J Mater Sci (2009) 44:4472–4481
DOI 10.1007/s10853-009-3678-2

Influence of high temperature and high acidic conditions on geopolymeric composite material for steel pickling tanks

Deeptangshu Chaudhary · Huihua Liu

Received: 13 January 2009 / Accepted: 9 June 2009 / Published online: 23 June 2009

Abstract Traditionally, most equipment that was used for containing corrosive solutions and fumes for the steel industry was fabricated from steel with a rubber-lined interior. An additional acid-resistant brick lining was used to protect the rubber lining and also to act as thermal insulation. This investigation focussed on a geopolymer material that would simultaneously solve the two issues, recurrent acid leakage from the brick-lined structure and minimal thermal stress during operation. The scope of this project was limited to identify the extent of weight loss in a strong HCl acidic environment for a standard geopolymeric composite at 90 °C. Accelerated tests on geopolymer samples were conducted over 110 days and the weight loss results were extrapolated over a time period of 300 days. Visual inspection showed that OPC had a reduced diameter after 40 days of acid exposure, and therefore, the overall acid penetration for OPC was significantly higher than GPC. The mechanism of weight loss or material degradation was not the key aim in this investigation, rather we aimed to obtain a new composition to withstand highly acidic condition (which are not encountered with ordinary Portland cement (OPC) concrete, e.g. in sewage conditions). The average weight loss of a theoretical slab of geopolymer sample in accelerated test conditions (90 °C and 22% HCl) is found to be 1.8 wt% in 60 days for single-face exposure and the average thickness loss predicted for a 40 mm thick slab is about 3.5% after 300 days of exposure in operating conditions of 18% HCl and 80 °C after reaching steady-state weight loss. This is significantly better compared to a standard concrete and brick slab or steel-lined rubber slab. This initial investigation indicated that with further scientific investigation and understanding of the material, the application could be broadened to further minimise acid corrosion over longer time periods (10 years).

Introduction

With a recent increase in demand throughout the world for new and rebuilt stainless steel finishing lines, there has been a renewed interest in improving production efficiencies. Steel pickling lines is an ideal area which would hugely benefit from the contemporary growth in the plastic equipment industry because pickling involves highly caustic environment at high temperatures but with no moving parts. Polymeric composites are ideal candidates because of their chemical resistivity and economics as plastic components have low installation and significantly reduced maintenance cost advantages.

Some trials have replaced the complete old brick-lined tank setup with polypropylene (PP) tanks as a solution to recurring leakage due to latter's chemical resistivity and overall lower cost. However, with poor design and increasing thermal stresses, PP tanks failed to capture any significant market in the conservative steel industry that is generally ignorant to the versatility of polymer properties. Further, their conservatism stems from established beliefs which call for a detailed functionality of any alternative solution. An alternative solution with a plastic replacement tank involves huge resource allocation and significant

---

D. Chaudhary (✉) · H. Liu
Department of Chemical Engineering, Curtin University of Technology, Perth, WA 6102, Australia
e-mail: d.chaudhary@curtin.edu.au

H. Liu
e-mail: Huihua.liu@postgrad.curtin.edu.au

capital outflow. For example, decommissioning the old brick-lined tank and its subsequent removal involves (1) enormous down time as removal of old tanks takes about 8 days, (2) significant resource tie-up with the decommissioning and (3) requiring specialised services for removal of the tanks.

Geopolymer is a mineral polymer from the geochemistry process [1]. Geopolymer source materials usually contain a high amount of silica and alumina with reactive amorphous phase or fine-grained phase [2]. Source materials such as low-calcium fly ash [3–6], high-calcium fly ash [7], metakaolin [8–10] and slag [11–13] can be used to make geopolymer. According to Xu and van Deventer [14], the source material for geopolymerisation can be a single material or combination of various materials. The alkaline chemicals used in geopolymerisation are $Ca(OH)_2$ [15]; NaOH [16]; Na_2SiO_3 (sodium silicate); a combination of NaOH and sodium silicate [17, 18]; a combination of KOH and NaOH [19]; KOH, potassium silicate and its combination [10] and sodium carbonate [20]. The final product of this process is a new cementitious material called amorphous aluminosilicate, and contains a small amount of quartz, mullite and maghemite, in the same form in metakaolin geopolymer [10]. A combination of alkaline solution determines the final product and geopolymer strength. An excellent review of the current state of art in geopolymeric composites has been compiled recently by Duxson et al. [21].

The structural evolution and phase stability of the metakaolin-derived geopolymer with different alkali cation types exposed to elevated temperatures had been comprehensively investigated in Peter et al.'s work by X-ray diffraction (XRD) and infra-red spectroscopy (FTIR) techniques. It has been shown that the phase composition of NaK- and K-geopolymers is more complex than Na-geopolymer and their durability in sodium and magnesium solution was affected by the type of cation present during preparation, which was ultimately attributed to the extent of cross-linking and polymer structure porosity available in the matrix [6, 22].

Therefore, the final strength of geopolymer can be significantly affected by the alkaline solution concentration apart from curing method and temperature. In dry-heat curing, the compressive strength of the geopolymeric samples may be about 15% higher than steam curing [23]. Kovalchuk et al. [24] stated that geopolymer paste hardens very slowly at ambient curing; therefore, this type of concrete is usually subjected to high-temperature curing. It was found that high-temperature curing in the range of 60–900 °C is necessary to complete the amorphous aluminosilicate process [25]. Heat will accelerate the reaction in a geopolymer mixture. Due to the fast reaction in high-temperature curing, a precuring or rest period in ambient temperature is necessary to increase the compressive strength. The recommended rest period is at least 24 h [18] and up to 3 days [26].

Several studies have been carried out to investigate the mechanical properties of geopolymer concrete and results strongly indicate similar behaviour to ordinary Portland cement (OPC) concrete [3, 4, 23–26]. On the other hand, the interesting aspect of geopolymer is its acid resistance in aggressive environment such as high-acid and -temperature conditions. Some studies have been carried out with fly ash geopolymeric material by Bakharev [6] and Song et al. [27]. In the case of OPC concrete, the calcium salts form rapidly because of acid attack, and then lead to reduction in strength and mass. Low-calcium fly ash is expected to have a high durability due to low calcium content (3–4%). Bakharev [6] stated that geopolymer with sodium hydroxide as alkaline activator has the best resistance to acid compared to geopolymer with sodium silicate or potassium hydroxide activators. Acid concentration also influences the deterioration degree of geopolymer concrete. Based on SEM analysis, Song et al. [27] found that geopolymer matrix remained identical after immersion in sulphuric acid solution with pH as low as 3. The gel-aggregate interface still existed and geopolymer gel still binds the aggregate around after sulphuric acid treatment.

The purpose of this study was specifically aimed at determining the deterioration of low-calcium fly ash geopolymeric material at high-acid and high-temperature conditions following compositions [3, 25] and curing history as recommended earlier [18, 22, 24] to obtain greater compressive strength geopolymeric material. The geopolymeric behaviour is assessed using a weight loss method. The assumption underlying the weight loss method is that the geopolymeric matrix reaction with the acid environment is uniform and reaches a steady state after a certain period of exposure, and this was corroborated by the rate of weight loss which was found to be stable. For accurate degradation behaviour, knowledge of individual component aging mechanisms, synergistic effects and spatial variability of the thermo-oxidative degradation is required; however, in the absence of methods to predict composite matrix behaviour, weight loss had been an accepted method to compare the thermal oxidative stability of different materials [27], like magnesium [28] and carbon/epoxy [29, 30].

In terms of material performance, we compared our results with two types of material—PP for its high acid resistance, but poor thermal stability, and OPC for its compressive strength data; however, it must be pointed out that literature has only limited data on acid resistance [27] and no data on combined acid and temperature resistance at very aggressive operating conditions.

Experimental setup

The experimental setup

The experimental setup consisted of five geopolymer samples, four OPC samples and four PP samples, all with different weights. The concrete samples were of identical dimensions; however, the PP samples were rectangular samples following plastic standard testing. Samples were prepared by premixing ingredients listed in Table 1 for 1 h in a mixer. This mix was poured into cylindrical moulds (dia 100 mm, height 200 mm) and the samples were cured for 24 h at 60 °C as a standard, and a prolong curing about 3 days was further allowed at room temperature [18, 24]. An acid testing rig was designed as shown in Fig. 1. This rig was capable of variable temperature, variable acid concentration bath for acid testing of geopolymer samples. In the first phase, three samples were subjected to conditions of (1) 0% HCl at 95 °C, (2) 12% HCl at 45 °C and (3) 30% HCl at 25 °C. Each sample was weighed for its weight loss (if any) after 4 days and the weight changes were observed against one control sample of geopolymer at 0% HCl and 25 °C (control sample). The test samples were dipped in acid such that all surfaces were visible to the acid attack. The objective was to assess the relative effect of the key parameter, namely, water, heat and acid. This phase of testing was carried out for 45 days and was then replaced by the second state of the experiment called the 'accelerated test regime'.

The purpose of any accelerated test regime is to obtain comparable data which predicts material behaviour over a longer period. This accelerated regime subjects the samples to controlled acid and temperature conditions (as the sample would experience in real time during operation) to gauge the behaviour and property change over a much longer period. In the second stage three geopolymer samples were kept at 95 °C in 22% HCl solution and weight changes was measured after every 4 days for a total period of 60 days. Two geopolymer samples used in this second phase were virgin samples (no prior acid contact) and a third sample was sample (GP3) from the first phase. Table 2 outlines the samples and their test conditions. GP1 and GP4 are repeats to access experimental errors due to sample preparation. The overall aim of the second phase was to assess how the weight change varies with change in acid quality. At the end of the second phase, all samples were tested for their compressive strength.

Table 1 Composition of the geopolymer tested

Material	Mass (kg/m3)
Aggregate, 10 mm	554
Aggregate, 7 mm	647
Sand	647
Fly ash	408
NaOH (14 M)	41
Sodium silicate	103
Super-plasticiser	6.1
Added water (100%)	25.8

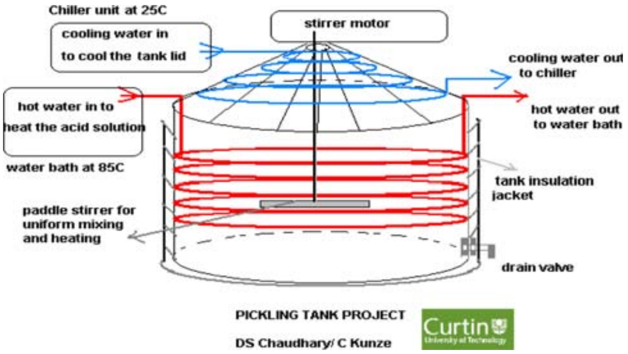


Fig. 1 Experimental rig to test geopolymer samples in hot acid bath

Table 2 gives us the sample testing conditions and description used in this study. The experimental weight loss measurement procedure consisted of taking the sample out from its experimental conditions and washing the sample with fresh water so that there is no acid traces left with the geopolymer samples. Once the sample is washed thoroughly with the water then it is cleaned with dry absorbent paper to absorb any water on the surface of the geopolymer material. The samples were left at room temperature for about 30 min to stabilise in the environment. After that geopolymer samples were weighted and the mass changes were recorded. To mimic the steel picking operation in a tank where only one face is exposed to the aggressive conditions, the weight loss values were later converted for a single face acid exposure condition using a theoretical rectangular slab of identical volume to weight ratio as the original samples. Being rectangular originally, the PP samples did not need this correction.

Accelerated testing

Quantitative accelerated life tests (QALT), are designed to quantify the life of the product and to produce the data required for accelerated life data analysis. This type of test involves the controlled application of accelerated stress conditions in order to stimulate product failure and provide life data more quickly. QALT tests can employ overstress acceleration to speed up the times-to-failure for the products under test. With usage rate acceleration, which is appropriate for products that do not operate continuously under normal conditions, the analyst operates the products

Table 2 Second-phase test samples, their nomenclature and their test conditions

Geopolymer specimens	OPC specimens	PP specimens	Experimental conditions
GPC	–	–	(Control)
GP1	OC 1	PP 1	95 °C in 22% HCl (virgin)
GP2	OC 2	PP 2	95 °C in 0% HCl
GP3	OC 3	PP 3	95 °C in 22% HCl (prior acid contact)
GP4	OC 4	PP 4	95 °C in 22% HCl (virgin)

under test at a greater rate than normal to simulate longer periods of operation under normal conditions.

In the following results, the inverse power law (IPL) model (or relationship) is used for accelerated stresses and is given by:

$$L(V) = \frac{1}{kV^n}$$

where:

L	represents a quantifiable life measure, such as mean life, weight loss, etc.
V	represents the stress level time
K	is one of the model parameters to be determined, $(K > 0)$
n	is another model parameter to be determined.

Such model has great applicability and can be applied to any system where the stress is applied in a controlled environment and the data is understood well. The parameter n in the inverse power relationship is a measure of the effect of the stress on the life. As the absolute value of n increases, the effect of the stress is greater. Negative values of n indicate an increasing life with increasing stress. An absolute value of n approaching zero indicates small effect of the stress on the life, with no effect (constant life with stress) when $n = 0$.

Results and discussion

Visual inspection

The visual inspection was carried out prior to weighing the sample. This is carried out to observe the attack of acid on the sample and observe any changes to sample surface (such as increase in porosity) due to acid attack on the sample. In all cases it was observed that although there were some large pores appearing in the samples soaked in the acid bath, the material offered significant resistance to the acid as well as heat attack after an initial paling of the surface (see Fig. 2a). Further, after the compressive tests were completed, the acid penetration depth was also calculated (Fig. 2b).

In terms of macroscopic investigation, it was found that the penetration of the HCl inside the geopolymer sample was approx. 10 mm from the outside surface for most samples (see Fig. 2b). In terms of the microscopic response to external stresses, the acid resistance was further verified at the end of second stage after conducting compressive testing on each geopolymer sample. These samples were then compared to the control OPC and PP samples. Table 3 tabulates the acid or water penetration depth for all the samples. GPC, PPC and OPC samples were kept in water

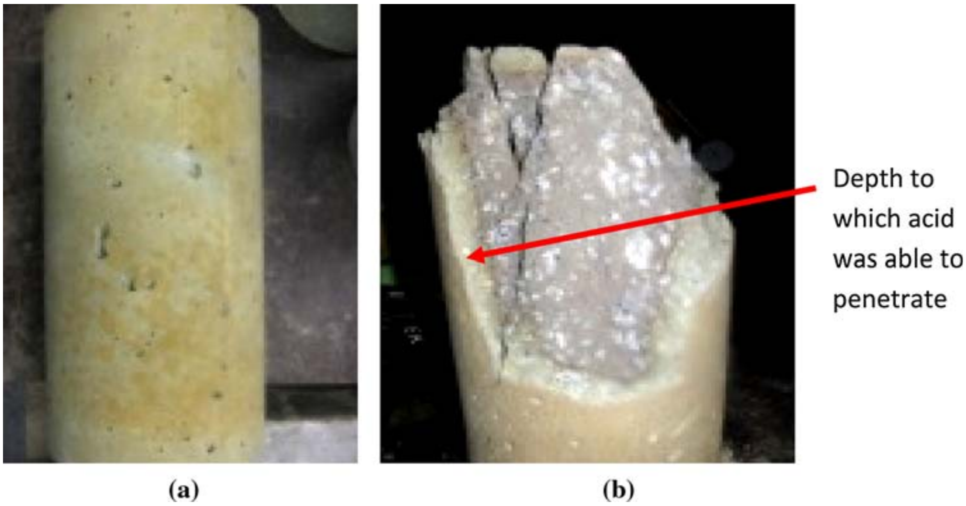


Fig. 2 **a** Paling of the surface and increase in the surface voidage. **b** The extent of the acid penetration can be seen from the depth of the colour change

Table 3 Acid/water penetration depth (by colour change) for samples after 110 days

Samples	GPC	GPC 1	GPC 2	GPC 3	GPC 4	OPC 1[a]	OPC 2	OPC 3[a]	OPC 4[a]	PP1	PP2	PP3	PP4
Depth (mm)	5.5	8.7	6.2	10.6	9.8	14.6	8.6	13.7	16.9	0	0	0	0

[a] Sample diameter at the end of the test was reduced by 8.5% on average

as they were the control samples. PP samples showed no change due to their resistance to acid.

Table 3 shows that the effect of temperature is significant for liquid penetration in concrete or geopolymeric samples in presence of acid. Clearly, temperature reduces the activation energy of the dissolution reaction at the interface of the acid and sample and causes more acid to be able to penetrate. It is worth mentioning that OPC samples had significant mass loss and there the acid penetration measurement only accounted for the sample diameter at that measurement point. Therefore, because of the reduced sample diameter, the real acid penetration depth is greater for OPC samples. It is also to be noted that PP samples only suffered heat (95 °C) distortion but no chemical attack.

Weight loss measurements

Figure 3 shows the results of geopolymer samples subjected to the phase-one acid testing. The geopolymer samples in hot water with no acid showed interesting results. There was more curing of the micro-structure of the sample because the sample gained weight over time. Also this sample showed greater compressive strength as compared to the control sample as shown in the next section (see Table 4). The control sample also gained weight in water, indicating that the curing process identified here can be further improved; however, the sample subjected to boiling water (95 °C) showed a slow progressive weight loss after an initial gain (possibly due to salt leaching effect). From Fig. 3, it can be conclusively said that the geopolymeric sample was acid resistant at both the temperatures (25 and 45 °C) as compared to the OPC sample. Furthermore, when we compare the slope of the OPC samples at 30% HCl, we can see that until about 20 days the rate of mass loss is comparable to GPC samples; however, there was excessive weight loss after 30 days of exposure and the slope of the curve corroborate the detrimental effect of aggressive acidic environment.

Table 4 Summary of the overall (actual measured) and predicted for a slab (acid exposure only on one side for a theoretical rectangular slab (length = breadth, thickness = 1 cm) with identical volume to weight ratio as that of the original cylinder) weight loss after the total 110 days of acid exposure

Samples	u.l%	c.l%	Samples	u.l%	c.l%	Samples	u.l%	c.l%
GPC	+1.3	0.5	OPC	−0.8	−0.3	PPC	0.0	0.0
GP1	−2.8	−1.1	OC 1	−6.2	−2.5	PP 1	0.0	0.0
GP2	+0.3	0.1	OC 2	−0.8	−0.3	PP 2	0.0	0.0
GP3	−2.6	−1.0	OC 3	−6.8	−2.7	PP 3	0.0	0.0
GP4	−3.0	−1.2	OC 4	−7.6	−3.0	PP 4	0.0	0.0

u.l and c.l refer to weight loss measured overall and predicted for slab, respectively

Figure 3 also shows the samples kept in acid bath (either at 25 or 45 °C). These showed progressive loss in weight over 45 days. Comparing their weight loss data in the acid bath indicates that the 12% HCl sample had a greater weight loss tendency as compared to the 30% HCl sample. This suggested that the effect of temperature is quite significant when water or acid is present. In presence of acidic

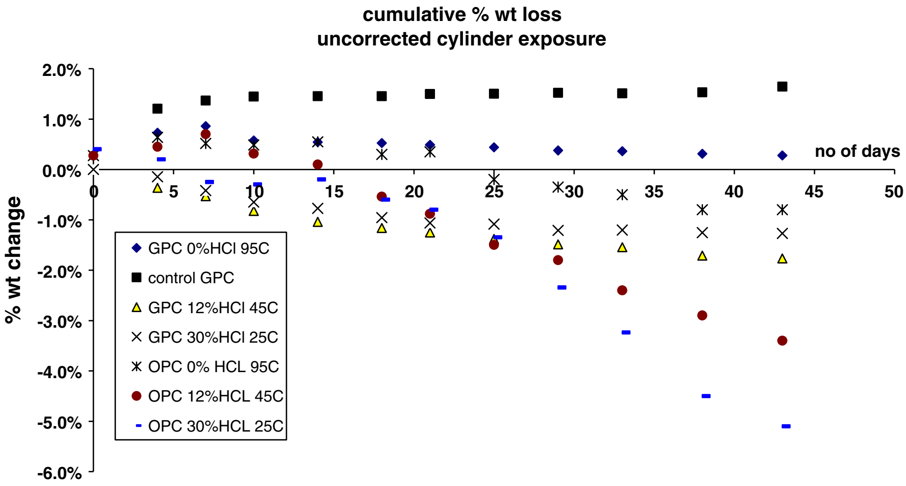


Fig. 3 Geopolymeric and OPC sample weight loss behaviour when subjected to different environment. Plastic samples had no weight loss

environment, the extent of material loss is increased, which is expected. When GPC and OPC samples are compared with any acidic environment, but at 95 °C, we can see from Fig. 3 that OPC suffers a cumulative mass loss of about 0.8% whereas the GPC samples at 95 °C gained about 0.3%. The 'unconverted cylinder exposure' means that the weight loss data is 'as is' for a cylindrical sample and has not been converted for a single side exposure for a theoretical rectangular slab with an identical volume-to-weight ratio as that of the cylinder. Once such correction has been applied, the material loss per unit weight per unit length of exposure can be compared to other control samples, such as PP and OPC. This conversion has been applied so that the data can be of direct use for pickling tank operations, where only once face of the material is constantly exposed to acid and heat.

Further, in the second phase of the 'accelerated regime' investigation, two virgin samples were included with the sample previously kept at 30% HCl at 25 °C. Fresh acid was added to the rig and the concentration was maintained at 22% HCl. Figure 4 shows the weight loss results of geopolymer samples in 22% HCl and 90 °C. This is significantly higher and harsher than the industrial pickling operating conditions of 18% HCl and 80 °C and such tests are therefore able to give variations in material property over an extended period of operation.

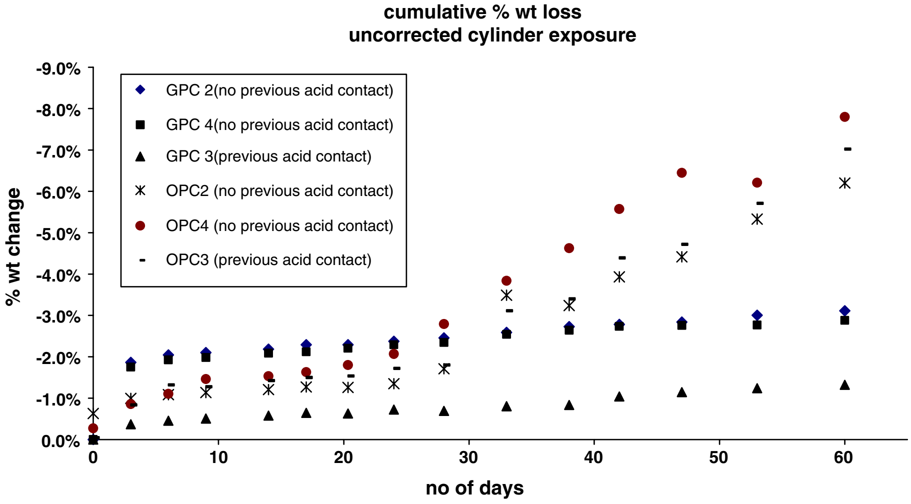
From Fig. 4, it can be seen that the GPC 2 and GPC 4 samples showed a progressive weight loss after a 2% initial loss within the first 8 days. After 60 days of acid contact at high temperature, the cumulative mass loss is about 3%. This indicates that for GPC samples, there is an initial mechanism of acid attack and once the acid penetrates into the geopolymeric structure, it reaches a steady state in terms of its degrading ability. Further, the 'old' sample with prior acid contact (30% HCl), showed comparatively lower weight loss over the same period of time (60 days). This is simply due to the fact that this sample had earlier lost an initial amount when exposed to the very high acid environment (30% HCl).

In terms of the OPC samples, Fig. 4 clearly shows a greater weight loss tendency which has not reached any steady state. Also, as expected, the mass loss in OPC samples is greater than GPC samples, but the interesting aspect is that OPC 3 (with previous acid contact) had similar mass loss compared to the other two virgin samples (OPC 2 and OPC 4). This was not the case with GPC samples. This highlights the poor resistance of OPC against a highly corroding and aggressive environment as compared to GPC. It is further noteworthy that within the same time frame, virgin OPC samples lost about 7% of their initial mass compared to 3% for GPC virgin samples.

Figure 4 also shows that for the OPC samples, the weight loss curve has interesting behaviour, and it has a tendency to reach a steady state until about 30 days before a second stage of weight loss occurring progressively at a greater time length until 60 days. Currently, there is insufficient information to speculate whether this is an experimental error or on the mechanism of this weight loss compared to the progressively increasing and levelling-off weight loss observed with the virgin samples. Clearly, fundamental experimentation on the microstructure of the GPC compared to OPC is required to assess what mechanism controls the weight loss or degradation steps and whether the weight loss has reached a steady-state value over exposure time and to determine the time frame to reach a 'plateau'—as in GPC.

Figure 5 shows an overall comparison of the average of the virgin sample behaviour and the used sample behaviour in terms of weight loss for GPC samples. After taking into account the ~1.3% weight loss for the used sample at 30% HCl condition and by comparing the two curves of weight loss in an 'accelerated testing regime' of 22% HCl and 90 °C, it can be seen that the weight loss behaviour of the old sample is different from the virgin samples. It is

Fig. 4 Geopolymer samples weight loss behaviour in an accelerated environment. The weight loss shown is for cylindrical samples and the weight loss for the used sample has not been converted for the loss in first phase



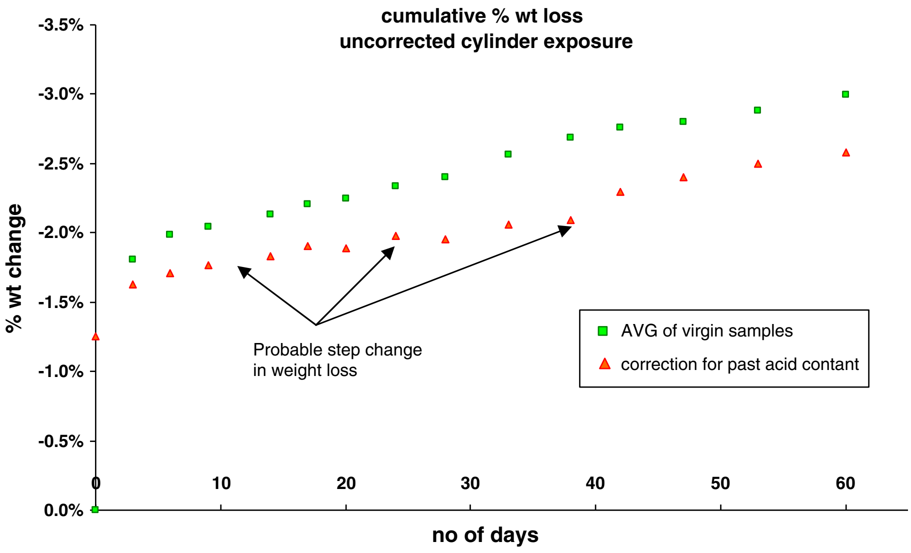


Fig. 5 Comparison of an average of the virgin and the used sample's behaviour under accelerated testing conditions. The weight loss shown is for cylindrical samples and the weight loss for the used sample has been converted for prior acid contact

possible that after extended period of testing, the two samples, namely, GPC 1/4 and GPC 3 will reach similar weight loss trend.

From Table 4, it is clear that the weight loss of OPC exceeds by 150% compared to GPC. As mentioned earlier, the PP samples did not show any observed weight loss because plastic is extremely acid resistant. However, at high temperature condition, there was significant (5–7%) shape distortion of the rectangular slab.

Compressive strength

At the end of the acid testing experiment (after 110 days) all the geopolymer samples were taken and compressive strength test were carried out according to ASTM 642. The results are shown in Table 5.

From Table 5, it can be seen that GPC samples consistently showed higher compressive strength values than OPC samples, for all acid conditions. OC 3 sample was the worst as it was exposed to acid in both the phases. A standard control (GPC) was tested for its compressive strength and its strength was 52.77 MPa. Also, it is interesting to note that sample kept in hot water showed greater compressive strength than that of the control sample. With regards to the PP samples, their torsional strength values showed no significant difference due to acid exposure; however, the observable difference was attributed to the thermal distortion of the samples. Thus, PP 3, which was exposed to the greatest fluctuation in temperature showed the lowest value of torsional strength. In terms of the geopolymer properties, this clearly shows that the current composition was better suited for high-acid and high-temperature environment. Further, this indicates a need for optimisation of the composition and curing mechanism that would increase the compressive strength and the acid resistance properties. Duxson et al. [31] had earlier shown that the microstructure of the geopolymer that is formed after curing is important in maintaining the physical properties. Clearly, our composition created a microstructure which was more suited for acid resistance than purely physical properties. Further work is currently been undertaken in our laboratory to relate the microstructure of current geopolymeric composition to its acid resistance.

Volume change correction for a theoretical slab of geopolymer

To understand the weight loss in terms of actual dimensional loss in a tank insert, the cylindrical samples were reduced to a slab volume (with length = breadth) and

Table 5 All geopolymer samples and their compressive strength test results

Samples	Compressive strength (MPa)	Samples	Compressive strength (MPa)	Samples	Torsional strength (MPa)
GPC	52.0 (±1.3%)				
GP1	46.1 (±2.3%)	OC 1	32.11 (±2.3%)	PP 1	26.16 (±2.3%)
GP2	–	OC 2	51.52 (±3.1%)	PP 2	23.7 (±3.3%)
GP3	41.58 (±2.6%)	OC 3	24.18 (±1.3%)	PP 3	21.15 (±3.3%)
GP4	48.36 (±2.2%)	OC 4	38.16 (±1.7%)	PP 4	28.36 (±3.7%)

For the PP samples we tested their torsional strength (which is the critical test for distorted samples)

Fig. 6 Cumulative % loss of thickness from a slab of 40 mm thickness and volume identical to the cylinder sample used

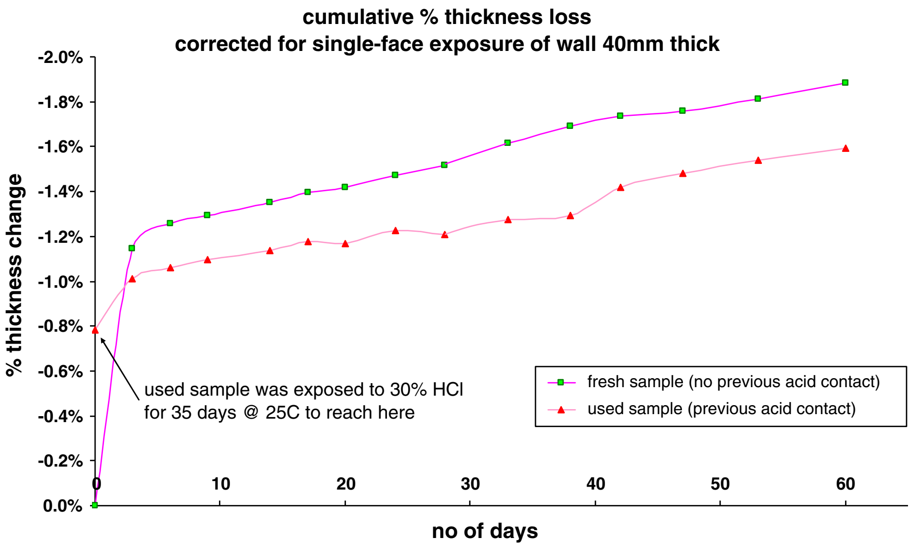


Fig. 6 shows the loss in 'thickness' of a slab having the same volume-to-weight ratio as that of the cylinder after this weight loss is predicted for a '*single-face* exposure'.

It can be seen from Fig. 6 that cumulative loss of 'thickness' over 60 days for the virgin or used geopolymer samples varies from 1.5 to 1.8%. Further, once the tendency to plateau will occur, the weight loss curve will progressively attain a steady state—meaning that the mass loss from the exposed face reaches a constant value. However, in reality, due to the regular changes in the acid concentration (due to 'charging' of the acid in tanks, this mass loss value is predicted to flatten and lower the extent of mass loss. In terms of the thickness loss *predicted*, this would mean that, for example, after a period of 300 days, there would be reduced mass loss and smaller reduction in thickness of the geopolymer slab.

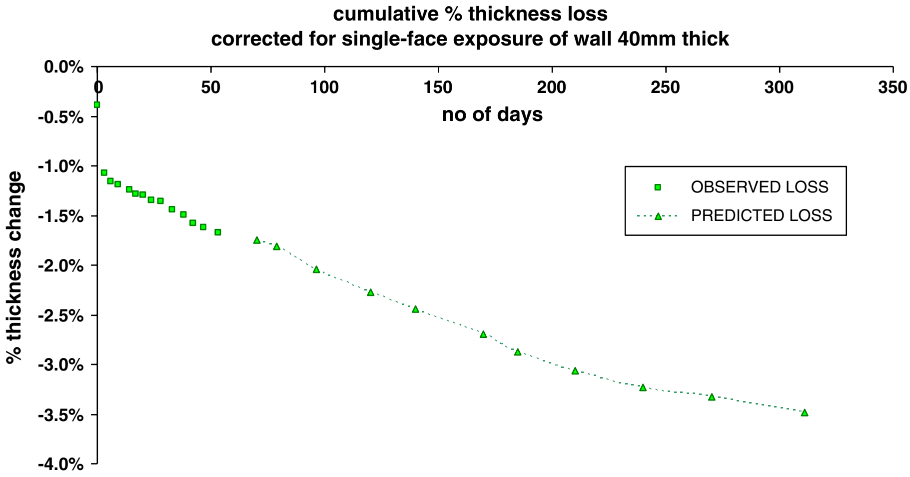
Figure 7 shows the predicted volume change (in terms of thickness change) using the inverse power law model for approximately 300 days of exposure of the geopolymer slab (*single-face* exposure) at 18% HCl and 80 °C. The predicted thickness loss is approx. 3.5% after 300 days for a slab of thickness 40 mm.

Inverse power law model of the weight loss

In the following results, the inverse power law (IPL) model (or relationship) is used for accelerated stresses and the model parameters are analysed to understand the effect of the individual stresses. Figure 8 shows a typical IPL plot with the best fit curve signifying the model parameters and Table 6 shows all the model parameters for the geopolymeric samples tested in high-acid and high-temperature conditions.

It can be seen from Table 6 values that the constant 'K' is very similar for all the tested samples. As described in "Accelerated testing", the parameter 'K' ($K > 0$) denotes the importance of the composition of the geopolymeric samples which gives the kind of microstructure to allow the

Fig. 7 Overall prediction using the Inverse Power Law model for approximately 300 days of exposure of the geopolymer slab



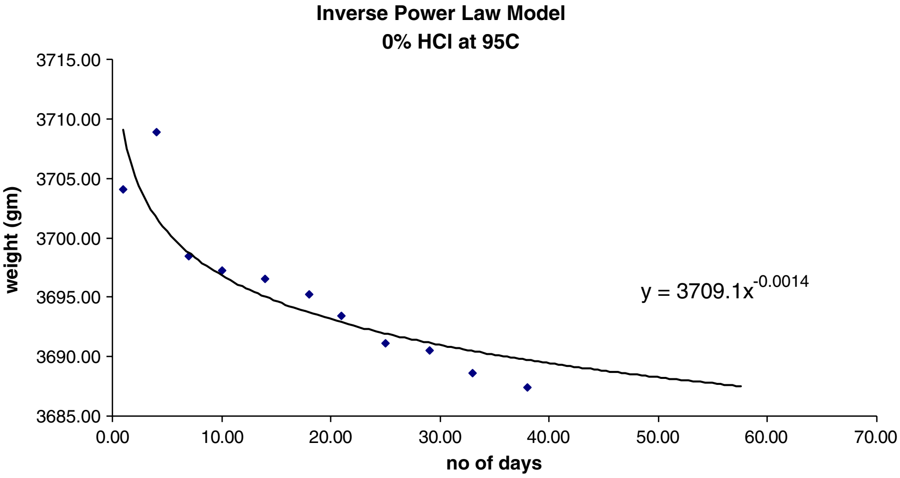


Fig. 8 Inverse power law model curve for a geopolymeric sample maintained at 95 °C with no HCl acid

Table 6 All geopolymer samples and their compressive strength test results

Geopolymer specimens	*K*	*n*	Experimental conditions
GPC	–	–	(Control)
GP1	3677.8	−0.0037	25 °C in 22% HCl (virgin)
GP2	3709.1	−0.0014	95 °C in 0% HCl
GP3	3788.5	−0.0063	95 °C in 22% HCl (prior acid contact)
GP4	3659.2	−0.0058	95 °C in 22% HCl (virgin)

For the PP samples we tested their torsional strength (which is the critical test for distorted samples)

acid to penetrate within the sample. Since our composition was identical is all cases, the '*K*' values are also similar. However, when we compared the '*n*' values, it is clear that it denotes the effect of the particular stress on the sample weight loss; meaning that when we compared a high-acid environment to a water environment, the *n* value was nearly 4.5 times higher for the acid environment. This can be understood by considering that any composition and curing technique will create a microstructure ('*K*' value) that will result in a particular rate of attack in a *stressed* environment. The '*n*' values are also useful in comparing the effect of acid and temperature alone, and it can be seen from the data in Table 6 that the effect of temperature by itself is quite significant for sample weight loss (compare GP1 and GP4). Finally, the usefulness of this analysis lies in creating different microstructures (and compare their *K* values) and the corresponding micro-structural ability to withstand the environmental stress (in this case, acid environment).

Conclusion

Accelerated tests on five geopolymer samples were conducted in two stages over 110 days and the weight loss results were extrapolated over a time period of 300 days.

The first stage was aimed at finding the relative effect of the key parameter, namely, water, heat and acid. The second stage was aimed at determining the extent of weight loss and compressive strength loss for geopolymer samples in a 22% HCl and 90 °C environment. OPC samples had significant mass loss and the acid penetration measurement only accounted for the sample diameter at that measurement point. Therefore, because of the reduced sample diameter, the real acid penetration depth is greater for OPC samples as compared to the GPC samples.

The first stage showed that curing was an important step to determine the relative rate of water absorption in geopolymer samples as samples showed a weight loss tendency in 95 °C water. Samples subjected to 12% HCl but 45 °C had a net weight loss greater than the 30% HCl sample kept at 25 °C suggesting that the operating temperature is important in determining the extent of acid attack. When the sample geometry was reduced from cylinder to slab, it was found that the average weight loss of a geopolymer slab in accelerated test conditions (90 °C and 22% HCl) is 1.8 wt% in 60 days for *single-face* exposure.

Further, the loss curve had a tendency to achieve steady state, and this will reduce the extent of mass loss (or thickness loss) over time if environmental conditions remain unaltered. The inverse power law prediction for thickness loss in a 40 mm thick slab is 3.5% after 300 days of exposure in operating conditions of 18% HCl and 80 °C.

In terms of the thickness loss predicted, this would mean that, for example, after a period of another 300 days, the net thickness loss would be comparatively lower. The geopolymer samples were also tested for loss of compressive strength after the acid tests and it was found that the compressive strength of the overall geopolymer material is reduced by ~18% after exposed to >22% HCl for 80 days.

Acknowledgements The authors are grateful to Dr. Prabir Sarker and Ms. Monita Olivia for helpful discussion and sample testing. This work was funded by a Curtin Linkage Grant and the authors are thankful for the financial support to conduct some of the testing.

References

1. Davidovits J (1994) In: Mehta K (ed) Proceedings of V Mohan Malhotra symposium, ACI SP
2. Perera D (2007) In: Freiman S (ed) Proceedings of the 1st international congress on ceramics, The American Ceramic Society
3. Hardjito D (2005) PhD thesis, Civil Engineering & Computing Department, Curtin University of Technology, Perth, Australia
4. Fernandez-Jimenez A, Palomo A et al (2006) ACI Mater J 103:106
5. Van Jaarsveld GS, Van Deventer JSJ, Lukey GC (2003) Mater Lett 57:1272
6. Bakharev T (2005) Cem Concr Res 35:658
7. Chindaprasirt P, Chareerat T et al (2007) Cem Concr Compos 29:224
8. Davidovits J (1988) In: Davidovits J (ed) World congress of geopolymer 1988, Institute of Geopolymer, Saint Quentin, France
9. Van Jaarsveld JGS, van Deventer JSJ, Lukey GC (2002) Chem Eng J 89:63
10. Palomo A, Grutzeck MW, Blanco MT (1999) Cem Concr Res 29:1323
11. Bakharev T, Sanjayan JG, Cheng Y-B (1999) Cem Concr Res 29:113
12. Shi C (1996) Cem Concr Res 26:1789
13. Palacios M, Puertas F (2006) J Am Ceram Soc 89:3211
14. Xu H, van Deventer JSJ (2002) Miner Eng 15:1131
15. Fan Y, Yin S, Wen Z, Zhong J (1999) Cem Concr Res 29:467
16. Buchwald A, Kaps Ch, Hohmann M (2003) In: Proceedings of the 11th international congress on the chemistry of cement (ICCC), Durban, South Africa, p 1238
17. Rangan BV (2007) In: Nawy EG (ed) Concrete construction engineering handbook. CRC Press, New York
18. Bakharev T (2005) Cem Concr Res 35:1224
19. Kriven WM, Bell JL (2004) Ceram Eng Sci Proc 25:99
20. Arjunan P, Silsbee ML, Roy DM (2001) In: 2001 international ash utilization symposium, Center for Applied Energy Research, University of Kentucky
21. Duxson P, Fernández-Jiménez A, Provis JL, Lukey GC, Palomo A, van Deventer JSJ (2007) J Mater Sci 42:2917. doi:10.1007/s10853-006-0637-z
22. Duxson P, Lukey GC, Van Deventer JSJ (2007) J Non-Cryst Solids 353:2186
23. Rangan BV (2006) Indian Concr Inst J 7(3):9
24. Kovalchuk G, Fernandez-Jimenez A, Palomo A (2007) Fuel 86:315
25. Hardjito D, Wallah SE, Sumajouw DMJ, Rangan BV (2004) Paper presented at concrete world: engineering & materials, ACI, Mumbai India, 9–12 Dec 2004
26. Sofi M, van Deventer JSJ, Mendis PA, Lukey GC (2007) Cem Concr Res 37:251
27. Song X, Maroozeky M, Brungs M, Chang ZT (2005) In: Davidovits J (ed) World congress of geopolymer 2005, Institute of Geopolymere, Saint Quentin, France
28. Schoeppner GA, Tandon GP, Ripberger ER (2007) Compos Part A Appl Sci Manuf 38:890
29. Pardo A, Merino S, Merino MC, Barroso I, Mohedano M, Arrabal R, Viejo F (2009) Corros Sci 51:841
30. Ammar-Khodja I, Picard C, Fois M, Marais C, Netchitaïlo P (2009) Compos Sci Technol 69:1141
31. Duxson P, Provis JL, Lukey GC, Mallicoat SW, Kriven WM, Van Deventer JSJ (2005) Colloids Surf A 269:47