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Evaluation of fire, high-temperature and water erosion resistance of fiber-reinforced lightweight pozzolana-based geopolymer mortars

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Abstract. The development of new building materials requires intensive analyses of their durability under aggressive conditions. Therefore, the current study evaluated the capacity of a fiber-reinforced lightweight pozzolana-based geopolymer mortar to resist the action of fire, high temperatures and water erosion. The fire resistance and heat absorption capacity were evaluated through the exposure of disc samples to a direct flame at 1000 °C and the measurement of reverse-side temperature, respectively. To evaluate the high-temperature resistance, uniaxial compression tests were performed on cubic samples after their exposure to oven-temperatures of 300°C, 500°C, 800°C, and 1000°C. Finally, water jet erosion and permeability tests were performed to analyze the interaction of the material with water. The results of the fire tests indicate that fiber-reinforced lightweight pozzolana-based geopolymer mortars are able to absorb up to approximately 65% of heat flame with only slight degradation. On the other hand, uniaxial compression tests showed a temperature resistance up to 800°C, above this temperature the cubic samples experimented significant volumetric changes that might have been caused by a reaction of remaining hydrogen peroxide or gas expansion. Finally, water exposure tests showed that the material is highly permeable and has a remarkable resistance to water erosion.

1. Introduction

The ordinary Portland cement (OPC)-based concrete is one of the most used materials in the construction industry with worldwide average consumption of 3 tons per person each year [1]. The advantage of this building material is its versatility: it is used to produce a great number of elements for different applications and structures with many colors, densities, strengths and durability capacities. Also, in order to reduce the dead load of the structures and be easily manageable, low-density OPC-based concretes are developed by the addition of lightweight aggregates or by using foaming agents that increase their volume [2-4]. However, OPC production is one of the major contaminant industrial activities, due to its fabrication represents 5-7% of carbon dioxide emissions



worldwide [2, 5-7]. For this reason, there has been greater development of new alternative materials to reduce the use of OPC leading to a more environmentally friendly construction industry.

Recent researches show that geopolymers are materials with similar properties in terms of mechanical properties and durability to OPC-based materials but with lower contaminant emissions in their production [6, 8, 9]. Geopolymers are obtained from the reaction between aluminosilicate sources and an alkaline activating solution. The most used aluminosilicate sources are industrial by-products and non-metallic minerals as fly ash and metakaolin and their mix with other waste materials [9]. In Peru, there is an abundant presence of natural pozzolana, a non-metallic mineral with high silica and alumina content that is extracted from volcanic ash deposits. In this context, a recent article has shown the possibility of using this material to produce high-strength geopolymers using natural pozzolana [11]. To produce lightweight geopolymer mortars a foaming agent was added to the mixture reducing the density. The presence of a foaming agent increases the volume of the material and generates a porous structure, known for its high degree of heat and water absorption [12, 13]. Additionally, 10-mm single jute fibers were added to the mixture to control shrinkage cracking problems. This article focuses on an evaluation of the durability of a fiber-reinforced light weight pozzolana-based geopolymer mortar under aggressive conditions by analyzing the effect of the action of fire, high temperatures and water erosion.

2. Materials and methods

2.1. Raw Materials

The materials used in this study consisted of a natural pozzolana and granulated quartz M40 as a precursor of the geopolymer and fine aggregate respectively, both materials provided by Compañía Minera Agregados Calcareos S.A (COMACSA). The natural pozzolana was extracted from Arequipa, Peru, while the granulated quartz M40 is a commercial product of the company. Particular size analyzer SediGraph 5100 was used to determine the particular size of both materials. The particle size analysis indicated that the natural pozzolana presented a d50 of 10.2 μm while the granular quartz M40 a d50 of 480.8 μm . The oxide composition of both materials is shown in Table 1.

Table 1. Oxide composition of natural pozzolan and granular quartz M40

Raw Material	Oxide							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	MgO	CaO	Na ₂ O	Others
Natural Pozzolan	53.55	10.81	0.98	4.07	0.55	1.43	2.30	26.31
Granulated Quartz M40	97.80	0.77	0.02	-	-	-	-	1.41

2.2. Alkaline solution and foaming agent

The alkaline solution used in this study was prepared mixing sodium silicate (28% SiO₂ + 8% Na₂O + 64% H₂O, technical grade, Abastecimientos Químicos Ciatex S.A.C.), sodium hydroxide in pearls (99.27% purity, technical grade, New China Chemicals Co., Ltd.) and distilled water. The dosage used in the preparation of the alkaline solution was SiO₂/Na₂O molar ratio of 1.08, Na₂O content of 8% and a water-binder ratio of 0.52. The foaming agent used was hydrogen peroxide (H₂O₂, 30% concentration, Movilab del Perú S.A.C.) with a content of 0.5% respect to the weight of the alkaline solution.

2.3. Jute Fiber

Jute is a plant fiber (*Corchoruscapsularis*, *Corchorusolitorius*) and mainly consists of cellulose (61-71%), hemicellulose (13.6-20.4%) and lignin (12-13%) [14]. Its usage in the geopolymer composite was decided based on its environmental benefits as Food and Agriculture Organization of the United

Nations (FAO) considers it as future fiber [15]. The jute fiber was obtained from commercial ropes and cuts to a length of approximately 10 mm.

2.4. Methods

The production of the fiber-reinforced lightweight pozzolan-based geopolymer mortar started by dry mixing pozzolana, fine aggregate (pozzolana to fine aggregate weight ratio of 2) and jute fibers (fiber content of 1% by weight of the pozzolana) for 1 min. Later, 95% of the alkaline solution was poured into the dry mixture and mixed for 3 min. The remaining 5% of the alkaline solution was combined with the hydrogen peroxide, then it was added into the previous mixture and mixed for another 1.5 min. Afterward, the lightweight mortar was placed into the corresponding molds for each test. Finally, they were cured in an oven at 65°C for 7 days.

First, the fire resistance was evaluated by the method proposed by Cheng and Chiu [16]. This test was performed by applying a flame at 1000°C (Figure 1a) on the surface of a geopolymer disc of 100 mm diameter and 10 mm thickness for 30 min (Figure 1b) and measuring the temperature on the opposite side (reverse-side temperature). The temperature was measured by BTMETER BT-1500 Pyrometer 30:1 Laser Thermometer Gun with a precision of 0.1°C. Temperature measurement was performed until it reached room temperature (22 °C). The reverse-side temperature is an indicator of the degree of heat absorption capacity. The evaluation of this test was performed on three samples in two stages: Stage I evaluated disc heating while Stage II evaluated the cooling phase.

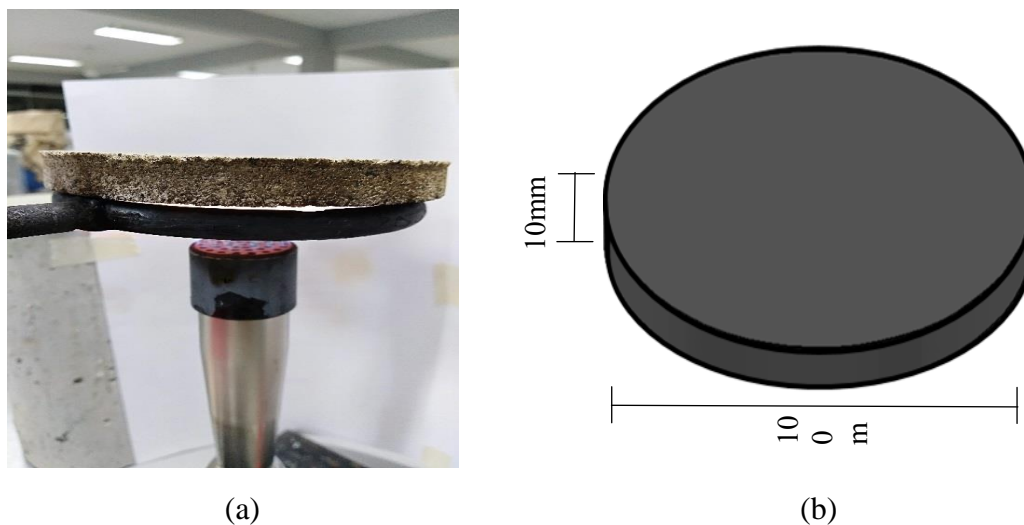


Figure 1. Fire resistance test: (a) Test; (b) Disc dimensions.

The high-temperature resistance was measured by the method proposed by Ranjbar et al. [17] and RILEMTC 129-MHT [18]. Cubes samples (five per each temperature) were placed in an oven at 300, 500, 800 and 1000° C using a heating rate of 10°C per minute. After that, uniaxial compressive strength was determined following the guidelines of ASTM C109 [19], and the bulk density was measured using the Archimedes method. The results of the bulk density and compressive strength of heated samples were compared to the ones exhibited by samples left at room temperature (22 °C).

Water erosion was studied based on permeability [20] and water jet erosion tests [21]. The permeability was analyzed by measuring the contact angle using a Ramé-hart contact angle telescope-goniometer (Figure 2a) [22]. The test consisted of placing a drop of 10 µL of water on the surface of the fiber-reinforced lightweight geopolymer mortar samples (50 x 25x 10 mm prisms) and measuring the contact angle (Figure 2b). Low and high impermeability materials are classified based on the contact angle: permeable if the contact angle is less than 90° and impermeable if the contact angle is

greater than 90° . On the other hand, the water jet erosion test was performed following the methodology proposed by Benites et al. [21]. The test consisted of applying a water jet with a pressure of 200 psi from a distance of 300 mm (Fig. 2c) on one surface of a fiber-reinforced lightweight geopolymer mortar sample (300 x 200 x 100 mm plates) during 1, 5, 10 and 15 min. The surface condition after the water jet indicates the level of erosion caused by water exposure at high pressures.

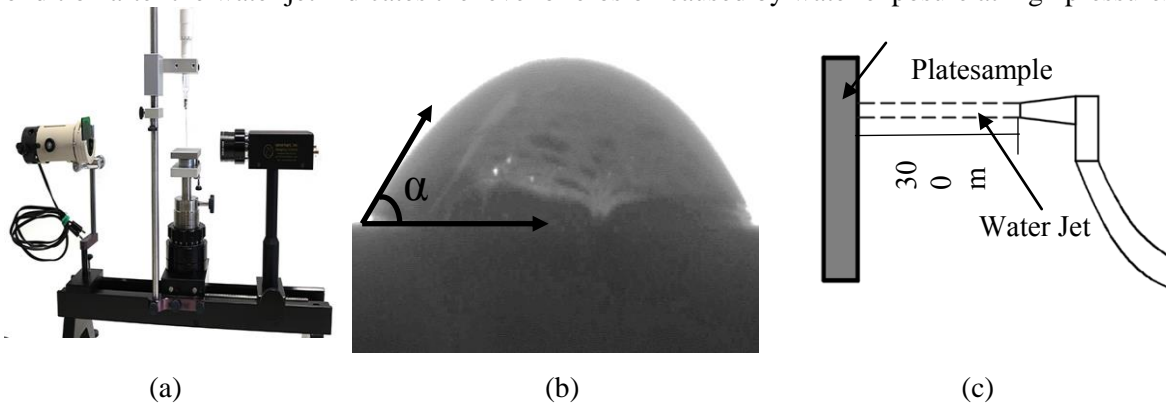


Figure 2. Water erosion test: (a) Ramé-hart contact angle telescope-goniometer; (b) Contact angle measurement; (c) Scheme of water jet test.

3. Results and discussion

3.1. Fire Resistance

Figure 3 shows the reverse-side temperature record during the fire resistance tests. At the beginning of Stage I, the material underwent a rapid change in its temperature until it reached its maximum temperature (less than 350°C) and remained constant until the flame was extinguished. This significant retention of heat transmission can be explained because the unconnected pores trap the hot air avoiding the heat flow from the flame to the reverse-side surface. While in Stage II, the record of the reverse-side temperature indicates that the disc has a slight delay in cooling compared to heating. Nonetheless, cooling phase temperature has a greater uniformity than heating and the data fits an exponential decay of the temperature.

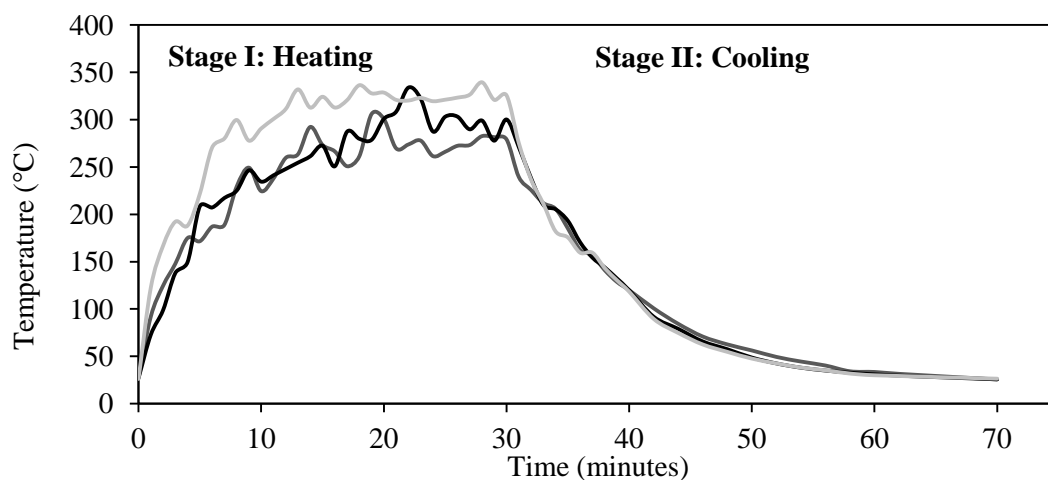


Figure 3. Reverse-side temperature vs. time from fire resistance tests.

As shown in Figure 4, the discs subjected to the action of fire retained their shape without noticeable deformations. However, there is a change of color to a darker one compared to the initial sample. This change of color was due to that the surface had a baking action by the effect of contact with fire. Another fact that can be deduced from the image is that the fibers added in the production still present in the sample, showing that the lightweight geopolymer acted as insulating protection to the action of fire.



Figure 4. Fiber-reinforced lightweight geopolymer disc: (a) Before test; (b) After test.

3.2. High-temperature resistance

Figure 5 shows the specimens subjected to high temperatures. A change to a darker color was seen as the temperature increased with no significant volumetric changes up to a temperature of 800 °C. This change of color was due to the fact that the surfaces of the samples suffered a baking action by irradiated heat in the oven. This demonstrates that the fiber-reinforced lightweight-based geopolymer mortar is not severely affected by long-term exposure at temperatures up to 800 °C. However, in the case of specimens exposed to 1000°C, a significant loss of the initial shape was observed. Figure 5e shows a cross-section view of a cube sample exposed to an oven-temperature of 1000°C. An increase in the size of the pores caused by a new expansion process is observed.

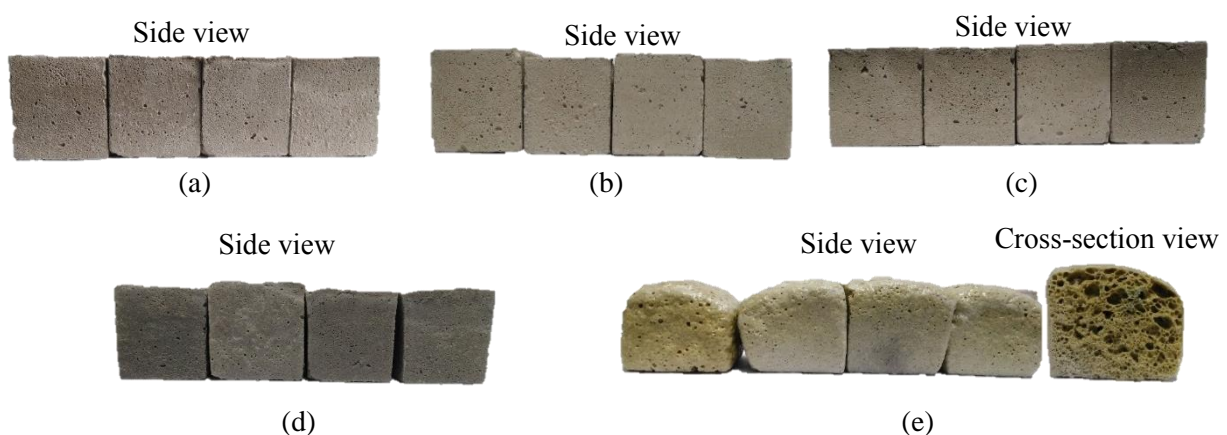


Figure 5. Cubes exposed to high temperatures: (a) Ambient temperature; (b) 300°C; (c) 500°C; (d) 800°C; (e) 1000°C.

The mechanical and physical characterization of the cubes is shown in Figure 6. The results of the uniaxial compression tests (Figure 6a) show that the compressive strength of the fiber-reinforced lightweight geopolymer mortar does not change significantly up to 800 °C, considering the standard deviations. However, remarkable lower resistance to compression loads was obtained in samples exposed to 1000°C (4.65 MPa). On the other hand, it can be seen in Figure 6b that the density of the samples remains constant until a temperature of 500°C while the lowest density (0.66 g/cm³) was recorded again at 1000°C. Similarly to qualitative analysis, mechanical and physical evaluation indicate that the fiber-reinforced lightweight geopolymer mortar is stable until 800°C. The reason why density does not dramatically change up to 800°C can be explained because there is no additional release of free water from the sample since it was released during the long curing period (7 days at 65°C). It is appreciable that the dramatic volumetric changes due to pore expansion results in a significant decreased in both compressive strength and density at 1000 °C

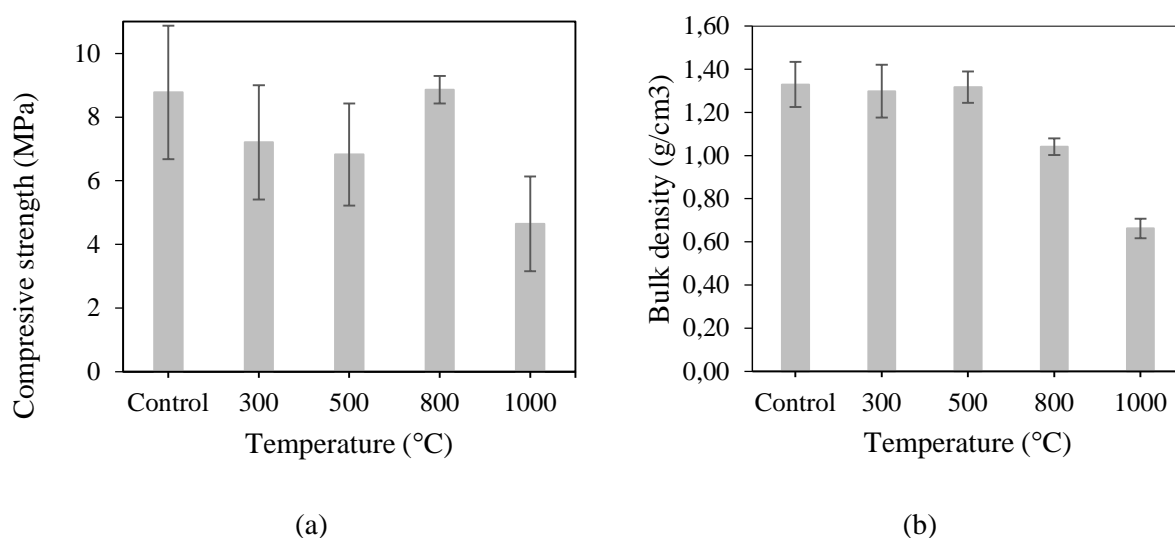


Figure 6. Mechanical and physical characterization of fiber-reinforced light weight geopolymer mortar exposed to high temperatures: (a) Compressive strength; (b) Bulk density.

3.3. Water erosion tests

The contact angle tests indicated a contact angle of $43.31^\circ \pm 9.06^\circ$. Therefore, the fiber-reinforced lightweight pozzolana-based geopolymer was classified as a material with low impermeability. In addition, it could be observed that the drops of water used for the test were absorbed in 3 seconds on average, demonstrating the high absorption rate of the material. Low impermeability is a well-known property of porous materials that tend to wet easily compared to denser ones.

Figure 7 a and b show the surfaces of the samples before and after the water jet erosion test, respectively. The pressurized water jet did not cause penetration into the surface of the specimens. Nonetheless, there is a slight erosion on the surface caused by the pressure of the jet and splashing water. The marks have a diameter of approximately 1.5 cm. Although it was proved in the previous test that the fiber-reinforced light weight geopolymer mortar is highly permeable, this material demonstrated remarkable resistance to water erosion.

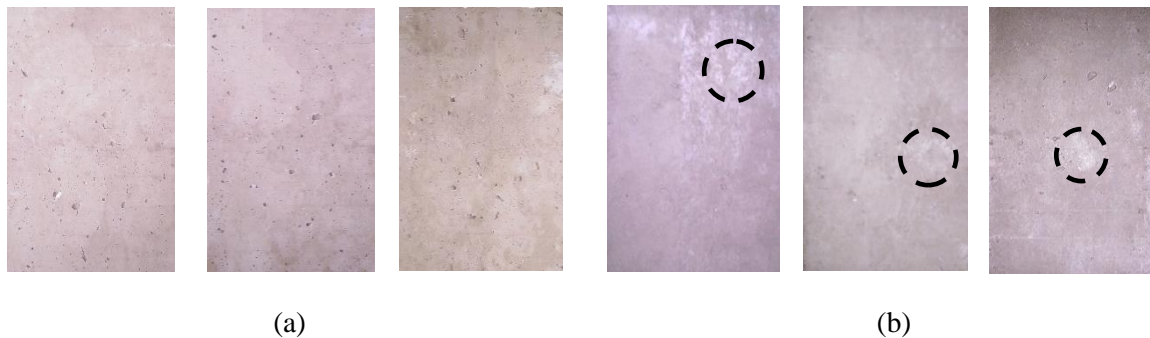


Figure 7. Sample surfaces subjected to water jet erosion tests: (a) Before testing; (b) After testing.

4. Conclusions

This research focused on the effect on physical and mechanical characteristics caused by the exposure to aggressive conditions of specimens produced with a fiber-reinforced lightweight pozzolana-based geopolymer mortar. The fire-resistance test demonstrated that exist a considerable heat absorption capacity of the fiber-reinforced light weight pozzolana-based geopolymer mortar. The temperature recorded on the reverse-side surface did not exceed 350°C, which would be equivalent to a heat retention capacity of more than 65% of the heat irradiated by the flame. It was observed that the specimens resisted without a major change in shape with only a slight change of color to a darker one. In addition, the results also indicated appropriate insulating conditions to the layers inside the element. The exposure to high temperatures indicated that the specimens that were exposed to temperatures of 300°C and 500°C did not evidence significant changes in both compressive strength and density. However, at temperatures of 800°C, the density dropped significantly, but the compressive strength and shape did not significantly change. The most significant change occurred at a temperature of 1000°C due to a new process of pore expansion, the initial shape is lost and both properties, density and compressive strength, decrease considerably. Finally, fiber-reinforced lightweight pozzolana-based geopolymer mortar demonstrated to be highly permeable based on contact angle tests. However, the waterjet test showed great erosion resistance of the material to pressurized water with no suffering significant surface damage.

Acknowledgments

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