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Effects of high-pressure/temperature curing on reactive powder concrete microstructure formation Masdar Helmi a , b, ? , Matthew R. Hall a , c , Lee A. Stevens a , Sean P. Rigby d a Nottingham Centre for Geomechanics, Division of Materials, Mechanics and Structures, Faculty of Engineering, University of Nottingham, University Park, Nottingham NG7 2RD, UK b Department of Civil Engineering, Faculty of Engineering, University of Lampung, 35135, Indonesia c British Geological Survey, Environmental Science Centre, Keyworth, Nottingham NG12 5GG, UK d Division of Manufacturing and Process Technologies, Faculty of Engineering, University of Nottingham, University Park, Nottingham NG7 2RD, UK highlights _ Pressure decreases the total pore volume, but increases the capillary pore volume.

_ Heat curing accelerates the hydration, pozzolonic and crack progression. _ Heat curing induces crystalline hydrate formation inside the capillary network. _ Both treatments result transformation in micro structural composition. _ Both treatments do not affect the ITZ phase. article info Article history: Received 12 March 2015 Received in revised form 26 November 2015 Accepted 22 December 2015 Keywords: Reactive powder concrete Thermal treatment Pore size distribution SEM Interfacial transition zone abstract Reactive powder concrete (RPC) comprises cement with quartz sand, superplasticizer, silica fume, and water which is processed by heat curing and/or pressure.

This paper presents the effect of treatments (static pressure of 8 MPa and heat curing at 240 °C for 48 h) on microstructure formation. Results indi- cated that pressure decreased the total pore volume, but increased the capillary pore volume due to the movement of grains. The space created could allow additional C–S–H growth during hydration (and later pozzolonic reaction).

Heat treatment accelerated the propagation of microcracks (formed during shrink age) due to thermal expansion of the solid phases, volumetric expansion of the air and increased pressure within entrapped voids. It induced further crystalline hydrate formation inside the capillary pore network. Pressure following by heat curing treatment ?rstly increased the capillary pore volume and then accelerated both the hydration and pozzolonic reactions with subsequent increased in skeleta l density. 1.

Introduction Reactive powder concrete (RPC) is an advanced concrete tech- nology produced using an enhanced composite powder mix, and processed by high-pressure setting and post-setting heat treat- ment [1]. Exclusion of macro defects and improved homogeneity of the microstructure is achieved by limiting maximum aggregate diameter to 0.6

l m, a water–binder ratio typically <0.18, lowering of the CaO–SiO 2 ratio by addition of condensed micro silica, and enhancing ductility using steel ?bre reinforcement [1–5]. RPC can achieve ultra-high performance in terms of mechanical properties, where uncon?ned compressive strength can range between 200 and 800 MPa, with fracture toughness of up to 40,000 J/m 2 , and an ultimate elongation of up to 0.007 m/m _1 [1].

The world’s ?rst major structure built with RPC is the Sherbrooke pedestrian/bike- way bridge (in Canada, 1997) [6], having a span of 60 m and with a compressive strength of 150 MPa and no steel bar reinforcement. The main beam of the bridge was treated by heat curing at 90 °C for two days in a vapour-saturated atmosphere. The diagonal stiffen- ers of this bridge were cast in a 75 mm tube metal frame to which the static pressure treatment at 2 MPa was immediately applied, followed by heat curing after one day.

Other RPC bridges have been developed around the world such as the Sunyudo (Peace) Footbridge with a single span of 120 m (Seoul, Korea), the Sakata-Mirai Footbridge with no reinforcement http://dx.doi.org/10.1016/j.conbuildmat.2015.12.147 0950-0618/? Corresponding author at: Nottingham Centre for Geomechanics, Division of Materials, Mechanics and Structures, Faculty of Engineering, University of Notting- ham, University Park, Nottingham NG7 2RD, UK. E-mail address: evxmh3@nottingham.ac.uk (M. Helmi).

Construction and Building Materials 105 (2016) 554–562 Contents lists available at ScienceDirect Construc tion and Buildi ng Materia ls j o u r n a l ho m e p a g e : w w w . elsevier.com/loc ate/conbuildmat and lighter by 80% (Sakata, Japan), the Shepherds

Creek Road Bridge with a thin permanent precast RPC formwork panels (Australia), and the Wapello Country Bridge with no reinforcement and used for a highway (USA) [7].

The thinnest precast concrete structure achieved using RPC is 20 mm thick and area dimensions 5 _ 6 m; a train station roof supported by a single column (150 MPa) [8]. Its potential for even greater mechanical properties give RPC the opportunity for many applications beyond those of conventional concrete, e.g. advanced lightweight composites [9] or impermeable containers for hazardous fluids or nuclear waste [10].

Heat treatment is typically applied to RPC after the final setting time using temperatures between 90 and 200 °C [11]. This accelerates the pozzolonic reaction and also modifies the micro structures of hydrates by changing the C–S–H chain length from trimer to pentamer [11]. Whilst temperature curing between 200 and 250 °C microstructural change and the formation of xonotlite with some dehydroxylation of cement gel can occur [12].

This xonotlite is formed spontaneously when water extracted from hydrates is trapped in the centre of samples during transient heating [13]. Applying heat curing only in an autoclave has produced RPC with an unconfined compressive strength >200 MPa [14–16]. Earlier work suggested that the combination of pressure and heat curing during processing may result in higher strengths than by heat treatment alone because the addition of pressure treatment to the fresh concrete increases the density and decreases the porosity [1,9].

Both density and porosity have close correlation to the bulk mechanical properties of concrete [17]. More recently, Helmi et al. [18] observed that heat treatment during the hardening stage has a significant effect on the early age compressive strength, whereas static pressure treatment during the setting stage has an insignificant effect.

However, in combination when heat treatment is applied after treatment by static pressing, the strength increases by a further significant amount due to pore-filling by tobermorite/ xonotlite formation from the pozzolanic reaction and enhancing the paste-aggregate bonding mechanism. Spherical pores (caused by air entrapment) in RPC generally remain air-filled with some calcium hydroxide crystals following standard curing, but can be partially-filled with the calcium- silicate hydrate (C–S–H) mineral, tobermorite (Ca 4+x (H 2 _2x Si 6 O 17)_ 5H 2 O) when autoclave cured [16] or the much harder mineral, xonotlite (Ca 6 (Si 6 O 17)(OH) 2) if the curing temperatures are sufficiently high.

Some researchers have suggested that this would lead to reductions in measured bulk porosity by pore ?lling with a C–S– H product and consequently alters the pore size distribution by reducing the modal pore diameter [11–16]. Helmi et al. [18] found that for a compressive strength/bulk density (f c / q d) plot (using Neville's equation – see Eq.

(1) [17]), the integer, n for non- pressure treated and pressure-treated samples were signi?cantly different, but the measured compressive strength was not. f c ¼ f c ; 0 ð 1 _ n Þ x ð 1 Þ where: n = porosity, f c = uncon?ned compressive strength, f c,0 =- compressive strength of hardened paste (at zero porosity), and x = scaling integer. This assumes that f c,0 equals 500 MPa for cement paste at w/ c = 0.45.

When heat treatment was applied in both cases, the heat treated samples ?tted the same f 0 / q d plot as the non-heat treated. This appears to be consistent with the general hypothesis that pore network geometry (resulting from entrapped air) is altered during the process. If true, the heat treatment component of the process could result in (i) increased pore air pressure by reduction in modal pore diameter, (ii) reduction in microcracks within the mortar phase, and/or (iii) change in macro defect stress distribution.

The aim of this study was to further the understanding of the process-structure-property relationships for RPC in terms of pore network geometry and interfacial transition zone. The purpose is to develop a basic conceptual model for heat treatment and static pressure setting, which could enable material design and optimisa- tion of RPC at the microstructural scale for use in advanced applications. 2. Materials and method The powder ingredients comprised CEM I 52.5

Portland cement according to BS EN 197-1:2011 [19] (Cemex, UK) that it had been used by [20] ; condensed micro silica grade 940-D (Elkem, Switzerland) [20] ; and ground granulated blast furnace slag (Hanson, UK) [21] . Some properties of these materials is shown in Table 1. Quartz sand conforming to BS 1881-131 and containing the grades A (2.36 mm– 1.18 mm), C (0.60 mm–0.30 mm) and E (0.15 mm–0.09 mm), mixed in a ratio of 2:1:1 (by mass), were used for the aggregate component (David Ball Ltd., UK).

A high range water-reducing admixture based on a polycarboxylate polymer was used as a super plasticiser (Grace Construction product Ltd, UK). The composition (by mass) of the RPC mix used for this study is presented in Table 2 . The materials were mixed dry at a speed of _ 120 rpm for 2 min in a forced action paddle mixer before increasing the speed to _ 450 rpm for a further 2 min.

The water and super plasticiser were premixed and added to the mixer incrementally with further mix- ing for 10–12 min until the mixture was consistent. The mixture was used to cast prisms (with dimension 40 _ 40 _ 160 mm) in a steel mould and vibrated on a table for 3 min. The mould was covered to avoid immediate evaporation and laid in a room at temperature about 10 ° C, in order to minimise the evaporation of water during mixture and setting in early age samples.

Four combinations of treatments were applied in this study: (A) without pressure and cured in water; (B) without pressure and heat cured in a drying oven; (C) with pressure and cured in water; (D) with pressure and heat cured in a drying oven. A static pressure of 8 MPa was applied 5 h after casting, and all samples were de- moulded after 2 days.

After that a staged heat curing cycle was applied as follows: preheated at 40 ° C for 2 h before increasing to 240 °C at a rate of 50 ° C/h; cured at 240 °C for 48 h; decreased at 50 °C/h down to 40 ° C. After this step, heat-cured sam- ples were stored in water at 20 °C until testing. The specimens used to determine the compressive strength were 40 mm cubes in accordance with BS EN 196- 1:2005.

All strength data presented is the mean average for three representative samples. A Philips XL30 ?eld emission gun environmental scanning electron microscope (FEG-ESEM) was used. Micrographs were recorded using an Everhart-Thornley type Secondary Electron (SE) detector and a Back Scattered Electron (BSE) detector sup- plied by K. E. Developments.

The FEG-ESEM operating conditions were 20 kV accel- erating voltage, 4.0 spot size, and 10 mm working distance. Three representative sub-samples were clean fractured from bulk specimens of each treatment type (A –D), i.e. 12 in total. Sub-samples were mounted fracture face down and cold mounted under vacuum in 2-part epoxy resin.

Mounted samples were ground using SiC paper (400, 600, 800 and 1200 grit) on 20 cm diameter wheels at a rotational speed of 250 rpm, followed by polishing using 6 l m then 1 l m diamond pastes. Polished samples were washed using acetone and dried under a hot air blower, fol- lowed by sputter coating with _ 15 nm thick carbon using an Edwards 306 vacuum Coater. Representative micrographs were recorded for randomly selected aggregate particles and analysed using ImageJ 1.47v (National Institutes of Health, USA).

Images were smoothed using a 2px median ?lter, before bracketing the upper/ lower limits of the greyscale histogram by manually adjusting contrast/brightness. The porosity could then be segmented manually using the default threshold algo- rithm. On each SEM sample, three cross-sectioned aggregates were randomly selected and grids measuring 5 l m (thick) _ 50 l m (wide) were applied from the aggregate surface at distances ranging from 0 to 75 l m (15 grids in total). The num- ber and total area of pores were assessed using the ‘analyse particle’ tool.

Porosity was de?ned by dividing the total area of particles with the area of grid. The mean Table 1 Properties of cement, silica fume and GGBS used in this study. Properties Cement a wt.% Silica fume a wt.% GGBS b wt.% Chemical SiO 2 20.09 >90.00 36.50 Al 2 O 3 4.84 – 12.50 Physical Loss in ignition 2.36 <3 8.50 Speci?c gravity 3.15 2.20 1.00 Blain ?nesse 395 22,400 – c Ground granulated blast furnace slag (Hanson, UK) [21] . a CEM I 52.5 Portland cement (Cemex, UK); [20].

b Condensed micro silica grade 940-D (Elkem, Switzerland) [20] . M. Helmi et al. / Construction and Building Materials 105 (2016) 554–562 555 average data from three samples was ?rst plotted, and the intersection between lin- ear trend lines (at the in?ection point that indicates the transition between ITZ and bulk porosity) was used to de?ne the ITZ distance.

Elemental mapping was performed using an Oxford Instruments Inca model Energy Dispersive X-Ray Spectrometer (EDS), which can achieve 133 eV resolution of the Mn K a peak at FWHM. Quantitative EDS was conducted using four iterations at a 35 ° take-off angle, and with 60 s of live time and 30–40% dead time. During EDS analysis of the interfacial transition zone (ITZ), a series of grid areas (10 l m wide _ 40 l m long) were scanned starting from the aggregate edge and moving away at 10 l m intervals up to a maximum distance of 70 l m.

Samples were pre- pared for FEG-ESEM by cleaving a sub-sample then supporting the exposed fracture surface using vacuum cold-mounting with epoxy resin. The mounted fracture sur- face was then ?attened by grinding on staged wet-SiC wheels (P240, P400, P800, P1200), followed by washing with industrial methylated spirit (IMS), and polishing with 6 l m colloidal silica suspension. Polished samples were sputter coated with carbon (approx.

20 nm thickness) in an Edwards E306 vacuum coater. Porosity analysis by mercury intrusion porosimetry (MIP) was performed using a Micromeritics Autopore IV 9500.V1 with a maximum pressure of 60,000 psi enabling the measurement of pore diameters from 360 to 0.005 l m. Irregular sub-samples (approx. 3–4 g) were sub-sampled by cleaving from the 40 mm parent cube samples and placed in a 5cc solid penetrometer.

The contact angle and surface tension parameters were taken as 140 ° and 0.485 N/m 2 , respectively. The chemical composition of RPC samples was analysed using thermogravimetric analysis (TGA) using a TA Instruments SDT Q600. TGA samples were taken from the inner core of cube specimens and then ground (using ceramic mortar) to a ?ne powder.

Sub- samples of the prepared powder (20–30 mg) were placed in Ti pans. Heating was ramped from ambient (20 ° C ± 2) to 1000 ° C at a constant heating rate of 20 °C/ min in a 100% N 2 atmosphere. Bulk powder X-ray diffraction (XRD) patterns for each of the four treatment types were measured on a Siemens D500 diffractometer, operating in Bragg–Bren- tano focusing geometry and using Cu Ka radiation (k = 1.5418 Å) from a generator operating at 40 kV and 25 mA.

The diffractograms were recorded in a 2h range from 10° to 60° , with a 2h step size of 0.02 and a step time of 1 s. Samples were prepared by oven drying to constant mass at 70 °C, followed by manual crushing using an agate mortar & pestle. Samples were mounted by placing on a powder sample holder and levelled using a glass slide. 3. Results and discussion 3.1.

Mechanical properties The 7-day and 28-day compressive strengths of RPC samples for all treatments (A–D) are compared in Fig. 1. All samples produced a double pyramid shape after failure. The untreated mixture (A) pro- duced a compressive strength of 77.75 MPa at 7 days, which increased by 13% to 89.58 MPa at 28 days.

Following static pres- sure (C) treatment during setting, the 28-day compressive strength increased to 119.38 MPa, which is a 41% increase over its 7-day strength. Since pressure treatment during setting is likely to reduce the pore volume and pore diameter of entrapped air voids, we can hypothesise at this stage that the relatively small increase in 7-day strength between A and C results from some reduction in the diam- eter of macro defects (pores).

Correspondingly, this would theoret- ically allow a larger proportion of the total pore volume to be ?lled by C–S–H and associated Portlandite during hydration, and so could account for the large increase in 28-day strength for C com- pared to A [14]. Heat curing treatment increased the mean compressive strength by 32% without pressure (B) and by 41% with pressure (D) at 7-day.

However, at 28-day the compressive strength of both decreased by 5% for B and 16% for D. Heat curing has a very signif- icant effect on 7-day compressive strength (B and D), which is most likely related to acceleration of the pozzolanic reaction. In addition, RPC containing silica fume with low water–cement ratio increased the aggregate-mortar bond being more stable at high temperature [22].

Heat treated samples (with or without preceding pressure treatment) had reduced 28-day compressive strength compared to the 7-day strength. These phenomena may happen in concrete with high cement content and cured in high temperature [17] . It may be explained that heat curing forms products of a poorer physical structures (more porous) which remain un?lled after 7 d onwards.

It also induced a non-uniform distribution product within the paste because of the rapid hydration reduce the avail- able time to diffuse hydrate products away from cement particles. As a result, there is a concentration product in the surrounding of cement particles which may prevent the future hydration in long- term and reduce the strength.

In addition heat curing induced the formation of micro cracks that are known to initiate by thermal expansion during accelerated heat curing [23], particularly since RPC contains a high proportion of ?ne-grained materials (cement, SF, GGBS). As each element has different heat expansion coef?- cient, so then it also may left microcrack between them after heat- ing in ambient temperature.

This is partially supported by the data and observations made using MIP analysis (see: Section 3.2) but requires further study. The crystallisation process of hydration that continuously increases after 7 days [12] may cause further propa- gation of these micro cracks. Although the bulk porosity decreases following static pressure treatment, the volume of micro cracks apparently increases and this is exacerbated by heat curing.

With the ratio SF + GGBS/cement at around 1.0, there exists a large quantity of silicate in the mixture which requires time to react with free Ca and/or Portlandite. The 28-day uncon?ned compres- sive strength of both heat treated samples was higher than untreated (A) by 19% (B) and 39% (D). Microcracks formed after heat curing are in?uenced by the vol- ume change of RPC, and can be inhibited by adding ?bres with a high elastic modulus, e.g.

carbon ?bres [24]. Therefore we hypoth- esise that the use of carbon ?bre can avoid the small decrease in compressive strength normally observed at 28-days, and even increase the stiffness after 28-days in combination with a heat cur- ing accelerated pozzolonic reaction. 3.2.

Pore evolution The correlation between 7-day compressive strength and bulk density (determined gravimetrically by the water saturation method) is shown in Fig. 2. Heat curing increased the density by up to 5% (B), pressure treatment increased density by up to 7% (C), and pressure followed by heat treatment increased density by 12% (D).

The data suggests that heat curing increased density for both with and without pressure.

Previous scholars have sug- gested that heat curing can result in pore ?lling by pozzolan- derived C–S–H phases; predominantly tobermorite but xonotlite can form at temperatures greater than 200 °C [1,9,14]. The bulk Table 2 Mass composit ion of RPC mix per m 3 . PC (kg) SF (kg) GGBS (kg) Quartz (kg) Water (kg) SP (l) A C E 498 208 332 488 244 244 200 55 Table 3 Summary of experim ental data from mercury intrusion poro simetry (MIP). Parameters Treatment Unit A B C D Total intrusion volume 0.0153 0.0262 0.0309 0.0433 mL/g Total pore area 1.989 4.460 4.211 7.004 m 2 /g Median pore diameter (volume) 50.1 61.8 29.5 24.0 nm Median pore diameter (area) 14.2 8.6 21.7 21.0 nm Average pore diameter (4V/A) 30.9 23.5 29.4 24.8

nm Bulk density at 0.51 psia 2.2108 2.1825 2.1488 2.1107 g/mL Apparent (skeletal) density 2.2884 2.3151 2.3017 2.3232 g/mL Porosity 3.3921 5.7282 6.6452 9.1496 % Stem volume used 45 63 42 69 % 556 M. Helmi et al. / Construction and Building Materials 105 (2016) 554–562 powder XRD diffractograms in Fig. 3 appear to con?rm this process and shows loss of the portlandite peaks (2h = 18.1° and 28.7°) following heat treatment (B and D), along with reduction in alite/- belite peak intensity and occurrence (with signi?cant intensity) of the shouldered xonotlite peaks (2 h = 28.9° and 31.7°) adjacent to the main calcite peak (2 h = 29.5°). Filling would therefore be expected to occur within the capillary pores.

Since the speci?c gravity of these mineral phases varies signi?cantly (portlandite G = 2.33; C–S–H gel G = 2.6; tobermorite G = 2.43; xonotlite G = 2.70 [25,26]) it is reasonable to assume that the substitution of portlandite inclusions and highly alkaline pore ?uids (e.g. Ca (OH 2) rich in this case; pH > 12) by the C–S–H phases could result in an increased bulk density along with increased gel pore volume.

This is supported by the A–D increase in skeletal bulk density determined by MIP with simultaneous increase in capillary pore volume (< 50 nm) within the cement gel (see Table 3). Note that this observation is somewhat masked by the A–D overall decrease in gravimetric bulk porosity (see Table 4) that occurred due to pressure setting (i.e.

compression of entrapped air voids) and/or heat treatment (i.e. partial ?lling of air voids with C–S–H phases). The change in capillary pore volume within the gel, that results from mineral phase transformations and associated changes to the skeletal density, has been estimated in Table 5.

Using the speci- ?c gravity values stated above, the calculations assumed that 100% of the pre-heat treatment C–S–H mass was amorphous C–S–H gel and that the difference (reduction) between pre- and post-heat treatment C–S–H mass resulted from transformation to the higher density xonotlite. Post-heat reduction in portlandite mass was attributed to the pozzolonic reaction and transformation to the two C–S–H phases.

These calculations suggest that for B , c. 24% of the increased capillary pore volume could be to the heat- induced mineral phase transformations whilst for D this was only c. 2%. It is hypothesised that the remaining capillary pore volume could be attributed either to loss of dissociated water from tober- morite phases or to thermally-induced expansion of air within the capillary pores. Fig.

4 shows the cumulative intrusion against pore diameter obtained using MIP for the A, B , C and D treatments of RPC. The pore size distribution in zone 1 for A and B closely overlap by 22.87% and 21.73% suggesting that heat treatment has no Fig. 1. Compressive strength for treatments A–D after 7 and 28 days. Fig. 2. Density-compressive strength correlation at 7 days [17] . M. Helmi et al.

/ Construction and Building Materials 105 (2016) 554–562 557 significant effect on macro pore volume, e.g. by pore filling from hydration products. In addition the size distribution for C and D also closely overlap by 11.06% and 7.12% and cross the A and B line but have less pore volume. It suggests that pressure treatment reduces the volume of entrapped air voids above 10 l m diameter and additional heat treatment changes macro pore by filling them with hydration product.

These measurements are supported by SEM observations and direct measurement of representative entrapped air voids (identified by their rounded bubble-shaped morphology) as shown in Fig. 4(a) without pressure and (b) with pressure. Zone 2 highlights a significant increase in pore volume at approximately 2–3 l m diameter for treatment B.

This is thought to be due to the compressed air voids followed by accelerated heat curing resulting in expansion of the compressed gas inside pores which could result in extended cracking. This expansion does not appear in curve D supposed that pressure increases gel pore which connect each other and able to release the compressed air after heat curing.

Zone 3 represents the vast majority of pore volume and includes only sub-micron porosity in the anticipated range for cement gel at this water/binder ratio (w /c = 0.22; approx. 5–50 nm [27]). Heat treatment only (B) decreases pore volume in the entrapped and gel pore ranges compared to A (untreated); pressure treatment only (C) increases both small entrapped air pores and capillary pore volume by approximately 50%; and com- bination of both (D) appears to increase capillary pore volume sub- stantially in the range of 10–50 nm by around 70%.

We tentatively suggest that the formation of xonotlite, which results in the release of water molecules from tobermorite, could account for this increase [13]. This is partly (55%) due to the increased volume of micro cracking, and partly (45%) due to formation xonotlite and replacement of portlandite (pore ?lling) by higher density C–S–H phases in capillary pores. 3.3. Composition and ITZ Backscattered electron (BSE) micrographs comparing treat- ments C and D are shown in Fig.

5a and b. Z (atomic) number con- trast enables portlandite (CH), as part of hydration products, to be clearly distinguished as the bright artefacts within the intermixed C–S–H, ettringite, and AFm phases.

Both treatments resulted in similar mortar phase distributions of (i) fully hydrated cement indicated by smooth-textured and grey level uniformly; (ii) resid- ual cement grains marked by a hydration rim; (iii) belite marked by striations; and (iv) ettringite crystals [28,29] . Qualitative com- parisons between several representative regions suggest that port- landite occurs less frequently after treatment D than for treatment C indicating an acceleration of cement hydration. Micro cracks (marked by white circle in Fig. 5a and b of approximately 0.2

I m width occurred at (i) the borders of cement-coated grains, (ii) within the mortar phase, and (iii) emanating from entrapped air pores. The results for mass of hydration products (determined using TGA) are shown in Fig. 6. The recorded mass loss was assigned as follows: 30–105 °C due to evaporation of free (capillary) water; 110–170° C due to decomposition of ettringite; 180–300 °C due to partial dehydroxylation of C–S–H (possibly indicating partial transformation of C–S–H gel to xonotlite); 400–500 °C dehydroxy- lation of portlandite ($Ca(OH)_2$); 700–900 °C due calcination of $CaCO_3$ [13,30] and dehydroxylation of xonotlite.

The mass loss cal- culation of all treatments (A–D) in this study is shown in Table 6. Following heat curing the dehydroxylation/transformation of C– S–H reduced its mass by 0.296 wt% (B) and 0.252 wt% (D) com- pared to A and C , respectively. In the same cases, the amount of quantity of portlandite was reduced by 1.042 wt% (B) and 0.307 wt% (D).

If the wt% changes are used to calculate the relative increase density, assuming transformation of C–S–H (G = 2.34) to xonotlite (G = 2.70) and portlandite (G = 2.33) to tobermorite (G = 2.43), this gives increase in bulk density of 2.981% for B and 2.226% for

D. At temperatures well below the dehydroxylation of portlandite, heat curing most likely has accelerated the pozzolonic reaction to react portlandite with the amorphous silica contained within condensed micro silica to form additional calcium silicate hydrate (C–S–H) assuming the reaction given in Eq.

(2) [31]: SiO 2 þ 1 : 7Cað OHÞ 2 þ 2 : 3H 2 O) Ca0 1 : 7 Si0 2 _ 4H 2 O ð 2Þ Fig. 7 presents the quantitative EDS results as atomic ratios that were calculated and plotted from the mean average for each sam- ple with statistical variance error bars. To distinguish the hydrates formed based on EDS, the classi?cations obtained using Eqs.

(3)– (5) were assumed [32]: C—S—H 0 :8 6 Ca= Si 6 2 : 5; ðAl þ FeÞ = Ca 6 0: 2 ð 3Þ CH Ca= Si P 10; ðAl þ Fe Þ= Ca 6 0: 4; S= Ca 6 0: 04 ð 4Þ AFm Ca= Si P 4 ; ð Al þ Fe Þ =Ca > 0: 40; S =Ca > 0: 15 ð 5Þ Fig. 3. XRD spectra for treatment A– D. Table 4 Gravimetrically-determined bulk density and porosity. Parameters Treatment Unit A B C D Density (SSD) 2168 2257 2250 2358 kg/m 3 Density (dry) 2070 2172 2209 2325 kg/m 3 Porosity 4.54 3.75 1.83 1.40 % Table 5 Summary of TGA inferred mineral transformations with corresponding capillary pore volume (in cement gel) increase.

Items Mass loss (wt%) A B C D C–S–H gel 0.997 0.701 0.946 0.694 Xonotlite 0 0.296 0 0.252 Portlandite 1.611 0.569 0.865 0.558 Total C–S–H 0.997 2.039 0.946 1.253 Pore volume (ml/mg) MIP: V p,gel 7.58 10.30 23.59 37.10 TGA: D V p,gel 0 0.78 0 0.32 558 M. Helmi et al. / Construction and Building Materials 105 (2016) 554–562 The atomic ratio of C–S–H and CH for all treatments (A–D) asa function of distance from the aggregate boundary (10 l m–50 l m) appeared to be insigni?cant (see Fig. 7). Within this region the occurrence of C–S–H is signi?cantly more dominant than that of CH within the paste.

This is most likely because the ITZ forms dur- ing cement paste (‘wall effect’) packing around the aggregates (grain) boundaries, the pores of which are subsequently ?lled by much smaller diameter SF grains; themselves latterly taking part in the pozzolonic reaction [33,34]. Local porosity was analysed inside grid areas of 5 _ 50 l m and the mean average of pore area fraction was taken.

ITZ thickness was de?ned as the distance from the aggregate edge where the porosity curves intersect with the observed porosity of the bulk paste (see Fig. 8a). The local porosity near to the aggregate bound- ary is slightly higher and decreases with distance towards the bulk paste. Fig. 8b shows that all treatments had similar slope trend lines resulting in an intercept (ITZ thickness) of 17–18 l m, but with different total porosity values (A = 4.3%; B = 3.4%; C = 5.4%; D = 5.3%). Static pressure treatment reduced ITZ thickness by a sta- tistically insigni?cant amount from 17.3 l m(A) to 16.9

l m(C), as did heat curing but for an increase up to 17.9 l m(B). It seems that the treatment which accelerated the hydration in RPC had no sig- ni?cant effect on ITZ thickness, elemental composition or local porosity. This is a signi?cant ?nding because it shows that pressure treat- ment, when applied during setting, does not adversely affect the degree of porosity within the ITZ phase rather it appears to affect the mortar phase by the same degree. 4.

Conclusions The processing of an RPC mixture by static pressure treatment (during setting), followed by heat curing (during hardening), resulted in a m ultiscale transformation with respect to micro structural composition and pore geometry. This had a signi?cant effect on the macro scale physic-mechanical properties but that does not involve any apparent alteration of the ITZ phase.

At ambient temperature (A), the crystallisation process of hydration in RPC naturally continued after 7-days and increased the compressive strength by 13% at 28-days due to the hardening proses. Although the RPC mixture in this study used ?ner Fig. 4. MIP cumulative intrusion-pore diameter plot. Cement pore size range classi?cation is interpolated from Domone (1999) [22] for a w / c of 0.18.

Part (a) shows example of entrapped air pores up to 200 l m diameter, identi?ed by their rounded bubble-shaped morphology. Following pressure treatment during setting, the very large pores appear to be compressed to <50 l m diameter (b), and (c) shows example micro cracks observed during heat curing. Fig. 5.

SEM micrograph showing typical micro cracks observed after heat curing, commonly originating both at the ITZ (a) and at the periphery of connecting pores (b). M. Helmi et al. / Construction and Building Materials 105 (2016) 554–562 559 materials, the uniformly hydrated mortar phase contained ran- domly distributed pores with diameters varying between 0.005 and 200 l m.

Hardened RPC also has a high amount of anhydrous materials due to the very low water–binder ratio and initial micro- cracks caused by the shrinkage process in cement paste. After static pressure treatment (C), the 28-day compressive strength increased by 33% compared to the untreated (A), which has a strong correlation with the observed changes in microstruc- ture.

Static pressure decreased the modal pore diameter by an order of magnitude with a corresponding reduction in total pore volume and the quantity of free water. This process also increased the capillary pore volume in the range <0.05 l m diameter, perhaps caused by dilation within the paste during setting. Pressure treat- ment should theoretically arrest shrinkage crack propagation and substantially reduce the volume of void macro defects.

However, it appeared to have no signi?cant effect on apparent ITZ width, which suggests that only porosity within the mortar phase was altered. Heat treatment alone (B) resulted in a greater increase in 7-day strength compared to untreated (A) and pressure treatment (C). However, this treatment reduced compressive strength by 5% at 28-days compared to 7-days.

Heat curing reduced the quantity of portlandite, most likely due to acceleration of the pozzolanic reac- tion that transforms portlandite to tobermorite. Inside the gel pore network, heat curing appears to induce further crystalline hydrate formation with subsequent increases in gel skeletal density (i.e. hydration reaction) and transformation of the existing crystalline hydrates; namely portlandite ? tobermorite and tobermorite ? xonotlite.

Regarding strength degradation, it suggests that the Fig. 6. Decomposition mass loss for treatments A –D determined using TGA. Table 6 Mass loss (wt%) for key temperature ranges determined by TGA analysis. Temp (° C) AB C D 30–105 1.052 0.845 0.884 0.763 110–170 1.028 0.592 0.901 0.650 180–300 0.997 0.701 0.946 0.694 400–500 1.611 0.569 0.865 0.558 700–900 1.077 0.913 0.859 0.945 30–1000 8.515 5.959 7.361 5.892 Fig. 7.

Spatial distribution of atomic ratio across the ITZ as a function of distance from an aggregate grain boundary determined using quantitative EDS. 560 M. Helmi et al. / Construction and Building Materials 105 (2016) 554–562 proportion of competent material has reduced due to further microcrack propagation. Logic suggest that heating would acceler- ate the propagation of microcracks (formed during shrinkage) due to thermal expansion of the solid phases as well as volumetric expansion of the air (and hence increased pressure) within entrapped voids.

In addition, heat curing also had no signi?cant effect on composition as a function of depth within the ITZ region (e.g. the Ca/Si atomic ratio) when compared to the mortar phase. Heat curing preceded by pressure treatment (D) resulted in the highest compressive strength at 7-days, but decreased by 16% at 28-days.

Pressure treatment ?rstly decreased the entrapped air void volume and densi?ed the cement gel as described in treat- ment C. Then both the hydration and pozzolonic reactions were accelerated and tobermorite was transformed by heat treatment, as explained for treatment B. The formation of additional porosity in the range 1–3 l m after heat treatment only (B) was not observed when heat treatment was preceded by pressure setting (D).

Since each transformation results in the dissociation of water and a signi?cant increase in speci?c gravity (and hence skeletal density) the gel pore volume is substantially increased in the range <0.05 l m. The decrease of strength at 28-days suggests that microcrack propagation is likely to have resulted from pressurisa- tion of entrapped air voids, i.e.

from compression followed by ther- mal expansion. This combination of treatment results in a microstructure that exhibits the highest early-age and late-age compressive strength for an RPC mix. Future research will focus on the interaction of ?bres within RPC microstructures to alleviate the issue of late-age microcracks propagation.

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Aitcin, Characterization of the granular packing and percolation threshold of reactive powder concrete, Cem. Concr. Res. 30 (12) (2000) 1861–1867 . Fig. 8. (a) Example plot of mean porosity as a function of distance (treatment D) with interpolated ITZ distance. (b) Bulk porosity as a function of distance from an aggregate grain boundary determined using segmentation image analysis of BSE micrographs. M. Helmi et al.

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