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ADVANCES IN GEOPOLYMER SCIENCE & TECHNOLOGY

Influence of curing schedule on the integrity of geopolymers

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Abstract The curing at ambient and controlled relative humidity (RH) with mild heating (40–60°C) of a metakaolinite-based geopolymer of molar ratios Si/Al and Na/Al of 2 and 1 respectively was studied. To obtain these geopolymers in a crack-free state, rapid drying during curing should be avoided. Curing at a lower RH (e.g. 30%) is preferable to that at a higher RH (e.g. 70%). Curing in an RH oven does not offer any advantage over curing at ambient followed by mild heating (40–60°C) in sealed containers. The compositions of the geopolymers were then varied somewhat to see the effect on open porosity for the same curing schedule. The compositions with Si/Al = 1.86 and Na/Al < 0.8 had an increased tendency to crack, probably due to the larger water loss during curing. The lowest open porosity of <1% was obtained for a geopolymer of composition Si/Al = 2.14 and Na/Al = 0.87, but this developed some cracks after 1 year. However, the geopolymer of composition, Si/Al = 2, Na/Al = 1, on which most of the work was carried out when cured correctly did not crack even after 1 year.

Introduction

Two common methods of making geopolymers are to add fly ash or metakaolinite (clay heated to ~750°C) to concentrated alkali solutions for dissolution and subsequent polymerisation and solidification. Curing at 25–90°C in a humid atmosphere completes the curing process. The ageing of geopolymers has been mentioned in the literature [1] and the importance of understanding the role of water on ageing has been highlighted [2]. During the dissolution of aluminosilicate in the alkali solution the water is consumed to form monomers of $Si(OH)_4$ and $Al(OH)_4$ and during the polymerisation some of the water is released [3]. This water forms a surficial film (~1 mm thickness) on the geopolymer. In a recent study of metakaolinite-based geopolymers [4] using infra red spectroscopy and thermogravimetric analysis it was found that the water which is essential for the initial geopolymer reaction is present after curing as ''free'' water on heating up to ~150°C and as interstitial water at ~150–300°C. There were <1 wt% of hydroxyl groups present. While the ~150°C temperature is somewhat arbitrary, on this basis the ''free'' water was estimated as 4–6 moles per Na or K and the interstitial water as 0.6–0.8 moles per Na or K. The latter values are closer to those of Rahier et al. [5] who obtained a molar ratio of 0.2. The amount of water required for the initial reaction varies from 3.3 to 12.5 moles per Na or K in the literature [4].

Several workers have reported on changes of properties under different curing conditions for geopolymers made containing fly ash or some metakaolinite [6–12]. However, we have found only two references in the literature on curing under controlled relative humidity (RH) [13, 14]. In the former work [13] fly ash-based geopolymers were steam cured at 80°C under 40–100% RH. High strengths at both 40 and 100% RH were obtained. In the latter work [14] on metakaolinite-based geopolymers curing at a higher

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RH (96%) decreased the strength, but curing at ambient with a lower RH (50–65%) resulted in higher strength.

The major effect on strength in most ceramics is the presence of porosity and the strength decreases exponentially with the porosity [15]. Earlier qualitative work by Palomo et al. [16] also showed that geopolymer strengths are also very much dependent on the porosity. The porosities in geopolymers are very much dependant on the amount of water present in the original precursors, as for ordinary Portland cement, where the compressive strength also decreases with the water/cement ratio as long as the workability of the mixture is satisfactory [17].

Based on the understanding that the role water plays, the work on the disposition of water [4] was extended to study the curing conditions of geopolymers at ambient and in controlled humidity. We are interested in using geopolymers for radioactive waste immobilisation, hence the cost of a raw material is not of a major concern. Therefore we have produced mostly metakaolinite-based geopolymers. Also the precursor clays to produce metakaolinite can be obtained in a purer state than for fly ash which helps in reproducibility of results. In the work reported here we looked at porosity and cracking tendency, both of which have an important influence on macroscopic strength properties and water entry into the bulk of geopolymers, under different curing regimes.

Experimental

Geopolymer sample preparation

Metakaolinite-based geopolymer of Si/Al and Na/Al molar ratios of 2.0 and 1.0 respectively were mostly prepared. The molar water to Na ratio was 7.2. The batch composition consisted of metakaolinite: 33.7 wt%, sodium silicate D (Na_2O/SiO_2 wt ratio: 0.5, PQ Corporation, Australia): 63.0 wt%, and deionised water: 3.3 wt%. Metakaolinite was produced by heating kaolinite (Kingwhite 80, Unimin, Australia) at 750°C for 15 h in air. Metakaolinite was added to the silicate solution, mixed for 5 min and the stiff slurry was poured into a polycarbonate cylindrical jar and covered with a tightly-fitting screw cap. The container was vibrated for 5 min on a table to de-air. Each batch consisted of ~20 g had dimensions ~40 mm dia. × 10 mm thick. Batches were cured under different conditions. All the studies were carried out on geopolymers made from this composition except for five compositions which were made to study briefly the possible effect of compositional variation on the bulk density and open porosity. Those five compositions were made from the same precursors as above but sodium silicate solution N (Na_2O/SiO_2 wt ratio: 0.31, PQ Corporation, Australia) was also added to achieve the required molar ratios. These compositions are given in the Results and discussion section.

Curing schedules

The geopolymers were cured under different schedules. The RH in the closed container used was ~85% for 24 h when measured by an RH probe when the closed container was placed in an oven maintained at 60°C at an RH of 10%. This clearly shows that the variation in the RH inside the container was minimal irrespective of the external RH. The ambient conditions varied from 70 to 90% RH and 21–23°C. Some runs were carried out in a RH and temperature controlled oven using geopolymer mixtures in open containers. On removal from the oven the lids were closed. Generally the lids were opened after 5 days and the sample removed on the seventh day. Variations to this procedure are given in Results and discussion.

Each geopolymer was examined under a low power optical microscope (×6) for any drying cracks, and crack widths and number densities were noted. Their bulk density and open porosity were determined on the seventh day for all geopolymers except where stated otherwise.

Density and porosity

The bulk density (BD) of each geopolymer was determined by (1) measuring the mass and dimensions and (2) measuring the mass and the volume by mercury volumeter. The open porosity (OP) was determined by four methods and the results are compared below. The methods used were (1) sample immersion in *n*-octane (non-polar liquid) for 24 h and measuring the mass absorbed and calculating the open porosity using the bulk density values determined above. Saturation with water was considered inappropriate because of leaching in water, especially Na [18]. This method of determining open porosity is only a comparative method but not an absolute determination. The true open porosity would be expected to be higher than the values obtained here because of incomplete permeation of the liquid into the pores. The method (2) was similar to method (1) but the sample was evacuated under vacuum before allowing the organic liquid to saturate the sample. The method (3) was to estimate the porosity using scanning electron micrographs at two magnifications by using a point counting method [19]. This was done only for two samples to correlate with the other two methods. The method (4) was to use nitrogen adsorption to estimate a range of pores in six samples to get an estimate of sub-micron pore size distribution.

The density and open porosity determined from different methods described above are listed in Table 1 to show the variance. It is clear the differences are small and have the same order of difference. Therefore for comparison between the samples predominantly the geometrical method for bulk density and the immersion in *n*-octane for 24 h for open porosity were used. In samples where there were cracks, the Hg method was used for bulk density determinations. It is also seen for nearly the same bulk density there is ~8% difference in open porosity. This is likely due to the tortuosity and the size of the capillaries in the geopolymers which restricts the flow of *n*-octane to the sample B (Table 1). This is seen for another set of samples below.

For comparison with the open porosity determined by *n*-octane with those determined by N_2 adsorption using the BJH desorption curve are listed in Table 2. The porosity for the pore size 1.7–300 nm is different to the micro pore size determined by the organic liquid. Although the bulk density varies inversely with porosity it is possible to get discrepancies as a result of pore structure. This work is currently under study.

Product variability

To study the possible product variability two sets of four batches of geopolymers under nominally constant curing conditions were made at the same time and in the same way. The bulk density was 1.36 ± 0.02 g/cm^3 for one set and 1.44 ± 0.02 g/cm^3 for the other. Similarly the open porosities [measured by the method (1) above] were 30.7 ± 0.6 and 29.3 ± 1.5%. This gives a reasonable confidence in the product reproducibility. The open porosity percentage values were rounded off to the nearest whole number because of the magnitude of the standard deviation hereafter.

Table 1 Bulk density and open porosity[a] of geopolymers determined by different techniques

Sample ident.[b]	Geometrical BD (g/cm^3)	Hg method BD (g/cm^3)	Ambient OP (%)	Vac. OP (%)	SEM OP (%)
A	1.51	1.49	17.6	22.6	22.9
B	1.49	1.48	12.9	14.5	11.0

[a] The mean values of open porosity for A and B are 21 and 13% respectively, although the vacuum method is probably the most reliable. The measurement methods are described in the text

[b] A Cured at 22°C/2 h; 60°C/24 h; *B* cured at 22°C/2 h; 65% RH/30°C/22 h followed by 65% RH/60°C/24 h. This sample had hairline cracks (<0.1 mm)

Table 2 Bulk density, open porosity and BJH porosity of different geopolymers[a]

Hg method BD (g/cm)	Ambient OP (%)	BJH porosity (%)
1.60	9	30.5
1.50	18	24.5
1.46	27	19.3
1.46	29	27.6
1.80	4	0.5
1.49	13	25.0

[a] Sample details are given under Results and discussion, it is sufficient just to list density and porosity values to compare with BJH porosity for the pore size range 1.7–300 nm

Phase analysis

About quarter of each sample was crushed to a fine powder and analysed by X-ray diffraction (XRD, Model D500, Siemens, Karlsruhe, Germany) using CoKα radiation. All the geopolymers were analysed by XRD on the day the bulk density was determined.

Scanning electron microscopic examination was carried out on one sample and the experimental details are given elsewhere [1].

Infra red spectroscopy

Infra red (IR) spectra were measured on four geopolymers using a Nexus 8700 spectrometer equipped with a Spectra-Tech diffuse reflectance accessory, and linearised HgCdTe detector. The samples were diluted to 2 wt% in KBr. Spectra were measured over the range 4,000–650 cm^{-1}, with a nominal resolution of 4 cm^{-1}, and consisted of an average of 126 scans.

Thermogravimetric analysis

Thermogravimetric (TG) analysis was carried out on one geopolymer heating from ambient to 1,000°C using a method similar to one described elsewhere [20].

Strength measurement

Compressive crushing strength was determined on geopolymers cured under three different curing regimes, using solid cylindrical specimens of 25 mm diameter × 30 mm length. A minimum of four specimens was used for each determination. The method used is given elsewhere [21].

Results and discussion

Phase analysis

As reported by others [10, 22], using our preparatory method, the geopolymers showed in XRD patterns the presence of a major amorphous phase (deduced by the presence of a very broad diffuse peak centred at a *d*-spacing of ~0.32 nm and traces of anatase and quartz). The crystalline phases originated from the precursor clay. The XRD patterns were essentially the same for all the geopolymers studied.

The backscattered scanning electron images of polished cross sections of geopolymers were similar to the previously published micrographs [18, 23], where a few anatase grains and quartz particles originating from the clay were seen in a geopolymer matrix. There were also some incompletely reacted metakaolinite in the matrix.

Curing schedule 1

The geopolymers were cured as listed in Table 3 and their bulk density, open porosity, compressive strength and shrinkage values are also listed. Although the containers were not hermetically sealed the evaporation of water was slow. A single crack was observed only for the 80°C cured sample and it was on the side of the sample as shown in Fig. 1a. The crack, probably arose because a relatively rapid loss of water compared to that at lower curing temperatures. In previous work [6] it was shown that a higher curing temperature yielded a higher crushing strength, so the open porosity would be expected to be lower as discussed in the Introduction. At higher curing temperatures the chemical reactions are accelerated and the polymerisation rate increases as shown by calorimetry [9].

Table 3 Geopolymers cured at ambient RH

Curing conditions	BD (g/cm^3)	OP (%)	Compressive strength (MPa)	Diametral shrinkage (%)
22°C/24 h; 80°C/24 h; 22°C[a]	1.47	24	–	–
22°C/24 h; 60°C/24 h; 22°C	1.38	31	57 ± 5	0.1
22°C/24 h; 40°C/24 h; 22°C	1.39	28	61 ± 2	0.3
40°C/24 h; 22°C	1.38	31	–	–
22°C	1.38	30	59 ± 2	0.2

Lid opened on the fifth day and samples removed on the seventh day. Bulk density and open porosity are a mean of three samples. Shrinkage and compressive stress values are a mean of at least four samples and measured on the eigth day after preparation (see text)

[a] Sample had a side crack of width <0.1 mm, see Fig. 1a

The compressive strengths of three geopolymers were determined on the eighth day after sample preparation (Table 3). The strength values are nearly the same, except for the slightly more porous material which was the least. This shows that when geopolymers are fabricated and dried the same way, the variation in strength (after 8 days) is minimised. The standard deviation was much lower than that in previous strength measurements on geopolymers of the same composition, which gave a value of 85 ± 11 MPa [21]. Also the shrinkage values were similar for the same three samples (Table 3).

To study the effect of comparatively small changes of geopolymer compositions on cracking, a further series of samples was made (Table 4). Their density, porosity, weight loss on curing, and a description of the cracking are also listed. They were cured at 22°C for 24 h and 40°C for 24 h, followed by the removal of the lid on the fifth day and the sample was removed from the container on the seventh day. There were larger cracks in the samples with higher moisture loss.

The compositions are listed in the order of increasing Na/Al molar ratio in Table 4. The water loss does not seem to follow this ratio. The water requirement varies depending on the composition, with low Na/Al ratios requiring slightly more water. The compositions with Si/Al = 1.86 and Na/Al < 0.8 had an increased tendency to crack, although the loss of water was nearly the same. The lowest open porosity of <1% was obtained for the geopolymer of composition Si/Al = 2.14 and Na/Al = 0.87, but this developed some cracks after 1 year. This did not occur for the geopolymers listed in Table 3. This latent cracking is due to the loss of water, noting that the geopolymers in Table 4 contained higher H_2O/Na molar ratio than those in Table 3 ($H_2O/Na = 7.2$).

Curing schedule 2

After a 24 h cure at 22°C in closed containers, the samples were placed in the RH oven with the lid open for 24 h. After removing them from the oven the lids were closed and the samples were cured for further 72 h (total of 5 days), and the geopolymer removed from the container on the seventh day. The variation of

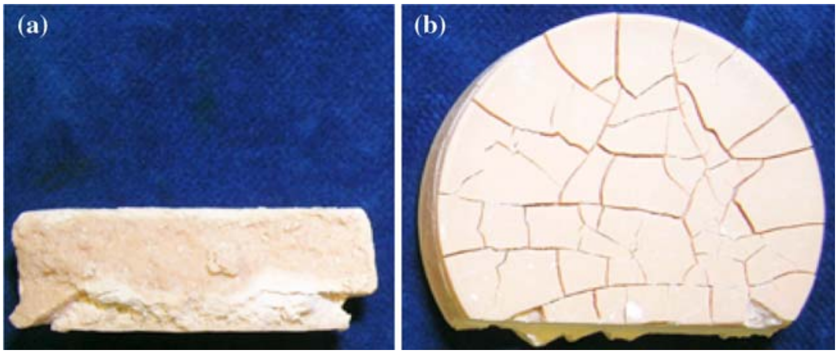

Fig. 1 (**a**) Geopolymer cured at 22°C/24 h; 80°C/24 h showing a crack on the side of the sample. (**b**) Many cracks on the top surface of geopolymer cured at 22°C/24 h; 70% RH, 40°C/6 h. Bar = 22 mm

Table 4 Different geopolymer compositions

	GP1	GP2	GP3	GP4	GP5
Na/Al[a]	0.68	0.77	0.8	0.87	0.9
Si/Al[a]	1.86	1.86	2.00	2.11	2.00
H_2O/Na[a]	10.7	8.7	8.7	8.6	7.4
BD (g/cm^3)	2.07	1.74	1.80	2.14	1.75
OP (%)	3	14	4	0.4	14
Wt. loss (%)[b]	16.7	18.2	15.7	15.2	18.8
Observations[c]	One crack inside below the top surface.	Two cracks Inside below the top surface.	No	cracks.	Hairline crack.
Cracks ~20 on the curved surface.					
Crack size (~) (mm)	1	0.5	–	<0.1	0.5

All kept covered in the container for 5 days before opening the lid and removing on the seventh day. Cured at 22°C/24 h; 40°C/24 h

[a] Molar ratio. H_2O is the total water expressed as a molar ratio

[b] Weight loss is the percentage weight loss of the sample, derived from loss of water between fabrication and the seventh day

[c] Observations after 7 days and after 1 year are the same except for GP3 which had developed a large crack ~1 mm size in the center between 7 days and 1 year

open porosity with RH for the geopolymers is shown in Fig. 2. The open porosity is least for 30% RH at 40°C. The bulk density varied inversely with the open porosity. The variation in open porosity at higher RH was much less. All the geopolymers had cracks as indicated. Exposure to 30–70% RH without sealing the sample caused rapid drying relative to a sealed container and this probably causes the cracking.

Curing schedule 3

To study the effect of early removal from the container at constant RH, the RH was kept constant at 30% at 40°C. The geopolymers were pre-cured in closed containers in the oven at three different temperatures (22, 40 and 60°C) for 24 h, prior to placing them in the RH oven (with containers open). The dependence of open porosity on the day of removal from the container is shown in Fig. 3. Removal of the samples from their containers under 3 days was difficult as they were too wet to slide out with ease. The 22°C pre-curing gave the lowest porosity after removal on the third day. The difference in open porosity for removal between fifth and seventh day under these conditions was minimal. Early removal allows more free water to remain in the geopolymer, hence it is more difficult for

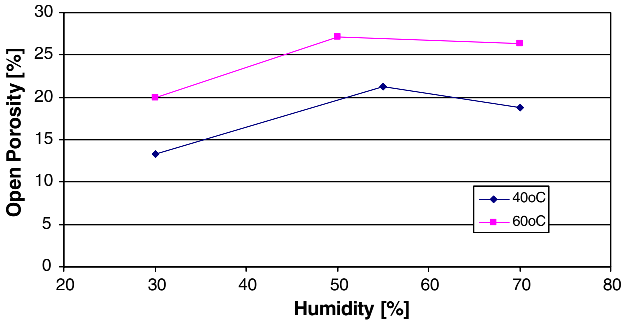

Fig. 2 Variation of open porosity (method 1) with RH at 40 and 60°C of geopolymers exposed to the RH oven for 24 h curing for 24 h at 22°C. Container lid opened on the fifth day and samples removed on the seventh day (the day the density and porosity were determined)

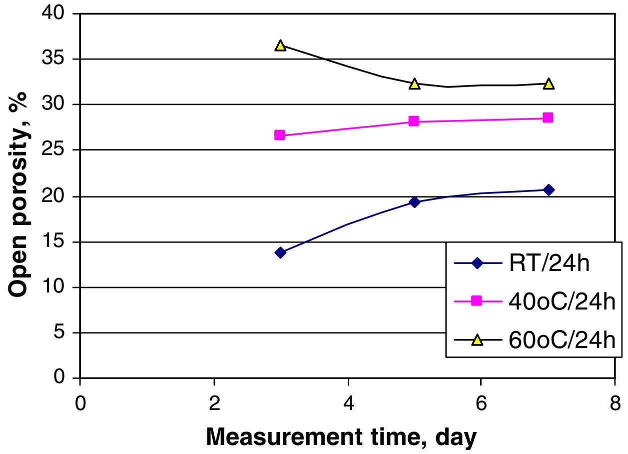

Fig. 3 Variation of open porosity (method 1) with the day of removal of sample from its container. Before removal of geopolymers were subjected to a cure indicated in the legend (RT = 22°C) The container lid opened and the sample removed on the day indicated

the organic liquid to penetrate during the test for porosity. This is clearly seen in Fig. 4 where the measured open porosity increases with loss of water (weight loss) and the bulk density is inversely related to the open porosity. The graph was plotted using the first three samples listed in Table 5. However, the three geopolymers had cracks, mostly on the top surface as indicated in Table 5. The number of cracks observed in Table 5 were much less than those shown in Fig. 1b for one of the worst cases.

Curing schedule 4

To try to eliminate cracks, a few more trials were carried out as listed in Table 5. After placing the samples in the RH oven with the lids opened, the lids were screwed on after removal from the oven. The lid was opened after 5 days of making and the sample removed on the seventh day for open porosity/bulk density determinations. The geopolymers exposed for a shorter time in the RH oven did not crack (Table 5). However, the crack-free samples had high open porosities. As discussed above the open porosity is related to the weight loss on curing. However, the fourth sample had a higher porosity in spite of a lower weight loss. After several trials it was possible to produce a crack-free sample with reasonably low porosity (11%) by curing at 22°C for 24 h and placing it in the oven at 30% RH at 40°C for 6 h and removing the geopolymer from the container after 7 days.

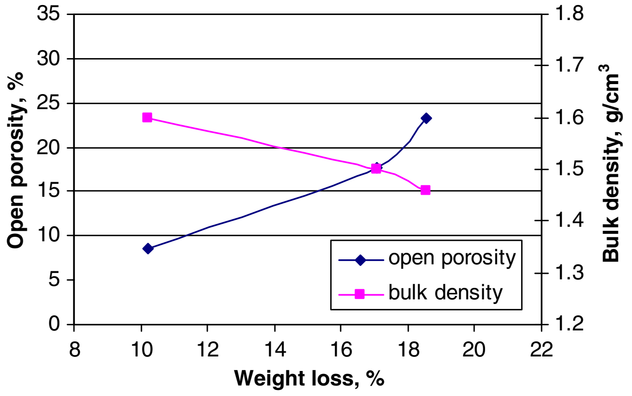

Fig. 4 Variation of open porosity (method 1) and bulk density with the weight loss for the first three geopolymers listed in Table 5

To see whether any further information could be obtained to understand the reason for cracking, four geopolymers, two cracked and two crack-free were analysed by IR spectroscopy and their spectra are shown in Fig. 5. The sample details are given in the figure caption. At 3,500 cm^{-1} the presence of a small concentration of hydroxyl groups is evident, and there are only minor difference between the results for the different samples. The water band at ~1,680 cm^{-1} of sample 4 shifted slightly for sample 1 to ~1,700 cm^{-1} and the carbonate band at ~1,470 cm^{-1} is less intense in sample 2. The main Si–O–Al framework bands at ~1,020 cm^{-1} are similar except for sample 1, which has a more pronounced shoulder at ~1,146 cm^{-1}. As for XRD analysis, the IR data do not clearly show any significant spectral differences which can be correlated with sample cracking.

Table 5 Curing of geopolymers with different RH and time[a]

Curing conditions	BD (g/cm^3)	OP (%)	Wt. loss (%)[b]	Observations
22°C/48 h; 40°C/35% RH 24 h	1.60	9	10.2	Cracks, ~0.2 mm size
22°C/48 h; 40°C/55% RH 24 h	1.50	18	17.1	Few cracks, ~0.8 mm size
22°C/48 h; 40°C/70% RH 24 h	1.46	23	18.5	Few cracks, ~0.8 mm size
40°C/48 h; 40°C/30% RH 6 h	1.46	29	14.0	No cracks
40°C/48 h; 40°C/50% RH 6 h	1.36	31	20.2	No cracks
40°C/48 h; 40°C/70% RH 6 h	1.48	27	18.8	No cracks

All kept covered in the container for 5 days (except when exposed in the RH oven) before opening the lid and removing on the seventh day. On removal from the RH oven samples were covered

[a] Weight loss is the loss of water from the original water content to the seventh day

[b] Cracks on the top surface, similar to those shown in Fig. 1b, but much less

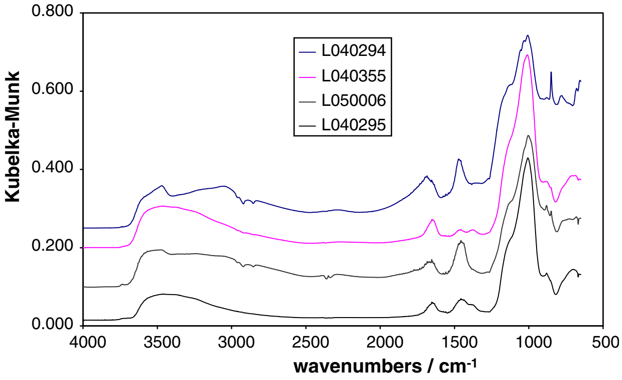

Fig. 5 IR spectra of geopolymers in the range 4,000–650 cm^{-1} at ambient temperature. Curing conditions are *sample 1*: 22°C/24 h, 80°C/24 h—cracked (Table 4); *sample 2*: 22°C/24 h, 60°C/24 h—crack-free (Table 4); *sample 3*: 22°C/24 h, 40°C/70% RH 6 h—cracked; *sample 4*: 22°C—crack-free (Table 4)

It is clear that the exposure to the RH oven atmosphere (30–70% RH), when the lid of the container is opened, causes a rapid drying of water which promotes cracking. A shorter exposure of ~6 h to the RH oven atmosphere prevents cracking (Table 5). It is seen that a maximum of ~20% weight loss due to water evaporation is lost over the time of fabrication and removal after 7 days (Tables 4, 5). A ~21% weight loss occurred when a geopolymer was kept at ambient for ~21 days [4]. At ~150°C nearly 33% of the water inventory (corresponding to 13% overall weight loss) is lost, as shown in Fig. 6. In Fig. 6 when the heating was started in the TG, the geopolymer had already sustained a weight loss at ambient. Therefore the weight losses observed (Table 5) are quite reconcilable with the water loss at much lower temperatures. This loss of water as discussed above changes the open porosity of a geopolymer as shown in Table 6 when heated. The bulk density has not changed greatly although the porosity has increased a lot. The change in porosity from ambient to 150°C is highly significant because as noted above ~33% loss of water occurs (Fig. 6). From 150 to 300°C ~6% weight loss (due to loss of water) takes place (Fig. 6) and this is reflected in the slight increase in porosity. At 500°C there is a slight decrease of porosity, with some sintering perhaps taking place. It has been shown that sintering takes place with increase of temperature above 500°C for K-based metakaolinite geopolymers [20]. Further weight loss on heating from 300 to 500°C is only <2% (Fig. 6) at which stage the sample contains very little hydrous species. Further work is being carried out to determine the weight loss in situ in the RH oven, using a load cell with unsealed samples.

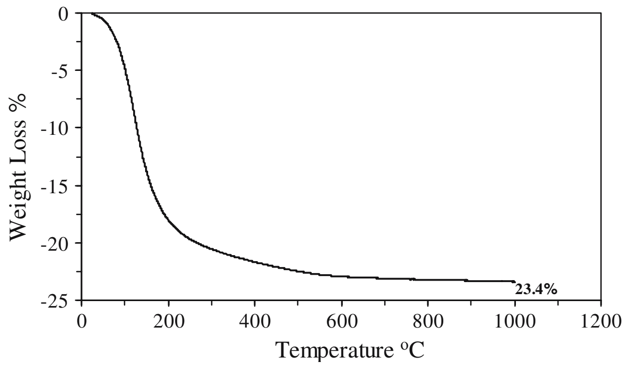

Fig. 6 TG plot of weight loss versus temperature of the geopolymer cured at 22°C/24 h, 40°C/24 h, after 14 days since preparation completed

Conclusions

To obtain geopolymers free of cracks, rapid drying during curing should be avoided by sealing the sample container. Curing in the absence of rigorous sealing, in an oven in which the RH was held at 30–70% does not offer any advantage over curing at ambient followed by mild heating (40–60°C). By varying the composition of geopolymers it is possible to get open porosity values <1%, as measured by 24 h immersion in *n*-octane. However, all such samples had cracks, although some were hairline cracks. When cured correctly the composition Si/Al = 2, Na/Al = 1 on which most of the work was carried out did not crack after 7 days and even after 1 year. The XRD analysis of all the geopolymers and the IR spectroscopy of selected samples did not show any structural changes under different curing conditions which could be attributed to the samples' propensity to crack. The water present in the geopolymer and its subsequent removal by evaporation clearly plays a major part in obtaining a crack-free geopolymer. The capillary pressure of occluded water which will drive cracking will depend on the fine structure of the microporosity and more detailed assessments on this direction are required.

Table 6 Geopolymers heated in air for 3 h

Temperature (°C)	BD (g/cm^3)	OP (%)
22	1.38	31
150	1.36	40
300	1.42	46
500	1.40	44

Cured at 22°C/24 h; 60°C/24 h, lid opened on the fifth day and samples removed on the seventh day

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References

1. Perera DS, Nicholson CL, Blackford MG, Fletcher RA, Trautman RL (2004) J Jpn Ceram Soc 112:S1008
2. Perera DS, Nicholson CL, Aly Z, Cassidy DJ, Vance ER, Finnie KS, Fletcher RA (2004) In: Conference Proceedings of Austceram 2004, 29 November to 1 December, 2004, Melbourne, CDROM
3. Mccormick PG, Gourley JT (2000) Mater Aust 32:16
4. Perera DS, Vance ER, Finnie KS, Blackford MG, Hanna JV, Cassidy DJ and Nicholson CL (2006) Ceram Trans 175:225
5. Rahier H, Simons W, Van Mele B, Biesman M (1997) J Mater Sci 32:2237
6. Swanepoel JC, Strydom CA (2002) Appl Geochem 17:1143
7. Palomo A, Alonso S, Fernandez-Jimenez A (2004) J Am Ceram Soc 87:1141
8. Yip CK, Lukey GC, Van Deventer JSJ (2003) In: Proceedings of the 28th conference on "Our world in concrete structures," 28–29 August 2003, Singapore, vol XXII, p 169
9. Alonso S, Palomo A (2001) Mater Lett 47:55
10. Palomo A, Glasser FP (1992) Br Ceram Trans J 91:107
11. Van Jaarsveld JGS, Van Deventer JSJ, Lukey GC (2002) Chem Eng J 89:63
12. Cioffi R, Maffuci L, Santoro L (2003) Resour Conserv Recycling 40:27
13. Feng D, Mikuni A, Hirano Y, Komatsu R, Ikeda K (2005) J Ceram Soc Jpn 113:82
14. Teixeira-Pinto A, Fernandes P, Jalali S (2002) Geopolymer international conference, 28–29 October 2002, Melbourne Australia, CDROM
15. Kingery WD, Bowen HK, Uhlmann DR (1976) Introduction to ceramics. Wiley, New York
16. Palomo A, Macias A, Blanco MT, Puertas F (1997) In: Proceedings of the 9th international congress on the chemistry of cement, November 1997, New Delhi, India, p 505
17. Aldridge LP(2005) Personal Communication
18. Perera DS, Vance ER, Zhang Y, Davis J, Yee P (2005) Proceedings of the world congress geopolymer, 29 June to 1 July 2005, Institute of Geopolymere, Saint-Quentin, France, p 57
19. ASTM E–562–02 (2002) Standard test method for determining volume fraction by systematic manual point count. American Society for Testing of Materials
20. Perera DS, Vance ER, Cassidy DJ, Blackford MG, Hanna JV, Trautman RL, Nicholson CL (2004) Ceram Trans 165:87
21. Latella BA, Perera DS, Escott TR, Cassidy DJ (2005) J Mater Sci 41:1261
22. Barbosa VFF, Mackenzie KJD, Thaumaturgo C (2000) Int J Inorg Mater 2:309
23. Scmucker M, Mackenzie KJD (2005) Ceramics Int 31:433