



Article Durability Behavior of Mortars Containing Perlite Tailings: Alkali–Silicate Reaction Viewpoint

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Abstract: Tailing incorporation into mortars has been the subject of much research in recent years. Despite this, most of these studies did not investigate the harmful effects resulting from the exposure of such mortars to an environment containing aggressive agents. This work investigated the effects of perlite tailing addition into mortars containing cement CP V-ARI MAX and hydrated lime. The raw materials were subjected to chemical characterization (X-ray fluorescence (XRF)) and mineralogical (X-ray diffraction (XRD)), while the samples immersed in 1 N NaOH solution were characterized by XRD, thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and compression strength (CS). The results showed the harmful effects of incorporating perlite tailings into the mortar investigated. Such a degradation was proven by linear expansion and compressive strength experiments accomplished in the samples after the test of resistance to an alkali–silicate reaction.

Keywords: perlite tailings; aggregate; alkali-silicate reaction

1. Introduction

Perlite is a natural vitreous volcanic rock that, when heated to around 900–1200 °C, expands 5 to 20 times its original volume [1], making it suitable for various applications in the most diverse industries, such as construction, paint industries, pharmaceuticals, soil and water filtration, and the plastics industry [2–5]. However, during the processing of raw pearlite (i.e., cutting, crushing, grinding, and particle size classification), a large volume of tailings is generated, with particle sizes ranging from microns to about 0.5 mm [6]. In this processing process, two types of waste are generated: the coarse perlite (CP) tailings from the crushing stages and the fine tailings (PP) from the grinding and granulometric classification stages. The expanded perlite production process also generates a significant amount of tailings; these are finely granulometry (<100 μ m) and extremely low bulk density (50–150 kg/m³), which makes them difficult to handle [7]. These tailings, whether natural or expanded, are often improperly disposed of, causing negative impacts on the environment [8–11].

The need to give an adequate destination to perlite tailings has generated numerous researches which using these tailings for different purposes [12–17]. The technical feasibility of using the tailings of expanded perlite in Portland cement mortar was studied regarding compression strength and proven by the study of Kapelusznaet et al. [8]. Erdoğan and Sağlık [18] investigated the use of natural perlite tailings (non-expanded) in



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). mortars as a partial replacement for cement (25% and 50%). The authors reported that incorporating 25 and 50% of the perlite tailings decreased the compressive strength by 4.45% and 32.29%, respectively.

Ramezanianpour et al. [14] incorporated calcined perlite powder (1 h at 850 °C), finely ground (fineness of 3100 cm²/g) as supplementary cementitious material (SCMs) in concrete. The authors reported that the incorporation of fine calcined perlite powders consumes lime and reduces the conductivity of the cement pore solution due to pozzolanic reactions. Most studies have focused on using expanded perlite tailings mainly as pozzolanic material [19,20]. Therefore, it is noted that there is a lack of studies related to the use of natural (non-expanded) perlite tailings. Furthermore, there is a gap in the literature about the harmful potential of these tailings concerning alkali-aggregate reactions (AARs).

Alkali-aggregate reactions occur between alkalis (Na₂O and K₂O) present in Portland cement and reactive aggregates in the presence of high moisture (80–85%) and adequate temperature beyond the pH [21,22]. An expansive gel is among the main products of AARs. Such a gel absorbs water, swells, and leads to the appearance of cracks and consequent collapse of the cement matrix [22–24]. AARs can be classified into three types: alkali–silica, alkali–carbonate, and alkali–silicate reactions [25]. The alkali–silica reaction occurs between the alkalis in the cement and the reactive minerals in the aggregate. Meanwhile, the alkaline–carbonate reaction occurs between certain dolomitic limestones and the alkaline solutions present in the pores of the concrete. On the other hand, the alkali–silicate reaction has the same nature as the alkali–silica reaction but with a slower process involving some silicates present in feldspars, certain metamorphic and sedimentary igneous rocks, deformed (strained) quartz, and expansive minerals. Furthermore, when alkali–aggregate reactions occur in cementitious matrices, they cannot be interrupted; thus, preventing the occurrence of these reactions is the best way to produce mortars with durability to chemical attacks [25,26].

In this context, there is a clear need to investigate the aggregates used in mortar and concrete because, even if types of cement with low alkali concentration are used, reactions can still occur because the aggregates can be constituted by reactive minerals formed by alkali silicates, which may react with the hydroxyl ions released by portlandite during the cement hydration process, causing the alkali–silicate reaction (ASR). It is known that the perlite tailings contain sodium and potassium aluminum silicate phases (i.e., anorthoclase, (Na, K) AlSi₃O₈) [27]. The reactive minerals in the anorthoclase react with the alkalis present in Portland cement, triggering the alkali–silicate reaction.

Therefore, it is necessary to investigate the harmful potential of perlite tailings in relation to AARs to be safely used as an aggregate in mortar and concrete without compromising their duration of usefulness. Thus, the objective of this work is to determine the harmful potential of tailings from perlite processing in relation to alkali–silicate reactions. For this, the perlite tailings were chemically and mineralogically characterized (X-ray fluorescence and X-ray diffraction), while evidence after the attack in a solution of 1 N NaOH was characterized by XRD, TGA, SEM, and CS.

2. Materials and Methods

2.1. Materials

The materials used in this research were cement CP V-ARI MAX, hydrated lime (Carbomil S.A., Limoeiro do Norte, CE, Brazil), and tailings from the processing of perlite (Bentonita do Nordeste S.A., PB, Brazil). The perlite tailings were received in two distinct granulometry: powdered and coarse perlite (PP e CP, respectively). The cement CP V-ARI MAX was used in this research because it has low alkalinity (0.69%) and does not have pozzolanic material in its composition; this makes it possible to investigate the influence of mineral tailings additions in the expansion process. According to information from the manufacturer, the cement CP V-ARI MAX has ~70% clinker. Calcitic lime was used because it has a content of CaO greater than 95%. The chemical composition of cement CP V-ARI and lime are shown in Table 1.

Raw Material	Oxides (%)										
	CaO	SiO ₂	MgO	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	K ₂ O	TiO ₂	Na ₂ O _{eq} ^a	LOI ^b	C ₃ A ^c
Cement Lime	61.35 62.54	19.1 0.56	1.33 1.20	5.33 0.27	4.1 0.15	4.87	1.04 0.25	0.5	0.69	2.2 35.0	7.18

Table 1. Chemical composition of the CPV-ARI MAX cement and lime used in this research.

^a $Na_2O_{eq} = Na_2O + 0.658(K_2O)$; ^b LOI: loss on ignition; ^c $C_3A = 2.650(Al_2O_3) - 1.692(Fe_2O_3)$.

The formulations cement-containing mortars, lime, and perlite tailings (PP and CP) investigated in this work were previously developed by our research group [27]. The mortar traces used were 1:3 (cement: 100% CP); 1:3 (cement: 85% CP + 15% PP); 1:1:6 (cement: lime: 100% CP), and 1:1:6 (cement: lime: 85% CP + 15% PP). During the preparation of the cementitious pastes, the workability was determined through the consistency index according to ASTM C1437 [28]. The water was added to the system until the consistency index reached 260 ± 10 mm.

2.2. Methods

2.2.1. Chemical, Thermal, and Mineralogical Characterization

The chemical composition was determined by X-ray fluorescence (EDX) in EDX 720 given by Shimadzu. The mineralogical characterization was performed by X-ray diffraction (XRD) in XRD6000 (Shimadzu) with radiation K α -Cu (40 kV/30 mA), a goniometer speed of 2 min⁻¹, and a step of 0.02°. The XRD data analysis was performed using the PDF database 02 of the International Center for Diffraction Data (ICDD[®]). The interpretation was performed by comparison with standards contained in PDF 02 (ICDD, 2003) [29,30].

SEM images were acquired from a cross section of mortar bars (10 mm \times 10 mm). A marble saw with a diamond disk was used to cut the samples. Before SEM images, a thin film (~50 nm) of gold was deposited at the cross section of the mortar bars. Such experiments were performed with the SANYU electron model SC-701, in which the experimental conditions were 2 min and 10 mA.

2.2.2. Resistance to Alkali–Aggregate Reaction

The resistance to the alkali–aggregate reaction was evaluated from linear expansion measurements performed on specimens with 25 mm \times 25 mm \times 285 mm dimensions, as determined by ASTM C1260 [31]. The samples were cured for 24 h in a humid chamber with 100% relative humidity and then immersed in water at a temperature of 80 \pm 2 °C for 24 h. After water immersion, the samples were measured with a digital caliper (initial reading) and then transferred to the 1 N NaOH, where they were kept at 80 \pm 2 °C for 28 days. The linear expansion measurements were obtained at intervals equal to 1, 7, 14, 16, and 28 days. After 28 days of curing, the samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermal gravimetric analysis (TGA).

The compressive strength (CS) was determined on cylindrical specimens (50 mm \times 100 mm), according to ASTM C39/C39M [32]. The strengths were determined in two steps: first for the specimens immersed after 28 days in distilled water at 80 °C \pm 2 °C and second for samples immersed in 1 N NaOH solution for 28 days at the same temperature. The results represented the average of three specimens and were determined in a universal mechanical testing machine (SHIMADZU AG-IS). The charge speed was 0.25 \pm 0.05 MPa/s.

3. Results and Discussion

The PP and CP tailings presented high contents of SiO₂ and Al₂O₃ (70% and 15%, respectively). Such SiO₂ and Al₂O₃ contents plus values of alkaline equivalents (Na₂O_{eq}) greater than 5% can trigger characteristic expansions of alkali–aggregate reactions (Table 2). The amorphous alkaline gel is the main product of such reactions and has high water absorption, which can cause expansion and consequent cracking of the cementitious materials [33–35]. Table 2 presents the chemical composition of the PP and CP tailings used in this

research. The PP tailings were used as filler to improve the packing of the system because they presented a maximum diameter (D_{max}) and fineness module (M_F) equal to 0.6 mm and 1.27, respectively (Figure 1). The D_{max} value measured from the CP tailings was 1.2 mm, and a fineness module was ~3.2; thus, it was used as the aggregate.

Table 2. Chemical composition of the perlite tailings investigated in this work.

Material	Oxides (%)										
	CaO	SiO ₂	MgO	Al_2O_3	Fe ₂ O ₃	SO ₃	K ₂ O	Na ₂ O	^a Na ₂ O _{eq}	^b LOI	
PP	1.5	73.0	0.6	15.3	1.1	0.1	3.3	3.0	5.2	2.1	
СР	1.2	71.8	0.6	15.4	0.9	0.1	3.2	3.5	5.6	3.3	

Cumulative retained mass (%) 0 D_{máx} = 0.6mm 20 M_F = 1.27 40 D_{máx} = 1.2mm 60 $M_{F} = 3.20$ 80 PP 100 1 0.1 10 Particle diameter (mm)

^a Na₂O_{eq} = %Na₂O + 0.658 %K₂O; ^b LOI: loss on ignition.

Figure 1. Particle size distribution of the PP and CP tailings.

Figure 2a,b show the linear expansion values measured from samples obtained from of the traces 1:3 (cement: 100% CP); 1:3 (cement: 85% CP and 15% PP); 1:1:6 (cement: lime: 100% CP), and 1:1:6 (cement: lime: 85% CP and 15% PP) immersed in a solution of 1 N NaOH for 28 days. For all samples, the linear expansion values measured at 16 days were above those previously seen in ASTM C 1260 [31], i.e., higher than 0.10%. The 1:3 (cement: 100% CP) and 1:3 (cement: 85% CP and 15% PP) samples presented linear expansions of 0.62% and 0.58%, while the 1:1:6 (cement: lime: 100% CP) and 1:1:6 (cement: lime: 85% CP and 15% PP) samples presented linear expansions of 0.62% and 0.58%, while the 1:1:6 (cement: lime: 100% CP) and 1:1:6 (cement: lime: 85% CP and 15% PP) samples presented linear expansions of 0.86% and 0.66%, respectively. These results indicate that traces containing only the CP aggregate presented higher linear expansion than those containing CP and PP tailings. There is a high probability that this linear expansion above the expected is related to the formation of amorphous alkaline gel synthesized from the alkali–silicate reactions, induced by the high contents of the SiO₂, Al₂O₃, and alkaline oxides (Na₂O and K₂O) detected in the CP and PP tailings.

Still, in Figure 2a,b, the relatively smaller linear expansion measured at samples contained 15% of PP tailings can be attributed to the filler effect. As PP tailings are a powdery material (see Figure 1), its addition to the trace amplifies the packing of the cementitious matrix, decreasing the permeability of the system and the probability of forming the amorphous alkaline gel from the solutions contained in the pores [36].



Figure 2. Linear expansions measured from the samples immersed in an aqueous solution of 1 N NaOH for 28 days: (a) traces 1:3 (cement: 100% CP) and 1:3 (cement: 85% CP +15% PP); (b) traces 1:1:6 (cement: lime: 100% CP) and 1:1:6 (cement: lime: 85% CP + 15% PP).

Figure 3 presents diffractograms measured from the perlite tailings (PP and CP) and samples after immersion in 1 N NaOH solution. The anorthoclase (JCPDS 09-0478) and quartz (JCPDS 46-1045) were the phases identified in the perlite tailings. The identification of aluminosilicate sodium and potassium phase (anorthoclase) favors the formation of the amorphous alkaline gel. It is known that such a gel is the main product of the alkali-aggregate reactions (RAAs). This is because, during immersion in an aggressive environment (1 N NaOH), the OH⁻ contained in pore solution comes into contact with the anorthoclase phase, make it soluble, and forms an amorphous alkaline gel. The amorphous band detected in the diffractograms of samples immersed in 1 N NaOH (Figure 3) is indicative of the formation of gels from the AAR [37]. Such a behavior is similar to that observed for silicates glasses, i.e., an amorphous band between 27° and 31° (2 θ) with a strong reflection in 28° [38,39]. Other phases rich in sodium and potassium were detected in samples immersed in 1 N NaOH, i.e., calcium silicate (JCPDF 29-0368), sodium oxide (JCPDF 06-0500), sodium iron sulfate (JCPDF 27-0718), P-portlandite (JCPDF 04-0733), silicon oxide phases (JCPDF 27-0605), iron sulfide (JCPDF 15-0037), and anorthoclase (JCPDS 09-0478). The SEM images inserted in Figure 3 ratified the formation of the gel mentioned above. The areas marked with ellipses indicate the amorphous silica gel formed inside the pores of the mortars investigated in this study. Similar results were reported by Munhoz et al. [26].

Table 3 presents the compressive strength (CS) values of the mortars after immersion in 1 N NaOH and distilled water solutions. As expected, the samples immersed in 1 N NaOH solutions presented lower CS values than those immersed in distilled water. The 1:1:6 (cement:lime:100% CP) sample was the exception because CS values measured from them were equal (considered the standard deviation) in both experimental conditions. This behavior agrees with the linear expansion, XRD, and MEV results that confirmed the presence of amorphous alkaline gels in the samples kept in an aggressive environment. Additionally, in both experimental conditions investigated, the sample contends PP tailings presented higher CS values than samples without them. As mentioned before, this behavior occurred because the PP tailings worked as a filler material, causing a decrease in its porosity and consequently better packaging of the trace. Although the mortars studied in this work present high linear expansion values (see Figure 2), that is, above those established by ASTM C 1260, the compressive strength results indicate that such mortars can be used for masonry according to ACI 530.1-02/ASCE 6-02/TMS 602-02 [40].



Figure 3. Diffractograms measured from as-received PP and CP tailings and mortars after the attack in 1 N NaOH solution. A—Anorthoclase; Q—Silicon oxide; S—Calcium silicate; X—Sodium oxide; F—Iron sulfide; O—Sodium iron sulfate; and P—Portlandite. SEM images were obtained from the mortars after attack in 1 N NaOH solution.

	Compression Strength (MPa)								
Trace	After Immersion in 1 N NaOH	Standard Deviation	After Immersion in Distilled Water	Standard Deviation					
1:3 (cement:100% CP)	9.69	± 0.7	13.04	±1.52					
1:3 (cement:85% CP + 15% PP)	12.12	± 1.1	17.07	± 1.13					
1:1:6 (cement:lime:100% CP)	5.80	± 0.30	5.70	± 0.13					
1:1:6 (cement:lime:85% CP +15% PP)	8.32	± 0.51	11.76	± 1.34					

Table 3. Compressive strength of mortars after 28 day of immersion in 1 N NaOH and lime water.

4. Conclusions

Powdered and coarse perlite tailings were incorporated into mortars containing cement CP V-ARI and calcite lime. All linear expansion values measured from samples immersed in the 1 N NaOH solution were above those established by ASTM C 1260, i.e., >0.10%. This behavior was credited to the amorphous alkaline gel, which is the product of the alkali–aggregate reaction. This gel absorbs water, expands, and cracks the cementitious matrix. Indeed, the high contents of SiO₂, Al₂O₃, and alkaline oxides (Na₂O and K₂O) detected in perlite tailings favored alkali–silicate reactions. The presence of the gel was confirmed by XRD and SEM experiments. The cracks caused by the swelling of the alkaline gel negatively influenced the compressive strength values, where samples immersed in 1 N NaOH solution had lower CS values than those immersed in distilled water. Despite the high linear expansion values, the measured CS values indicate that the samples in this work can still be used in masonry, according to ACI 530.1 -02/ASCE 6-02/TMS 602-02.

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