinity, which in turn stabilizes the formation of calcium-silicate-hydrate (C-S-H), a crucial component for the strength and durability of cementitious materials. The expansive product formed, $CaSO_4 \cdot 2H_2O$, is noteworthy for its expansion properties. However, this expansion is typically limited to the internal spaces of the hydrated cement paste, resulting in little or no overall expansion of the material.

Secondary ettringite formation from residual Anhydrous C3A:

$$C3A+3CaSO_4 \cdot 2H_2O+26H_2O \rightarrow C3A \cdot 3CaSO_4 \cdot 32H_2O \quad (2)$$

From hydrated aluminates (monosulfoaluminate):

$$C3A \cdot CaSO_4 \cdot 18H_2O+2Ca(OH)_2+2SO_4+12H_2O \rightarrow C3A \cdot 3CaSO_4 \cdot 32H_2O \quad (3)$$

These reactions describe the formation of secondaryettringite

$$C3A \cdot 3CaSO_4 \cdot 32H_2OC3A \cdot 3CaSO_4 \cdot 32H_2O \quad (4)$$

The first reaction involves residual anhydrous tricalcium aluminate (C3A), and the second reaction occurs with hydrated aluminates (monosulfoaluminate), which is a complex compound involving calcium hydroxide, sulfate, and water. Secondary ettringite formation is a significant aspect of cement chemistry. The presence of ettringite can impact the properties of concrete, including its volume stability and resistance to sulfate attack. The specific reactions outlined highlight the different pathways through which ettringite can form in the presence of sodium sulfate.

### 3.3.1. Loss of mass

The variation in the curves presented in Fig. 11 demonstrates significant mass losses for all the studied mortars. The reference mortar M0 exhibits the highest mass loss compared to those incorporating natural perlite. The mass of mortars incorporating natural perlite gradually decreases with the substitution of cement. After 12 weeks of immersion in the sulfuric acid solution, the mass loss of M10, M15, M20, M25, and M30 is lower than that of M0.
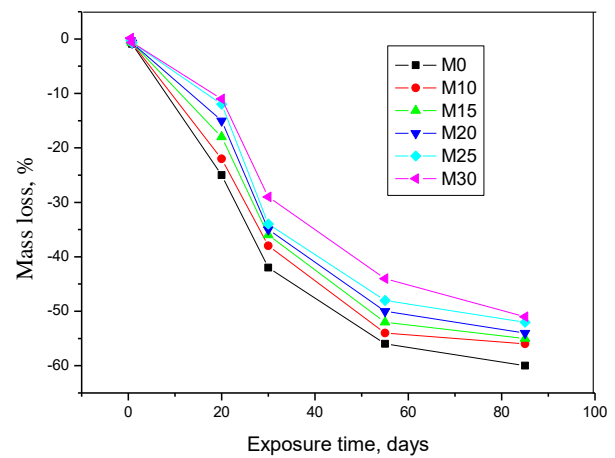


**Fig. 11.** The variation in the loss of mass of mortar specimens when preserved in a 5 % $Na_2SO_4$ (sodium sulfate) solution

### 3.3.1. Microstructure analysis by XRD

After 12 weeks of immersion in the sodium sulfate solution, mortar samples M0 and M25 from the outer layers were analyzed by XRD. The XRD spectra of mortars prepared with and without natural perlite are shown in Fig. 12. The mortar samples chosen from the outer layers of the prisms of each mortar were analyzed by XRD after immersion in the sodium sulfate solution.
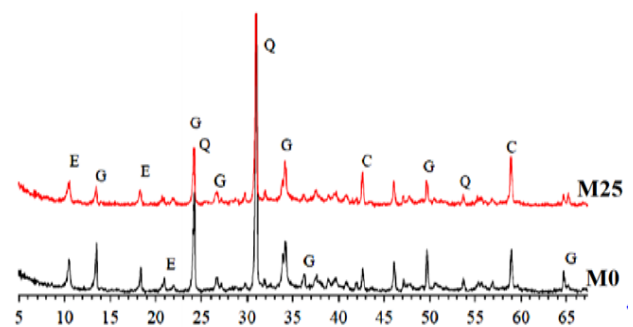


**Fig. 12.** XRD patterns of M0 and M25 mortars after 12 weeks in a 5 % $Na_2SO_4$ solution. E – ettringite; G – gypsum; P – portlandite; Q – quartz; C – calcite

Superimposing the spectra of different mortars reveals peaks with high quartz intensity, attributed to the siliceous nature of the sand used in mortar preparation. It should also be noted that after 12 weeks of exposure to sodium sulfate attack, no recognizable ridge of portlandite could be observed in the outer layer of the M0 and M25 mortars. This absence may be linked to the total leaching of this product following its reaction with sulfates.

## 4. CONCLUSIONS

Based on the provided information, the conclusions drawn from the study on the use of natural maghnia perlite in cement can be summarized as follows:

1. Pozzolanic classification:
- According to ASTM C618, natural perlite from Maghnia is classified as a class N pozzolanic addition.
- Mineralogical analysis via XRD revealed the presence of a potentially reactive amorphous phase.
2. Chemical resistance and $SO_4^{-2}$ ions:
- Incorporating natural Algerian perlite enhances mass and benefits the chemical resistance of pozzolanic cement to $SO_4^{-2}$ ions.
- Visual examination and negligible volume variation post-tests indicate no apparent degradation, demonstrating the resilience of perlite-based mortars to sulfate ions.
3. Physico-mechanical characteristics:
- Cement with natural Maghnia perlite requires more water for the paste to reach normal consistency.
- The addition of natural perlite accelerates the setting times of pastes.
- The reference mortar M0 exhibits the highest mass loss compared to those incorporating natural perlite.
- The mass of mortars incorporating natural perlite gradually decreases with the substitution of cement. After 12 weeks of immersion in the sulfuric acid solution.
4. Influence on resistance:
- Perlite level influences resistance, with increased perlite correlating to a proportional decrease in resistance.
5. Early-stage degradation:
- Early-stage progressive degradation and dissolution of the cement matrix are attributed to the decomposition of calcium hydroxide.
- This process leads to the formation of a significant quantity of gypsum, which subsequently dissolves.
- This chemical transformation is crucial for understanding observed changes in the cement matrix, emphasizing the dynamic nature of materials in early-stage degradation.
- The addition of natural perlite to cement causes a reduction in the maximum temperature of the mortar.
- The strength losses observed on the 28th day in the reference mortar immersed in a 5 % $Na_2SO_4$ solution, are the result of the dilution effect of the cement.
- The strength gains observed, with the increase in strength being directly proportional to the percentage of natural perlite in the cement. This gain in strength reflects the pozzolanic activity of natural perlite, which develops with age, contributing to increased strength.

## Acknowledgments

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