Original Article

Mechanical and microstructural characterization of geopolymeric concrete subjected to fatigue

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Cements from alkali-activated reactions are unleashing opportunities for the future application of inorganic polymers, known as geopolymers. These materials are eco-friendly, since their manufacturing process does not involve carbon dioxide (CO₂) emission and it makes possible the use of industrial waste as raw material. In this work, a geopolymeric cement concrete (GCC) was developed through adequate portions of geopolymer components. Its characteristics were compared with Portland cement concrete (PCC), through the establishment of some parameters of design such as consumption of binders, water/aggregates ratio and cement content. The mechanical performance of these concretes was evaluated with emphasis on the fatigue behavior. The results showed a better fatigue performance of CCG in comparison with PCC in several parameters. Better matrix/aggregate adhesion in the CCG in comparison with PCC was also observed in the microstructural analysis, which may explain its superior fatigue performance.

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1. Introduction

Geopolymerization is a relatively recent technology applied to fabricate a cement material, which became a viable alternative for the ordinary Portland cement [1–3]. In civil construction, this technology relies on the chemical reaction of amorphous silica and alumina-rich solids in a highly alkaline solution at ambient or slightly elevated temperatures to form an aluminosilicate inorganic polymer known as geopolymer [4]. In the fabrication of a geopolymeric cement, aluminosilicate compounds are partially dissolved in alkaline, NaOH or KOH, solutions. After reaction and drying, an amorphous matrix is formed containing non-reacted crystalline particles. The Al/Si ratio is the main variable in the geopolymerization process [2]. By contrast, the Portland cement is fabricated by high temperature (~1400 °C) calcination of a mixture of clay and lime-bearing minerals to produce a clinker. A small amount of
gypsum (CaSO₄·2H₂O) is added to retard the setting process. The clinker is supplied as ground fine powder, which needs to be mixed with water for construction use. Different than geopolymer cement, Portland cement depends on water inside is microstructure for integrity. The technical performance of a geopolymer is comparable to that of Portland cement in the fabrication of concrete. However, geopolymer has additional advantages, such as rapid development of mechanical resistance and superior durability. Moreover, it is associated with sustainable development owing to significant reduction in energy consumption and lower greenhouse gas emissions [4]. Due to the absence of water in its microstructure, another advantage of geopolymer cement over Portland cement would be a higher stability at elevated temperatures [5].

Concretes made with geopolymer cement also display advantages in terms of mechanical performance and temperature resistance in comparison with Portland cement concretes [6]. Indeed, in Portland cement concretes the decomposition of Ca(OH)₂ around 500 °C causes significant damage. On the contrary, in high temperature applications, geopolymer concretes present comparatively superior performance up to 800 °C [6]. Despite the existence of comparative works on the mechanical performance of Portland cement and geopolymer concretes [5,6], the fatigue behavior has not yet been compared. Indeed, fatigue is a relevant problem in Portland cement concretes that tend to develop microcracks, anticipating rupture with practically no plastic deformation [7,8]. In the case of concretes, the damage caused by repeated load cycles can be modeled to evaluate the fatigue resistance [8]. This model predicts that a cumulative damage generated by cycling load is determined by the equation proposed by Miner [9]:

\[ D = \sum \left( \frac{n}{N} \right) = 1 \]  

(1)

where \( D \) is the cumulative fraction of damage, \( n \) the number of applied load cycles at a certain stress level and \( N \) the total number of cycles that is expected to cause rupture at that stress level. Based on this model, the present work compared the mechanical behavior and developed microstructure of a geopolymer with those of Portland cement concretes.

### 2. Materials and methods

The synthesis of geopolymer cement was performed with three basic materials: silica (SiO₂), obtained from sodium silicate; potassium hydroxide (KOH), commercially available as alkaline source; and calcium oxide, from blast furnace slag. Following standard chemical reactions [10], a geopolymer with expected characteristics was obtained. The Portland cement was a commercially supplied high strength type CPV-ARI-RS, from Holcim, Brazil. Both geopolymer and Portland cements were used to fabricate concretes with similar amounts of ~4 mm quartzite sand and ~12 mm pebbles. Sand and pebbles were washed in running water and sieved to eliminate particle sizes lower than 1 mm. The concrete weight composition was: 20% cement, 37% sand, 35% pebbles and 8% water.

Prismatic concrete specimens with \( 400 \times 100 \times 100 \text{mm}^3 \) were prepared by mixing the components inside molds placed for 15 s on a vibrating table. Both geopolymer cement concrete (GCC) and Portland cement concrete (PCC) specimens were kept inside the molds to gain enough consistency for a period of 48 h. Flexural tests were conducted according to the Brazilian standards [11] in a MTS (Material Test System) equipment at room temperature (RT). Ten specimens were three-points bend tested for flexural resistance of each type of concrete investigated. A conventional statistical analysis (mean value ± standard deviation) was performed. Fatigue tests were conducted in a servo-hydraulic MTS machine with 10 ton of capacity operating with variable frequency, 1 to 10 Hz, at RT depending on the type of fatigue test. “Maximum Constant Stress” tests were performed at 10 Hz to determine the number of cycles, \( N \) in Eq. (1), up to rupture. In this case, specimens were subjected to a stress corresponding to fatigue stress ratio (FSR) of 0.7, 0.75, 0.8 and 0.85. “Variation of Frequency” tests were carried out with same FSR value to a total of \( N \) cycles necessary for rupture under 1 and 5 Hz. “Maximum Variable Stress” tests were also performed at 10 Hz to determine \( N \) in fatigue tests with variable increasing and decreasing FSR. For each FSR of both GCC and PCC, five specimens were fatigue tested and conventional statistical analysis was performed.

After rupture the specimens fractured surface was analyzed by scanning electron microscopy (SEM) in a model JSM 5800-LV Jeol microscope operating with secondary electrons at 20 or 25 kV. All SEM samples were gold sputtered before observation.

### 3. Results and discussion

The average flexural strengths (maximum stress) obtained in three-points bending tests [11] for both concretes made of GCC and PCC are presented in Table 1. The values in this table, within the standard deviation, indicate that both concretes, GCC and PCC, display similar flexural strength. These values served as references for the fatigue tests.

<table>
<thead>
<tr>
<th>Type of Concrete</th>
<th>PCC</th>
<th>GCC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural Strength (MPa)</td>
<td>5.4 ± 0.8</td>
<td>5.9 ± 0.3</td>
</tr>
</tbody>
</table>

Figure 1 – Typical flexural load vs. deflection for the investigate concretes.
A typical variation of the applied load with deflection during corresponding flexural tests is shown in Fig. 1. In this figure, it should be noticed that up to a load of 3 kN, both PCC and GCC have the same behavior given by the perfectly superimposed curves. This might be associated with an initial linear elastic stage, which has the same modulus (~27 GPa) for both concretes. Beyond this limit, the GCC deviates from the common straight line, more accentuated (inclined) than the PCC. The final rupture of GCC occurred not only at higher load, corresponding to the apparently higher stress in Table 1, but also with higher deflection than PCC. In practice, this suggests that concretes made of geopolymer cement are stronger and more deformable that are desirable characteristics for civil construction, especially road paving.

Fig. 2 shows the variation of the number of cycles to rupture (N) as a function of the ratio between fatigue applied stress (FSR) to the maximum stress. As expected, one sees in Fig. 2 that the value of N markedly decreases for both types of concrete with increasing FSR. Within the standard deviation, both PCC and GCC show similar fatigue behavior in Fig. 2 with slightly superior mean N values for the concrete made with geopolymer cement. This apparent superior fatigue behavior of GCC can only be claimed with a low degree of statistical relevance. For practical purpose, based only on the results of Fig. 2, both investigated concretes present similar fatigue behavior. The effect of cycling frequency, however, permits a more precise comparison.

The effect of cycling frequency was evaluated for FSR = 0.75, which is a condition in Fig. 2 for the greatest difference between the number of cycles to rupture in both concretes. Thus, Fig. 3 depicts the change in numbers of cycles to rupture (N) with load cycling frequency for 0.75 ratio. This figure indicates that above 5 Hz there is a significant increase in N with a tendency of greater fatigue resistance for the GCC. In practice, the results in Fig. 3 reveal that high frequency fatigue conditions, such as those that may occur in industrial constructions with vibrating equipments and pavements with heavy traffic, favor GCC over PCC.

The fatigue tests with variable stress also revealed an advantageous condition for the GCC. Fig. 4 shows the cycles to rupture (N) for FSR = 0.80, corresponding to three distinct conditions. Condition (1) of constant applied fatigued stress, which is the case shown in Fig. 1 for 0.80 ratio. It revealed practically no difference between values of N for the GCC and PCC. Condition (2) for decreasing applied fatigue stress, FSR varying from 0.85 to 0.70, also disclosed no difference. Condition (3) for increasing applied fatigue stress, FSR varying from 0.70 to 0.85, disclosed a remarkable difference in favor of GCC. Indeed, its value around N = 9000 cycles is almost twice that for PCC.

The reason why the fatigue behavior of concrete under increasing applied stress is superior than under decreasing stress, Fig. 4, is still not clear. In principle, conditions (1) and (2) are similar and support the fatigue model of Eq. (1). Condition (3), however, demands a new model for its explanation. This is now being investigated with particular emphasis on the superior performance shown by the geopolymeric cement.

The SEM fractographs contribute to the comprehension of the possible reason for the superior performance of GCC over PCC. Fig. 5 shows the typical microstructural aspect of both concretes used to fabricate the investigated GCC ab PCC concretes. The geopolymer cement, Fig. 5(a), displays a relatively more uniform microstructure with less small pores than the Portland cement in Fig. 5(b). Both
microstructures, however, show large pores that might be associated with microcracks. In spite of these existing microcracks that could equally affect the fatigue performance, the more homogeneous microstructure of the geopolymer cement, as concrete matrix, would contribute to better fatigue behavior.

Fig. 6 illustrates the difference in the fractographs of GCC and PCC, both ruptured at a constant FSR = 0.75 and 10 Hz for a total N around 5000 cycles. In particular, it should be paid attention to the interface between the sand and the cement matrix. In Fig. 6(a) for GCC, the fracture surface was apparently due to a crack breaking simultaneously through sand and cement without affecting their interface. On the contrary, for PCC in Fig. 6(b), cracks are seen at the sand/cement interface in association with small particles. These particles were found by EDS to be portlandite crystals that are inherent of Portland cement. The absence of portlandite, which is a brittle phase, in GCC might explain the superior adherence between the geopolymer cementitious matrix and the concrete aggregates.

As a final remark, the results in Figs. 2-6 disclosed a fatigue performance of GCC, which is practically similar to PCC for constant and decreasing applied stresses as well as for cycling frequency lower than 10 Hz. On the other hand, an increasing applies stress during fatigue results in markedly higher performance of GCC in terms of number of cycles to rupture. The absence of a brittle phase portlandite contributes to the GCC improved fatigue life and greater total deformation as compared to ordinary concrete made of Portland cement. In principle, this indicates an advantage of using GCC as pavement for heavy traffic.

4. Conclusions

- Geopolymer cement concrete (GCC) has, in quasi-static load conditions, a tendency to be stronger and more deformable (less brittle) than ordinary Portland cement concrete (PCC).
- Under load cycling fatigue condition, both GCC and PCC display similar performance at constant and decreasing applied stress. A tendency to superior number of cycles to rupture (N) occurs above cycling frequency of 5 Hz.
- Under increasing applied fatigue stress, GCC performs better than PCC with markedly higher values of N. The more homogeneous geopolymer cement as well as the absence of brittle particles of portlandite in the interface between the geopolymer cement and aggregates apparently contributes to the improved performance of GCC. In practice, this superior fatigue performance of geopolymer cement-based concretes might be an advantage over ordinary Portland cement concrete in vibrating civil constructions, such as heavy traffic pavements.

Conflicts of interest

The authors declare no conflicts of interest.
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