



Sulfate and acid resistance of lithomarge-based geopolymer mortars

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HIGHLIGHTS

- Lithomarge-based geopolymer mortars had very good sulfate resistance.
- Geopolymer microstructure was unaffected by sulfate (Na_2SO_4 and MgSO_4) attack.
- Geopolymer mixes showed better acid resistance than Portland cement mixes.
- H_2SO_4 and HCl solutions caused dealumination of the geopolymer microstructure.

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ABSTRACT

The resistance of room temperature cured geopolymer mortars (GPM) against chemical attacks, *i.e.* sodium and magnesium sulfate solutions, and sulfuric and hydrochloric acid solutions, was evaluated. GPMs were formulated using a lithomarge precursor (low-purity kaolin) to achieve 28-day characteristic compressive strengths of 37.5 and 60 MPa. Their performance was compared with those of equivalent Portland cement mortars (PCMs) having the same paste volume and strength grade. GPMs with both strength grades showed superior performance against sulfate attack when compared to PCMs. No visual deterioration was observed in GPMs, the mass and length changes were relatively small, and no changes to the microstructure were detected – in contrast to severely deteriorated PCMs. As confirmed by visual observations and lower mass loss, GPMs showed better resistance to attack by both acids than PCMs. GPMs provided a better quality (lower permeability) of an acid-degraded layer, lowering the degree of further deterioration. The main mechanisms of the matrix deterioration of GPMs in both acids was dealumination of the hardened binder, with a higher degree of changes detected for sulfuric acid.

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1. Introduction

Portland cement based concrete, being the most versatile and widely used construction material, frequently operates in environments where it is exposed to aggressive aqueous media. Contact of highly alkaline ($\text{pH} > 12.5$) hardened cement paste (hcp) with water carrying aggressive ions can cause chemical as well as physical degradation [1,2]. Three common types of chemical degradation mechanisms are: an ion exchange reaction between aggressive medium and the hardened binder, reaction leading to leaching of ions from the hcp, and reaction causing growth of

expansive products within the pore structure of hcp [1]. These chemical processes often occur simultaneously and are directly responsible for physical changes to the hcp microstructure, *i.e.* altering porosity, permeability and integrity of the concrete [2]. With respect to the aggressive species, two common types of chemical attack are external sulfate attack and acid attack [3–6].

External sulfate attack is associated with applications where a structural element is in contact with sulfate-rich environments such as contaminated soil or ground water, sea water or wastewater treatment infrastructure [4,5]. Severity and extent of the attack depends on factors related to concrete itself, such as the type of cement used and the overall quality of concrete, but also on the properties of the aggressive medium, *e.g.* sulfate ion concentration and mobility, type of the cation (most common being Na^+ , K^+ , Mg^{2+} and Ca^{2+}), or pH. [4,5]. In the majority of sulfate attacks, the most vulnerable compounds to react with waterborne sulfate ions are

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calcium hydroxide (CH) and phases containing aluminium, such as AFm (e.g. monosulfate) and unreacted C_3A [5–7]. Reactions will result in the formation of expansive salt crystals, such as ettringite and gypsum (with ettringite being more devastating than gypsum [8]), within the hcp pore structure [9]. Consequently, expansion and cracking result in severely compromised structural integrity of the attacked concrete. Cracking also leads to further propagation of the attack.

Concrete can be exposed to a wide range of attacks caused by both organic and inorganic acids [5,10]. Acidic media can originate from agriculture, urban and industrial human activities, as well as occurring naturally [5,10]. The severity of acid attack, in addition to composition and quality of the concrete, depends on: the acid type; concentration and pH of the acid solution; on the availability of acid solution to react with concrete; and finally – on the medium surrounding the concrete (whether it flows and/or contains abrasive particles) [11,12]. The focus of this work is on strong mineral acids, namely sulfuric acid (H_2SO_4) and hydrochloric acid (HCl). Their actions lead to strong decalcification of the hcp, and then (at lower pH) to removal of Al^{3+} and Fe^{3+} [5,10]. The order of dissolution of calcium bearing phases is as follows: CH > AFm > Aft > C–S–H [5,12]. In the course of decalcification new compounds are precipitated and, depending on their solubility in water, they may leach out or remain in the pore structure [12]. The action of H_2SO_4 is especially severe because the acid attack is coupled with the sulfate attack [5]. Progression of the acid attack front causes loss of alkalinity coupled with an increase in porosity and permeability, thus leading to mass and strength loss [12]. Inability to maintain Ca^{2+} ion concentration in the hcp and a more open microstructure of the altered zone causes further ingress of the attack front [12].

As a result of the typically low resistance of Portland cement based materials to the actions of sulfate and acid attack, the service life of the exposed structure is reduced. This has multifaceted consequences: financial, social and environmental, associated with costly maintenance or replacement of the damaged structure. The problem of chemical attack may be addressed by applying layers of sealants or coatings on the concrete surface, or creating a physical barrier between concrete and the aggressive environment via protective overlays [13–15]. This should limit/prevent ingress of aggressive media into the concrete microstructure. Whilst effective, these solutions proved to be costly and labour intensive [16]. An alternative approach is to improve the performance of concrete by modifying its composition; however, such solutions vary in effectiveness. Typically, to improve sulfate resistance of concrete, either cements with reduced C_3A content are used (sulfate resistant cements) or reduced CH content and permeability of hcp are sought after, for instance by using blended cements [5,17–19]. The resistance of cement-based materials to acid attack strongly depends on the content and type of calcium bearing hydration products [11], intrinsic permeability of undamaged concrete [11] and most importantly – on the permeability of the acid-degraded layer [12]. To improve these features, investigations were conducted into the use of blended cements, partial replacement of Portland cement with additions (also called supplementary cementitious materials) or use of polymer modified cements. However, conflicting reports on their effectiveness to provide acid resistance are reported [20–26]. Recently, a promising solution has emerged in the form of geopolymer binders which have been reported to have improved resistance to sulfate [27–33] and acid attack [21,27,30,31,34–40] due to their ceramic-like microstructure.

Geopolymers are a low-carbon alternative to Portland cement-based binders in mortar and concrete. They typically consist of a powder precursor, primarily composed of amorphous aluminosilicates, and a liquid chemical activator containing an alkali

source, providing elevated pH, in the form of hydroxides, silicates, or their blends [41]. When mixed, the two components undergo a dissolution/condensation reaction to form a ceramic-like amorphous microstructure [41]. Geopolymers are a sub-group of a much wider group of materials, called alkali activated materials [42].

As the definition currently stands [41,43], there is a wide range of potential precursors and activators that may be used and which would produce geopolymers of varying quality. In terms of the precursor, the most common candidates are high purity kaolin [44,45] and different types of clays [46–48], or waste/by-product materials, such as slags [41] and ashes [41,49–51]. However, some of these materials may not be readily available across the globe or are too expensive. It is well known that in the UK and Europe, the supply of good quality fly ash for concrete applications is limited [52] and will become more so due to the move away from fossil fuels for electricity generation [53]. While almost all of the UK produced slag is used in cement production, a continuous demand of fly ash for use in blended cements or as partial replacement of Portland cement will cause increased pressure on its supplies [52]. Heath et al. [54] anticipated that current global production of fly ash and slag meets only 20% of Portland cement demand and will most likely fall below 10% by 2050. It is estimated that, despite being limited, the UK has larger resources of kaolin than fly ash [52]. However, high costs involved in the production of high purity metakaolin (made from clays containing at least 85% kaolin [55]), render it uneconomical for use in the majority of geopolymer concrete and mortar applications [56]. Consequently, locally available clays with lower kaolin content are of interest. Some of them have already been reported to produce geopolymer binders with compressive strength of at least 50 MPa upon calcination [57–62]. In Northern Ireland, a large deposit of metamorphose lateritic lithomarge forms a part of the Interbasaltic Formation (IBF) [63]. Lithomarge is a soft rock, primarily containing kaolinite ($Al_2Si_2O_5(OH)_4$), gibbsite ($Al(OH)_3$), goethite ($FeO(OH)$), hematite (Fe_2O_3) and various smectite minerals [64]. Geopolymer binders with strength exceeding 50 MPa were successfully formulated with calcined lithomarge obtained from rocks containing at least 60% w/w of kaolinite [61].

Sulfate and acid attack on clay based geopolymer binder systems has previously been investigated using geopolymers formulated with pure metakaolin [27,33,36,38,39]. In order to encourage the use of less expensive kaolin geopolymer binders, this research aimed to assess and directly compare the resistance of lithomarge-based geopolymer and neat Portland cement mortars to chemical attack by sulfate (Na_2SO_4 and $MgSO_4$) and mineral acid (H_2SO_4 and HCl) solutions. Mortars were formulated with characteristic compressive strengths of 37.5 and 60 MPa, to additionally assess the influence of strength grades on the resistance to chemical attack.

2. Experimental programme

The methodology of the research will be first outlined, followed by the description of materials and mix proportions used. Mortar mixing and sample preparation will then be described, followed by the presentation of testing procedures.

2.1. Methodology

To allow for a like-for-like comparison, two geopolymer mortar (GPM) mixes and two Portland cement mortar (PCM) mixes were selected from work reported elsewhere [62]. Mortars with both binders were optimised to have equivalent paste volumes of 500 L/m³ and characteristic 28-day compressive strengths to satisfy normal (37.5 MPa) and high strength concrete (>60 MPa) applications.

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