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banahCEM – COMPARISON OF PROPERTIES OF A LATERITE-BASED GEOPOLYMER WITH CONVENTIONAL CONCRETE

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ABSTRACT: This paper presents the overview of a project, as well as selected results from the experimental work aimed at direct comparison of the physical and durability properties of room temperature cured geopolymer and Portland cement concretes (GPCs and PCCs, respectively). Geopolymer binder was formed by reacting low purity geologically-originated lateritic clay, banahCEM(a), with an alkali silicate activator, banahCEM(b). Economical and “industry friendly” mix design of GPCs was developed to satisfy common medium and high strength applications. In order to allow a like-for-like comparison, both GPCs and PCCs were proportioned with equivalent paste volume and characteristic compressive strength. It was found that in the first 24 hours after mixing the GPCs achieved 55–75% of their 28-day strength, while equivalent PCCs gained 37–43%. Selected durability properties of developed geopolymer mortars, such as acid (solutions of H₂SO₄ and HCl) and sulfate (solutions of Na₂SO₄ and MgSO₄) resistance have been found to be better than those of Portland cement systems. Room temperature curing and reported engineering properties make this geopolymer binder most suitable for harsh environment applications, where rapid strength gain is of essence, *e.g.* repair applications, pre-cast industry (fast mould turnover), tunnel or mine linings.

Keywords: acid attack, geopolymer concrete and mortars, setting time, strength development, sulfate attack.

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INTRODUCTION

Geopolymer-based concretes are a novel class of construction materials, where the cementitious binder is replaced with typically low carbon geopolymer alternatives. Geopolymer binders are produced by reacting an alumino-silicate precursor (often a waste or a by-product material) with an alkali-silicate solution, also called chemical activator [1]. An inorganic polymerisation reaction results in the formation of hardened material with three dimensional amorphous microstructure. Thanks to unique, ceramic-like microstructure, geopolymer-based materials have been reported to have potentially superior/equivalent physical and durability properties when compared to conventional materials made with Portland cement [2]. However, where the concrete/construction industry is concerned, the geopolymer concrete still has to be proven to be more user-friendly and cost-efficient, and comply with specific engineering properties in order to gain more popularity.

Recognising the benefits of geopolymer binders, a Northern Irish company banah UK Ltd, which was established in 2008, undertook a challenge to develop and commercialise such a binder for the UK market. Development of a novel, low-carbon geopolymer binder system, called banahCEM, was the result of an intensive five-year R&D programme undertaken by the company. As shown in Figure 1, this binder consists of two components: a powder precursor – banahCEM(a) and a chemical activator – banahCEM(b), and by mixing with water and aggregates, it can be used to produce concretes and mortars. The powder precursor is based on an aluminosilicate, namely altered basalt (lithomarge) sourced from the Interbasaltic Formation of the Antrim Lava Group (Northern Ireland) [3,4]. The precursor is manufactured by lithomarge calcination at *ca.* 750 °C, followed by grinding. The chemical activator is an aqueous solution of alkali silicate.

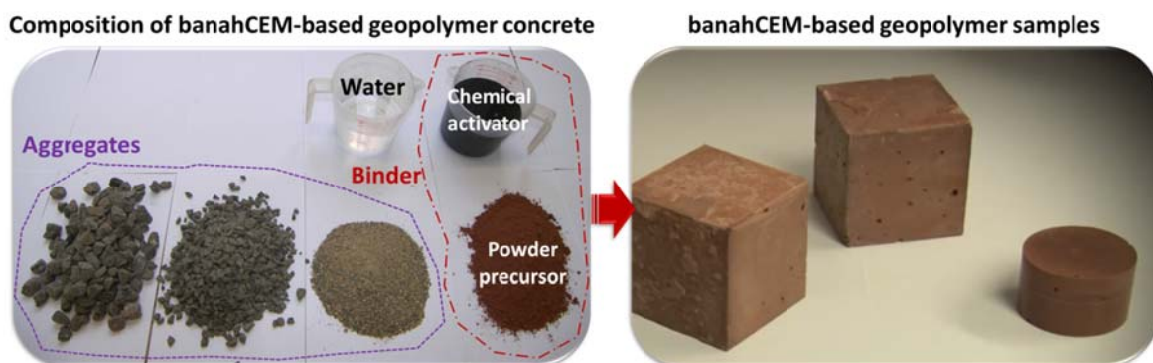


Figure 1 Materials required for banahCEM geopolymer concretes and mortars

In order to commercialise the product, the initial effort led to a three-year R&D Invest Northern Ireland funded project, which started in February 2014, involving banah UK Ltd and Queen's University Belfast. The aim of this ongoing project is to provide strong proof to designers, specifiers, clients and developers, *via* extensive testing programme, that the novel geopolymer-based binder system (banahCEM) can be produced and placed with similar ease as Portland cement concretes (PCCs) and mortars (PCMs). Concrete mix designs and quality control systems are being developed to assist in the larger scale production of the binder and commercialisation of the banahCEM-based geopolymer concretes (GPCs) and mortars (GPMs) for a range of products (Phase 1a). Low energy GPC and GPM mixes are being optimised to investigate the practicalities of producing mixes similar to those currently used in construction, *i.e.* having characteristic compressive strengths of 37.5 and 60 MPa (Phase 1b). These mixes are being comprehensively tested to provide reassurance that the new binder will have fresh, mechanical and durability properties at least as good, if not better, than those of Portland cement. Specifically, this is being achieved by checking whether design strength of geopolymer mixes can be obtained whilst having acceptable fresh and early age properties, *i.e.* workability and setting time, required for casting structural elements (Phase 2). In addition to ease of placement, values of structural properties such as strength in compression and tension, thermal expansion, modulus of elasticity, creep, shrinkage, permeability and fire resistance of these new concretes and mortars are being measured (Phase 3). The durability of these new systems is being evaluated by testing their resistance to freeze-thaw action, acid attack, sulfate attack and alkali-silica reaction (Phase 4). Moreover, chloride ion ingress and corrosion of reinforcement steel is being tested to evaluate the protective properties of banahCEM-based materials.

Finally, conventional PCC and PCM mixes, with the same paste content and characteristic strength as corresponding geopolymer mixes, are being designed and tested using the same experimental programme as outlined above. This allows for a like-for-like comparison of banahCEM and Portland cement systems.

This paper presents results obtained from the ongoing research project. Objectives of this part of work were to characterise the materials used in this research, and to study the strength development, setting time, as well as selected durability properties, *i.e.* resistance to acid and sulfate attack, of nominated geopolymer mixes. The results were benchmarked against analogous cement mixes.

EXPERIMENTAL PROGRAMME

Materials

Geopolymer binder was based on a two component system produced by banah UK Ltd: banahCEM(a), being the powder component, and banahCEM(b), the liquid component. As described earlier, banahCEM(a) is an aluminosilicate precursor [3,4]. Aqueous solution of alkali silicate with water content of 41.2% was used as a chemical activator. The solution had a specific gravity of 1.57. Portland cement CEM I 42.5N, produced by Quinn Cement in Northern Ireland and conforming to the requirements of BS EN 197-1:2011 [5], was used in conventional cement concretes. Chemical compositions of banahCEM(a) and Portland cement, determined using X-ray fluorescence spectrometry, are shown in Table 1. A polycarboxylate-based superplasticiser Chemcrete HP3 produced by Larsen Building Products, with specific gravity of 1.1 and solid content of 35%, was added to conventional

concretes. Water from the mains supply (17 ± 1 °C) was used as the mixing water, throughout.

Table 1 Chemical composition and physical properties of banahCEM(a) and Portland cement

ELEMENTAL COMPOSITION [%]	banahCEM(a)	PORTLAND CEMENT
SiO ₂	32.04	20.21
Al ₂ O ₃	24.99	4.79
Fe ₂ O ₃	25.21	2.78
CaO	7.78	63.01
MgO	1.71	1.93
MnO	0.37	0.08
TiO ₂	3.17	0.27
Na ₂ O	0.36	0.19
K ₂ O	0.15	0.59
SO ₃	0.22	2.60
P ₂ O ₅	0.14	0.12
LOI [%]	3.08	3.16
Specific gravity [-]	2.89	3.13

Three aggregates, *i.e.* 0–5 mm concrete sand, 4–10 mm crushed basalt and 10–20 mm crushed basalt, were sourced in Northern Ireland. The oven-dry particle density and water absorption (tested according to BS 812-2:1995 [6]) of all aggregates are reported in Table 2.

Table 2 Basic physical properties of used aggregates

AGGREGATE NAME	PARTICLE DENSITY ON THE OVEN-DRY BASIS [kg/m ³]	1-H WATER ABSORPTION [%]	24-H WATER ABSORPTION [%]
0–5 mm concrete sand	2695	0.9	1.1
4–10 mm crushed basalt	2790	1.4	2.2
10–20 mm crushed basalt	2751	1.2	2.1

Mix Composition

An extensive experimental programme was undertaken to develop the mix design of banahCEM GPCs and GPMs. Geopolymer mixes were proportioned by varying two of the mix proportion parameters, *viz.* paste volume and water to solid (w/s) ratio. Two GPC mixes, with slump of 50–100 mm (determined according to the procedure described in BS EN 12350-2:2000 [7]) and 28-day specific characteristic compressive strengths ($f_{c,28}$) of 37.5 and 60 MPa (referred to as GPC-37.5 and GPC-60, respectively), were selected for further examination. Their proportions are given in Table 3. For comparison, two cement-based concretes (PCC-37.5 and PCC-60), having the same paste volume, workability and $f_{c,28}$ as the corresponding GPCs, were designed following the BRE mix design guidelines [8] – mix

proportions are also given in Table 3. Importantly, a superplasticiser was added to PCC mixes during mixing process to obtain the essential workability.

Table 3 Proportions of geopolymer and Portland cement concrete mixes

MATERIALS [kg/m ³]	MIX CODE			
	GPC-37.5	GPC-60	PCC-37.5	PCC-60
banahCEM(a)	265	363	-	-
banahCEM(b)	188	258	-	-
Portland cement	-	-	336	468
Superplasticiser	-	-	1.2	0.6
0–5 mm concrete sand	794	739	794	739
4–10 mm crushed basalt	476	443	476	443
10–20 mm crushed basalt	714	665	714	665
Water for aggregate 1h absorption	23	21	23	21
Total added water	87	57	191	197
Free water content[kg/m ³]	141	142	169	176
Binder content [kg/m ³]*	376	515	336	468
Total paste content [kg/m ³]	517	656	503	643
Total paste content [L/m ³]	275	325	275	325
w/s ratio [-]	0.375	0.275	-	-
w/c ratio [-]	-	-	0.502	0.376

* – for geopolymer mixes it represented banahCEM(a) and solid part of banahCEM(b)

To achieve the required $f_{c,28}$ of 37.5 and 60 MPa, mortar mixes were designed with different w/s ratios for GPMs (0.375 and 0.275, respectively) and w/c ratios for PCMs (0.60 and 0.42, respectively). Paste volume of all mortars was kept constant at 500 L/m³, so the effect of aggregates on the properties of mortars could be ignored.

Variability in concrete and mortar production was also taken into consideration, in accordance with the guidelines of BRE for design of normal concrete mixes [8]. Therefore, concrete and mortar mixes were designed with the 28-day target mean strength ($f_{m,28}$) of 50.6 MPa and 73.1 MPa (margin of 13.1 MPa was added to each $f_{c,28}$).

Mix Preparation

To ensure that no other parameters influenced the results, all constituent materials were stored in dry locations at room temperature (20 ±2 °C) prior to batching. Before mixing, all aggregates were oven-dried (at 105 ±5 °C) for more than 48 hours, until a constant mass was reached, subsequently cooled and stored in plastic bags until mixing. All mixes were batched following exactly their pre-determined mix proportions, *i.e.* no additional water (other than what is given in the mix design) was added during mixing to adjust the workability.

The concrete mixes were prepared in a Croker RP50XD, 82 kg capacity rotating pan mixer, in 18 L batches. The mixing procedure consisted of the following steps:

- Step 1 – Pre-saturation of aggregates started 30 minutes before the actual concrete mixing (Step 2). Dry aggregates were placed in the mixer's pan with ½ of the total water (free + pre-saturation water) and mixed for 1 minute.

- Step 2 – The dry portion of binding material, *i.e.* banahCEM(a) or Portland cement, was introduced into the mixing pan followed by 1 minute of mixing.
- Step 3 – The remaining water (free + pre-saturation water) and the chemical activator (in the case of GPCs), or the superplasticizer (in the case of PCCs), were added to the mix. This was followed by 6 minutes of mixing. The beginning of this step is referred to as time zero.

The mortar mixes were made in a Hobart mixer in 3.5 L batches using similar procedure to that described above.

Sample Casting, Demoulding and Conditioning

All specimens were cast in two layers. Each layer was compacted on a vibrating table. After casting, the moulds with samples were covered with polythene plastic sheets and placed in the conditioning room (RH > 95% and 20 ±1 °C). Samples were demoulded at 24 ±0.5 hours, counting from the time zero, and placed in plastic boxes on 15 mm plastic supports. Boxes were filled with water to the height of 5 mm, then covered with tightly fitting lids and stored in the conditioning room (20 ±1 °C). This procedure allowed the conditioning of the samples at RH of > 95% and prevented unintentional carbonation of the samples, and leaching of alkalis.

Test Techniques

Compressive strength of concrete specimens at given ages (3-hour, 6-hour, 12-hour, 24-hour, 3-day, 7-day, 28-day, 91-day, 182-day and 365-day) was determined by crushing three 100×100×100 mm cubes each time (at a constant loading rate of 200 kN/min). The average of three measurements is reported in MPa.

Initial and final setting times of concretes were determined with penetration resistance method described in ASTM C 403 [9]. A wet-sieve method, using a 5 mm sieve, was used to sieve out coarse portion of aggregates and obtain a mortar sample. Mortar samples were cast in plastic moulds (150 mm size cubes) and compacted. Samples were left in the conditioning room at 20 ±1 °C and between experiments were covered to prevent water evaporation. Penetration resistance results were plotted against time. For each mix, the times of initial and final setting (counting from the time zero) were determined as the times when the penetration resistance equalled 3.5 and 27.6 MPa, respectively. Setting time results are reported in minutes.

Resistance to inorganic sulphuric (H₂SO₄) and hydrochloric (HCl) acid attack was tested based on the general guidelines provided in ASTM C 267 [10]. After five weeks of curing, sets of four 50×50×50 mm mortar cubes from each mix were placed in plastic boxes containing acid solutions (20 ±1 °C) with concentrations of 0.10, 0.31 and 0.51 moles of H₂SO₄ or HCl per kg of solution. Every 7 days, any loose material was removed from the samples by gentle brushing under a stream of tap water. Subsequently, the mass of each cube was recorded, and they were returned to the boxes holding fresh acid solutions. This procedure was repeated for 8 consecutive weeks. To exemplify acid resistance of tested specimens, the mean cumulative percentage of mass loss (for 4 cubes) during 8 weeks of testing for samples immersed in acid of concentration of 0.51 mol/kg solutions (which corresponds to 5% H₂SO₄ and 1.9% HCl by weight of solution) is reported in this paper.

Sulfate attack resistance was tested similarly to the procedure described in ASTM C 1012 [11]. After five weeks of curing, sets of three 25×25×285 mm mortar bars from each mix, equipped with 6 mm stainless steel balls at each end of the bar, were placed vertically in plastic boxes containing 0.352 moles of Na₂SO₄ or MgSO₄ per litre of solutions. Samples were kept in the solutions (20 ±1 °C) for 52 weeks during which their length was measured at specific intervals (every week for the duration of the first 4 weeks, then every two weeks for the duration of 8 weeks, and for the remaining 40 weeks they were tested every 4 weeks). During the first 12 weeks of testing, sulfate solutions were renewed every 2 weeks, and every 4 weeks afterwards. The mean length change (for three bars) at week 32 of measurements, given in microstrains, is reported in this paper.

RESULTS AND DISCUSSION

Concrete Strength Development

Strength development of GPC and PCC mixes over the period of one year is shown in Figure 2. As designed, all mixes achieved their 28-day target mean strength. As expected, mixes with lower w/s ratio (for GPC) or lower w/c ratio (for PCCs) had higher compressive strength.

In comparison to PCC mixes, GPCs had very high initial strength. At the age of 3 hours, it was possible to demould and test the GPC samples (the 3-hour strength was *ca.* 7.5 MPa for GPC-37.5 and *ca.* 15 MPa for GPC-60), while PCC samples were still soft, as they have not reached their initial setting time yet (see next section). At the age of 24 hours, GPC-37.5 had strength 1.5 times higher than PCC-37.5, while GPC-60 strength was almost double that of PCC-60. Up to the age of 28-days the strength gap between the corresponding GPC and PCC mixes decreased, with clear strength crossover effect, in favour of PCC mixes, at 28 days.

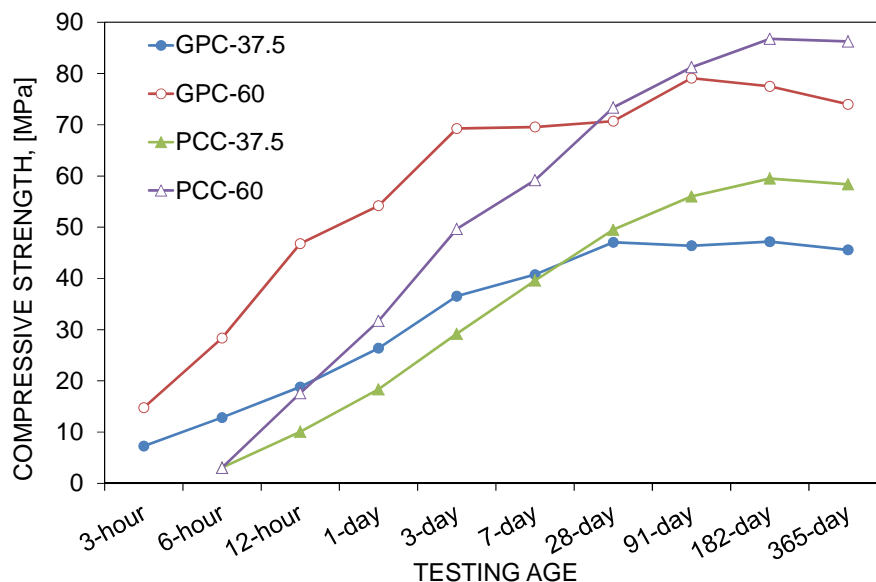


Figure 2 Strength development of geopolymer and Portland cement concretes

The strength gain of GPCs was very rapid up to the age of 3 days. Afterwards, the strength development was relatively slow in comparison to PCCs. Beyond the 28-day mark, there was no noticeable change in the strength of GPC mixes, indicating that the geopolymer reaction

was nearly completed at this age. On the other hand, cement hydration continued, resulting in further increase in strength of PCCs. Importantly, in the first 24 hours, the GPCs achieved 55–75% of their 28-day strength, while equivalent PCCs gained 37–43%.

Setting Time

The initial and final setting times found for selected concrete mixes are shown in Figure 3. GPMs showed relatively shorter initial and final setting times than those obtained for cement mixes. The ratio of initial setting time between PCC-37.5 and GPC-37.5 was 1.9, while for stronger grades it was 2.5. Similar ratios were obtained for final setting times.

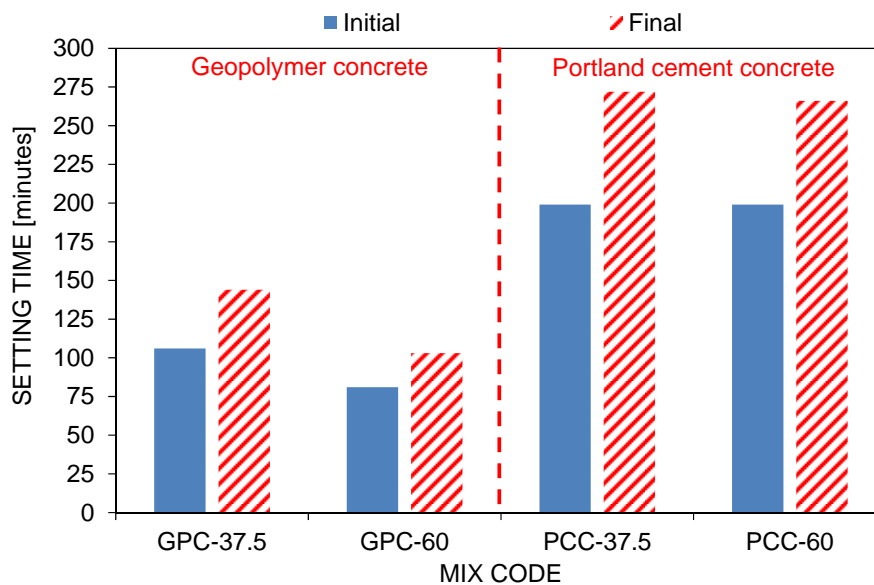


Figure 3 Setting time of geopolymer and Portland cement concretes

Where GPCs are concerned, both setting times increased with an increase in w/s ratio (setting times of GPC-37.5 were higher than those of GPC-60). For lower strength grade PCC mixes, the initial and final setting times were only marginally higher than those of PCC-60. This was unexpected, since the setting times of Portland cement systems are known to increase with an increase in w/c ratio [12]. However, superplasticiser was used in both PCC mixes, which might have affected the setting times [12]. The difference between initial and final setting increased with an increase in the w/s ratio or w/c ratio.

Acid Attack Resistance

The cumulative mass losses obtained for mortar samples during 8 weeks of immersion in acid solutions are shown in Figure 4. When GPM mixes are compared to PCMs of the same strength grade, it is clear that geopolymer-based ones lost less mass than the conventional mixes, hence showing better resistance to H_2SO_4 and HCl acid attacks.

Irrespective of the binder used and strength grade, H_2SO_4 attack caused larger mass loss than that of HCl (*ca.* 1.5 times larger for GPMs and at least 2.3 times larger for PCMs). Where Portland cement is concerned, both acids have a dissolution effect on hardened cement paste caused by hydrogen ions (primarily dissolution of portlandite and decalcification of C-S-H and C-A-S-H phases) [13]. Indeed, as the pH of the solutions was periodically measured, a

rise in the pH over the period of seven days (one cycle) was clearly observed. In addition, H_2SO_4 acid leads to sulfate attack. Expansive acid reaction products (gypsum and, later on, ettringite) can precipitate on the samples' surface and within pores of already degraded near-surface layer, leading to microcracks and sprawling caused by induced tensile stresses [14]. In the case of HCl, chloride ions penetrate into the cement matrix, causing monosulphate to react forming Friedel's salt and ettringite [13]. For banahCEM mortars, the mechanisms of the matrix destruction caused by these two acids are uncertain, but they appear to be distinctly different, and this requires further detailed investigation. As reported by Gao *et al.* [15], immersion in HCl solution of metakaolin-based geopolymer samples, made with potassium activator, caused leaching of KOH, $KHCO_3$ and K_2CO_3 . The increase in pH of the acid solution used for banahCEM seems to support these findings.

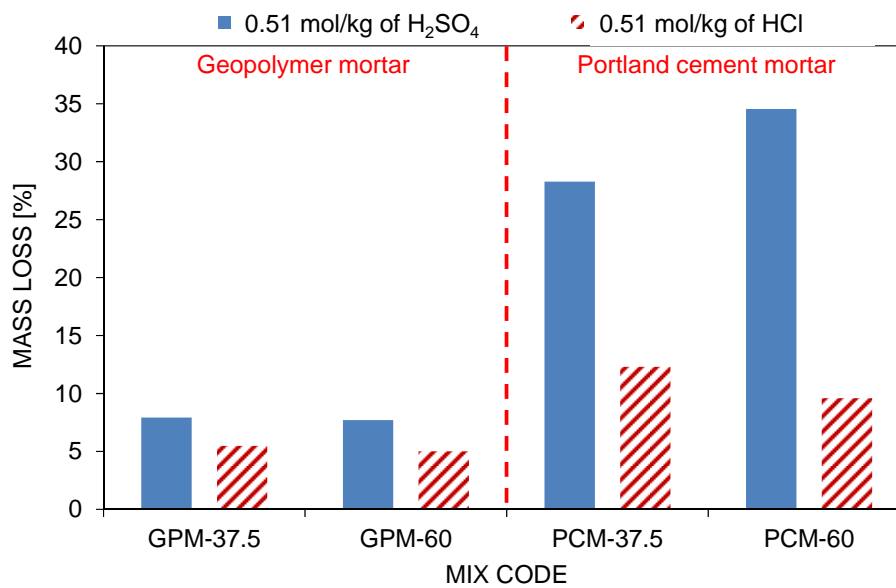


Figure 4 Mass loss of GPM and PCM samples due to the acid attack

Sulfate Attack Resistance

Regardless of strength grade, up to the age of 32 weeks, samples of GPM mortars proved to be stable in sulfate solutions, exhibiting relatively small change in length (shrinkage/elongation below 200 microns for both Na_2SO_4 and $MgSO_4$ solutions). As expected, attacked by sulfate ions, PCM mixes showed large expansions [16]. Mix PCM-37.5 immersed in Na_2SO_4 was already disintegrating at 32-week, while PCM-60 had expansion exceeding 1200 microstrain (cracks can be observed at samples of both PCM mixes, while PCM-37.5 bars showed large curvature – see Figure 5). For $MgSO_4$ PCM-37.5 and PCM-60 had expansion at the level of more than 4500 and 700 microstrain, respectively.



Figure 5 Samples of GPMs and PCMs after 32 weeks of exposure to Na_2SO_4 solution

General Remarks

Presented results are very promising for designers and producers of concrete, both ready-mix and precast. It appears that it is possible to use banahCEM to cast structural elements and demould them as quickly as 3–12 hours later, potentially without providing any external sources of heat to speed up the strength development. Rapid development of strength could enable fast mould turnover on the work site. The binder could revolutionise the precast concrete production by allowing for as many as four concrete castings a day. This would lead to maximised use of moulds, concrete factory production assets, and space, providing precast producers with substantial savings on one hand and increased production capacity on the other. Reported durability properties, *i.e.* resistance to acid and sulfate attack, makes this geopolymer binder most suitable for harsh environment applications, where rapid strength gain is of essence, *e.g.* repair applications, tunnel or mine linings.

CONCLUSIONS

Geopolymer concrete and mortar mixes were designed with 28-day characteristic strength of 37.5 and 60 MPa to satisfy common medium and high strength structural applications. Their physical and durability performance were compared with performance of equivalent Portland cement mixes (having the same paste volume and 28-day compressive strength). Based on the selected results, obtained during the ongoing research project, the following conclusions have been reached:

- GPCs showed very rapid compressive strength development, achieving 55–75% of their 28-day strengths within the first 24 hours after mixing. Corresponding PCCs gained 37–43% within this time.
- In general, GPCs had shorter initial and final setting times than the equivalent Portland cement mixes. Setting times of GPCs increased with a decrease in strength grade (increase in w/s ratio). Since superplasticiser was added to PCCs, setting times of lower grade mix (with higher w/c ratio) was only slightly longer.
- GPM samples exhibited lower mass lost than the conventional cement mixes, hence showing better resistance to attack of both tested acids (H_2SO_4 and HCl). Irrespective of the binder used and strength grade, H_2SO_4 attack caused larger mass loss than that of HCl . It was recognised that further work is required to explain the action of these acids on GPMs.

- After 32-weeks of immersion, geopolymer mortars showed superb resistance to attack by sodium and magnesium sulfate salt solutions, irrespective of the strength grade of the mortar mix. PCM mixes were found to expand in these two media, with the lower grade mixes being more prone to the sulfate attack. Larger expansion was recorded for PCM samples stored in Na₂SO₄ solution.

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