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Thermo-mechanical properties of fibre reinforced cement-based foam exposed to sulphate

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HIGHLIGHTS

- Thermal conductivity of cement-based foams was evaluated after exposure to sulphate solution.
- Exposure to sulphates leads to a densification of cellular structure due to the formation of expansive products.
- An initial self-sealing of cells leads to a rise in thermal conductivity, followed by a drop upon expansive cracking.
- Cell closure, crack formation and their subsequent sealing is manifested upon toughness factor and thermal conductivity.

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ABSTRACT

This paper presents the effect of sulphate exposure on the thermal conductivity of cement-based foams and examines their correlation with certain mechanical properties. The study was conducted on cement-based foams with a cast density of 475 kg/m^3. Along with a plain mix, another series with polypropylene fibre at 0.2% volume fraction was prepared to investigate the role of flexural toughness on thermal conductivity. The tests were conducted on prisms of cement-based foams immersed in a sodium sulphate solution after specific intervals of exposure. Whereas the flexural toughness was measured based on standard four-point bending tests, a transient plane source (TPS) thermal analyzer was employed to measure thermal conductivity. The microstructure of the exposed specimen was analyzed with the help of scanning electron micrograph and the phases were identified using X-ray diffraction. The results reveal that thermal conductivity increases over the initial stage of sulphate immersion and subsequently drops to a plateau value. This rise-and-fall was mimicked by the flexural toughness factor.

1. Introduction

Cement-based foam comprises of Portland cement, water and foam. The foam may be introduced either by adding surfactants to the mix constituents or by the addition of a preformed foam to the starting mix slurry. The density of this composite may range from 300 to 1600 kg/m^3. Cast density not exceeding 800 kg/m^3 is used in non-structural application such as engineered void fill and thermal insulation. These low density composites are typically produced without using any fine aggregates. While certain lightweight fine aggregates may be introduced, it is commonplace to use supplementary cementing admixtures [1]. With a cast density below 800 kg/m^3 and specified compressive strength less than 8.3 MPa, the cement-based system examined here is classified as a low-density controlled low-strength material (LD-CLSM) [2]. The use of microfibre reinforcement, typically made of polypropylene or glass, helps control the shrinkage cracking in cement-based foams. Their efficiency depends upon the fibre type and the internal air-void network [3]. As cement-based materials contain calcium hydroxide and alumina-based products that are prone to attack by sulphate ions [4], the expansive nature of newly formed sulphate-bearing crystals in the presence of a sulphate rich agent is a cause for concern especially given the ease of ingress in this cellular solid. Although LD-CLSM mixtures are not usually designed for performance in aggressive chemical environments, given that cement-based foams are used in geothermal insulating application across Canada, the behaviour of this material under sulphate exposure merits close scrutiny. Little is known about the behaviour of high-density cellular concrete in sulphate rich environments [3,5]. Conspicuously, there is no data regarding the role of fibres. An earlier report by the authors showed that sulphate exposure led to a closure of the cells and strengthened the composite, especially for early durations of exposure [6]. A recent study by the

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authors on cement-based foams with high sulphate resistant cement showed an increase in thermal conductivity, upon exposure to sulphate solution, followed by a nearly constant thermal conductivity with continued exposure [7]. However, specimens with relatively higher density showed a slight reduction following an increased observation of thermal conductivity. Further, a considerably lower thermal conductivity was observed when Portland cement was replaced partially with supplementary cementitious admixtures such as fly ash.

Cement-based foam is a highly porous material with a minimum porosity of approximately 40% for a cast density of 1500 kg/m^3 [8]. Considering its cellular microstructure, the composite may reasonably be assumed as a two phase system consisting of solid cellular walls made of cement-paste and cellular voids filled with fluid. Factors that affect the thermal conductivity of such cellular systems include thermal conductivities, volumetric fractions, and distribution of these two phases [9]. The size of the cellular voids is known to influence on the thermal characteristics of these materials [10]. The irregularity of microstructure together with the tortuosity of cell wall renders a complicated heat transfer mechanism through typical cement-based porous media. The effective thermal conductivity is dependent on several contributing factors – thermal conduction through solid and fluid phase, convection within cellular boundaries, and radiation through the whole media [11]. Of these, it is thermal conduction which is the dominating mechanism in cement-based foams, based on the typical cell size and the ambient temperature [12].

In Canada, cement-based foams are used as insulation in geostructural applications and therefore, the emphasis is on achieving low thermal conductivity at a given density and strength. Whereas the mechanical performance of cement-based composites is adversely altered by the exposure to a sulphate rich environment, a preceding study by the authors showed that to the contrary, sulphate attack produces a self-healing effect on low-density cement-based foams, and in fact enhances their mechanical performance [6]. This was most prominent at lower densities. Note that such mixes are primarily used for their application in thermal insulation. However, the self-healing was seen to alter the cellular structure within the composite and so was presumed to conflict with its desirable thermal properties. The present study examines changes to the thermal conductivity in low-density cement-based foams as a result of sulphate exposure and the attendant consequence to the solid phase composition and integrity of the composite. Tests were conducted on specimens extracted after increasing periods of exposure up to 90 days in a sulphate bath. Mechanical properties, in particular the flexural toughness factor was evaluated to capture changes to the microstructure, specifically those arising from the expansive products after sulphate exposure. The effects on thermal conductivity were further explained through microscopic observation and X-ray diffraction.

2. Experimental details

2.1. Materials and specimen preparation

In order to produce the low-density cement-based foams, a cast density of 475 kg/m^3 was chosen in this program. It represents the material most common in geostructural application across Canada. The specimens were cast by means of the mixing-foam method whereby a preformed and stable foam was added to the slurry made with cementitious materials and water. Type HE (High Early Strength) cement conforming to CSA A3000 [13] was used along with Class C fly ash, the latter included as a pozzolanic admixture at 20% cement replacement by mass. The fly ash conformed to ASTM C618 [14]. Note that Type HE was chosen so as to achieve quicker strength gain, again a common practice during the production of cement-based foams in Canada. Fly ash is known to reduce autogenous shrinkage [15] in concrete as well lower the thermal conductivity [7]. In cement-based foams, an optimal water-to-cementitious material ratio (w/cm) is desired. If the w/cm is too low, it leads to the instability of the foam because of the binder's tendency to draw moisture away from the air bubble. Thus, after considering the water demand due to the high early strength cement and foaming agent, a w/cm of 0.53 was used in this program. The water content so chosen was sufficient to render the cement-based foams workable even in the presence of micro-fibres. The detailed mix proportion is presented in Table 1. The cement–water slurry was tested for flow properties in order to ensure its free flowing and self-levelling abilities. Using a Marsh Cone test for the slurry, it was found that the Marsh Cone flow values depend upon the desired cast density of the cement-based foam [16]. Using a base mix of 350 mL, it was found that a Marsh Cone flow value equal to 45 s resulted in a target cast density of 475 kg/m^3. A locally available synthetic foaming agent was used to prepare the foam to be mixed with the cementitious slurry. The foaming agent conformed to ASTM C869 [17]. Table 1 lists the mix proportions used in this study. A predominantly closed cell structure was obtained using this particular foaming agent. The foam was prepared with a foam generator activated by a 0.70 MPa compressed air source, which was calibrated to achieve a stable bubble structure. A second series of cement-based foams was prepared by adding polypropylene microfibre at 0.2% volume fraction, usually associated with shrinkage crack control [18]. The polypropylene micro-fibres were 20 mm long and of 0.33 Tex. They were added to the base slurry before the addition of foam. The fibres had an ultimate tensile strength of 550 MPa, an elastic modulus of 3.5 GPa and a specific gravity of 0.91. The preformed foam was then added gradually to the slurry. The density of the composite was checked intermittently and the addition of foam was continued as necessary until reaching the desired cast density. The mixing time was carefully monitored so as to avoid the loss of internal air-void structure. The amount of foam required for a certain cast density was very close to the amount that was calculated during mix proportioning, which indicates the stability of foam structure despite the rigor of mixing. The specimen was cast in metal moulds without the use of any external vibrator.

As mentioned earlier, the mechanical performance was assessed through flexural tests. The mixes were cast into prisms of dimension 150 mm × 150 mm × 900 mm. From this, prisms with a dimension of 50 mm × 50 mm × 200 mm were sawn to be tested in flexure. As the plain and fibre reinforced beams split into two upon failure at the end of the flexural test, the resulting two halves from each beam were retrieved for extraction of further specimens. Later, cubes of 50 mm side were extracted as per ASTM C42 [19] for compression tests. Prisms with a dimension of 50 mm × 50 mm × 12 mm were also salvaged from flexural test specimens for thermal conductivity test.

2.2. Sulphate bath

The deterioration in cement-based composites due to sulphate attack is usually evaluated by studying the change in physical dimension of the prismatic specimen as specified in standard test method such as ASTM C452 [20] for internal sulphate source and ASTM C1012 [21] for external sulphate source. In the present case regarding cement-based foams, it was noted that most sulphate related distress is due to the transportation of sulphate bearing fluids from the surrounding environment as opposed to an internal generation of sulphate ions. Consequently the authors decided to assess the change in thermal conductivity and mechanical performance of cement-based foams after immersion of prismatic specimens into sulphate solution for specific durations of time. Accordingly, a sulphate bath was prepared conforming to ASTM C1012 [21] with 99% anhydrous Na_2SO_4. Appropriate adjustment was made considering the purity of commercial grade anhydrous sodium sulphate to attain a concentration of 50 gm/L. The pH of the solution was found to be 6.5 which lay within the specified range recommended in ASTM C1012 [21]. The temperature of the bath was maintained at 23 °C, while the ratio of the volume of the solution to that of the specimen was equal to 4 which is within the range specified in the standard. The container was sealed to prevent any loss of moisture. A separate series was placed in a water bath to serve as the reference data for neutral initial pH. The specimens were extracted periodically from the baths for further testing. After the flexural test, the broken halves of each specimen were salvaged for cube compression testing and thermal evaluation. Altogether, 15 prisms from each mix were immersed in sulphate solution and an equal number in the water bath. Three specimens were extracted from the bath after 7, 15, 30, 60 and 90 days of exposure. An additional series of three specimens per mix was left unexposed to serve as the basis of comparison. At each stage of extraction, the solution was renewed to maintain the solution-to-specimen volumetric ratio for the subsequent immersion. In order to observe the onset of surface cracking, a visual comparison was made by photographing each specimen before and after the immersion. These photographs were taken at approximately similar conditions of camera position and lighting.

2.3. Mechanical tests

The specimens extracted from sulphate and water bath after each exposure period were tested for flexure. The prisms were subjected to four-point flexural test in accordance with ASTM C1609 [22]. A yoke was placed around the specimen in order to attach two LVDTs which would allow the measurement of mid-span deflection without any errors that might arise from support settlement or accidental twists. The load–deflection time histories were recorded using a data acquisition system, which captured data at 5 Hz. Subsequently, cubes sawn from the intact portion of the tested specimens were tested in compression as per ASTM C109 [23] to assess