# Treatment effects on the compressive strength of Reactive Powder Concrete (RPC) at 7 days

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**ABSTRACT**

Reactive powder concrete (RPC) is produced by controlling three main factors: additives to mix composition, pre-setting application of pressure, and post-setting heating. Densification of the mortar is achieved by the application of pressure (either unidirectional static load or omnidirectional air pressure load) in order to minimize the presence of macro defects in the form of entrapped air voids. Heat curing is applied after final setting with temperatures of at least 90 °C in order to accelerate the hydration and pozzolanic reactions. The purpose of this research was to characterize the relative effect of these three treatment approaches on the compressive strength of RPC at 7 days. The variables assessed in this study include heating rate, treatment ~~curing~~ types; and with/ without static pressure (8 MPa). The results show that a heating rate of 50 °C/hr preceded by pressure application for 2 days were the optimum conditions for statically-pressed RPC samples. Assuming variables of 8 MPa static pressure and 2-day heat curing at 240°C, compressive strength increased by: 6 % using static pressure only, 60 % using heat curing only, and 83 % using both static pressure and heat curing. Further work will investigate the micro structural and chemical composition within the interfacial transition zone and mineral product evolution during hydration in combination with high temperature/ pressure curing conditions.

## 1. INTRODUCTION

Concrete's widespread use can be attributed to the low cost of materials, construction process, and maintenance (Neville, 2011) and yet it constantly faces ever more complex problems of application in the construction sector leading to increasing demand for high concrete performance. This requirement can often be achieved through the use of pozzolanic/ latent cementitious mineral additives such as silica fume, fly ash, ground granulated blast furnace slag, and natural pozzolans (Larrard & Sedran, 2002; Long et al., 2002; Liu et al., 2000).

Reactive powder concrete (RPC) is ultra-high performance concrete (UHPC) which is produced by controlling three main factors: composition, pressure during curing and post-set heat curing (Richard & Cheyrezy, 1995). Composition improvement is obtained by a microstructural engineering approach; by elimination of coarse aggregates, reduction of water-binder ratio and lowering the $CaO-SiO_2$ ratio through the introduction of silica-rich pozzolana (Bonneau et al., 2000; Matte & Moranville, 1999).The addition of steel fiber reinforcement adds significant ductility to the densified mortar (Chan & Chu, 2004). Pressure applied to the fresh mixture makes the RPC denser, while heat curing accelerates both the pozzolanic reaction between amorphous silica and calcium hydroxide as well as the silicate and aluminate hydration reactions. By implementing these techniques, the mechanical properties of RPC can achieve ultra high performance, such as a compressive strength ranging between 200 - 800 MPa, a fracture energy of up to 40,000 $J/m^2$, and an ultimate elongation of up to 0.007 $m/m^{-1}$ (Richard & Cheyrezy, 1995).

CEM I 52.5 MPa Portland cement is one of the main constituents of the RPC microstructure typically at a dosage between 800-1000 $kg/m^3$. This high content affects not only the production cost but also the heat of hydration leading to potential risks associated with shrinkage and creep-induced strain. The addition of fibre reinforcement (commonly steel) can offset these problems whilst contributing to ultra-high mechanical properties and strain capacity which enables very thin components to be manufactured (Richard & Cheyrezy, 1995; Yazici et al., 2009). For these reasons, Yazici et al (2008) conducted research on a number of cements to find the optimal amount of additive silica fume (SF) and pulverised fly ash (PFA), or ground granulated blast furnace (GGBS). These results suggested that the composition of RPC containing 35wt% silica fume and up to 40wt% GGBS could give compressive strengths of more than 200 MPa.

Yazici et al (2010) used an autoclave to apply curing treatments at 2.0MPa setting pressure and 210 °C air temperature (after demoulding). These conditions were reached within a 2.5 hr heating period after which heat curing was applied for 8 hrs. A steam curing method (95% RH, 90 °C) was used by Cwirzen (2007) to study the optimal conditions for heat curing;

the starting time at which heat is applied after demoulding and the duration of heating. Heat treatment was initiated at 24, 48 and 168 hours after casting, whilst total heating duration was applied at 1, 2 and 4 days for each starting time. The result appeared to suggest that the optimal conditions for heat curing are a total duration of 48 hours initiated after 2 days of setting (after casting) since these conditions resulted in the highest measured degree of hydration. Other methods using a drying oven as the heat curing equipment has been applied by Sadrekarimi (2004) and Tam et al (2010) with different rates and maximum temperatures. Sadrekarimi (2004) cured samples with a constant heat rate of 10 °C/hr to 240 °C, whilst Tam et al (2010) placed samples in a drying oven initially at 100 °C then increased the temperature by 50 °C/hr to reach of 250 °C . The high temperatures attributed both to the progression of cement hydration and to the pozzolonic reaction of with silica fume (amorphous) and quartz flour (crystalline). In heat curing, the variables of temperature, duration and heating rate are very important since they impact the crystallisation of hydrates, pozzolonic reaction, microstructure development and associated bulk mechanical properties (Tam and Tam 2012).

Sadrekarimi (2004) states that RPC has potential to be applied in several types of precast structures, such as prestressed structures, pressure precast pipes, or impermeable container for hazardous fluid or nuclear waste. Blais & Couture (1999) claim that the world's first major structure built with RPC is the Sherbrooke pedestrian/bikeway bridge in Canada. The bridge having 60 m of span applied structural innovations in the confinement of concrete which is without any steel bar reinforcement. Another commercial application was to produce the roof structure of a train station; claimed to be the thinnest precast concrete structure using RPC (Vicenzino et al., 2005) having dimensions 5 x 6 m by 20 mm thick and supported by a single column. Since RPC structures will be produced by precast methods, the early age strength is an important factor in practice. This paper presents the effect of treatments on compressive strength and properties of RPC at 7 days by considering firstly the optimal condition of heating rate and pressure duration.

## 2. METHODOLOGY
The mix ingredients comprised Portland cement (PC), silica fume (SF), slag (GGBS), quartz sand, and super-plasticizer (SP). The cement type of CEM1 52.5 (Rugby, UK) was used according to BS EN 197-1:2011. The cement replacement materials comprised silica fume, which was microsilica grade 940-D (Elkem, Switzerland), Ground granulated blast furnace slag (GGBS) (Hanson, UK), and quartz sand (David Ball Ltd., UK) conforming to BS 1881-131 and containing the grades C, D, and E mixed in a ratio of 2:1:1 by mass. A high range water-reducing admixture based on a polycarboxylate polymer was used to increase the workability. The composition of RPC used

was adopted from Yazici et al (2008) as presented in Table 1. In this study SF content was reduced to 25% of the initial cement content, the water-binder ratio was increased to maintain workability, and no fibre reinforcement was added.

Table 1: Composition of RPC per cubic meter

| PC (kg) | SF (kg) | GGBS (kg) | Quartz (kg) | | | Water (kg) | SP (ltr) |
|---|---|---|---|---|---|---|---|
| | | | C | D | E | | |
| 498 | 208 | 332 | 488 | 244 | 244 | 200 | 55 |

The process of mixing followed the steps of Yazici et al. (2008). Firstly, materials were mixed in the dry condition at a speed of ~120 rpm for 2 mins, then the speed was increased to ~450 rpm for a further 2 mins. Finally, the water and SP were premixed and added to the mixer incrementally with further mixing for 10 - 12 mins until the mixture was consistent.

The treatments of this study are modified from previous references (Yazici et al., 2008; Cwirzen, 2007; Sadrekarimi, 2004; Tam et al., 2010) in terms of pressure and heating methods. Firstly 8 MPa pressure was applied by static loading on the fresh mixture about 5 hrs after casting (after initial set). Secondly, heat curing was applied using a drying oven where samples were preheated at 40 °C for 2 hrs before increasing to 240 °C at a rate of 50 °C/hr. Heat curing was applied at 240 °C for 48 hrs before decreasing to 40 °C. After this step, curing was continued in water for 2 days at 20 °C. The heating rate was defined from the preliminary study on two types of specimen, pressure treated and non-pressure treated.

Four combinations of treatments were applied in this study: without pressure and cured in water (A); without pressure and heat cured in a drying oven (B); with pressure and cured in water (C); with pressure and heat cured in a drying oven (D).The specimens used to determine the compressive strength and other properties were 40mm cubes measuring in accordance with BS EN 196-1:2005. All data presented is the mean average of data for three representative samples.

## 3. RESULT AND DISCUSSION

3.1 Effect of heating rate and pressure duration
Both types of samples were heated in a drying oven at different rates: 10, 50 and 100 °C/hr up to a maximum temperature of 240 °C. The result for the optimal rate was then applied to a study of pressure duration with variables of 1, 2 and 3 days applied pressure. The result of compressive loading- at 7 days is presented in Table 2 where NP and WP denotes samples that were non-pressured and with pressure, respectively.

A heating rate applied at 10 °C/hr may lower the dehydration rate by allowing sufficient time for thermal diffusion (Sadrekarimi, 2004). Using this rate the compressive strength was strong enough at 7 days

with 79 MPa for non-pressured and 100 MPa with pressure. By increasing the rate to 50 °C/hr, compressive strength increased by 41% (NP) and 48% (WP) compared to that achieved at 10 °C/hr. This be because the higher rate of heating accelerated the hydration reaction(s) and pozzolanic reactions. When the rate was increased to 100 °C/hr, the compressive strength was similar to that achieved using 10 °C/hr heating rate. It is suggested that this may have occurred because the quick heating rate imposed water content to evaporate immediately from CSH gel and induced high pressure in the pores. It is feasible that this pressure might increase the number of, or widen, micro cracks within the hardened cement paste. In addition, it is suggested that the more rapid heating rate may have induced a significant number of micro cracks (Richard & Cheyrezy, 1995) perhaps resulting in the lower observed compressive strength and so requiring further research.

For all three heating rates, pressure treatment combined with heat curing could increase the compressive strength more than 30% comparing to the ones non-pressure treated.  This percentage is similar to result in Yazici et al (2008) and higher than in Sadrekarimi (2004) who observed increments of 30 % and 21%, respectively. Richard & Cheyrezy (1995) state that the pressure applied on the plastic mixture before and during setting has three effects: reducing the volume of entrapped air voids, removing excess pore water, and decreasing the bulk porosity.

Pressure duration has a direct effect on the start time for heat curing, because samples are heated only after demoulding. For the mix design used in this study,  the optimum pressure duration was found to be 2 days resulting in an unconfined compressive strength almost three times that of non-pressured samples (Table 2). Although pressure treatment may change the physical microstructure by densification (Richard & Cheyrezy, 1995), in the absence of heat treatment it appears to have no significant effect on the compressive strength. Illston (1996) explains that, during the first day of hardening, tobermorite [C-S-H] gel is formed in much higher quantities than ettringite and portlandite [$Ca(OH)_2$] and starts to form $C_4AF$ hydrate. During the second day, the volume of hydrated phases increases and the volume of ettringite decreases as it converts to monosulphate, where during the third day the density of hydrate phases begins to increase significantly. The optimum time at which to apply heat treatment to enhance compressive strength is 2 days after casting where the majority of ettringite has converted to monosulphate and prior to the onset of hydrate phase densification. The authors tentatively suggest that this could be due to heat-induced acceleration of hydrate phase densification whilst avoiding the risk of microcrack initiation/ enlargement resulting from thermal expansion of partially-hardened hydrate phases.

Table 2: 7-day compressive strength (MPa) at different heating rates and durations of applied pressure

| NP | WP | NP | WP | NP | WP |
|----|----|----|----|----|----|
| Heating rate | | | | | |
| 10 °C/h | | 50 °C/h | | 100 °C/h | |
| 79 | 100 | 112 | 148 | 76 | 104 |
| Duration of applied pressure | | | | | |
| 1 day | | 2 days | | 3 days | |
| 43 | 51 | 112 | 148 | 101 | 104 |

## 3.2 Effect of treatments on density and porosity

Density and bulk porosity are properties of conventional concrete that are inversely related to one another and with strong empirical correlation to compressive strength (Neville, 2010). However, the properties of RPC appear to be significantly more sensitive to the temperature and heating rate during curing. The pressure treatment impacts significantly on the compacting of microstructure (Richard & Cheyrezy, 1995) while heat curing influences the crystal type formed during hydration (Tam & Tam, 2012). In this study, density and porosity were determined gravimetrically. The relationship between treatment types A to D and the porosity/ density, along with mean compressive strength, are given in Figures 1 and 2.
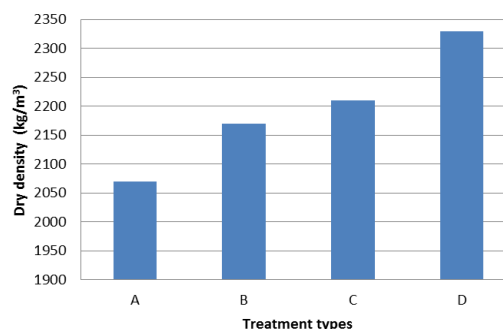


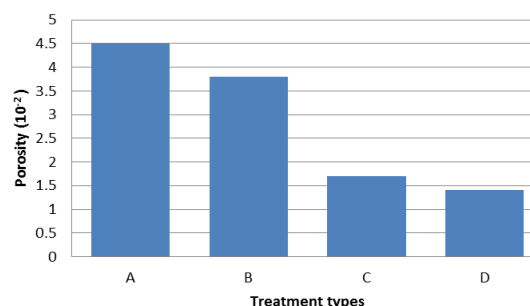Figure 1: Correlation between treatment types and dry bulk density



Figure 2: Correlation between treatment types and bulk porosity

The results appear to show that pressure-only treatment (A and C) increased the bulk density by 6.5%, but significantly reduced the bulk porosity by 62.2% primarily by reduction of entrapped air volume though expulsion of free water. Previous studies have shown that spherical entrapped (air-filled) pores can occur in RPC despite the high percentage of

superplastizer and typically range in diameter from 10 to 300 μm (Yaziqi et al., 2008). The application of pressure during setting appears to reduce both the diameter and volume of these entrapped air pores, which densifies the matrix and increases bulk density as confirmed by a separate study (Yaziqi et al., 2010). It is known that pressure applied to fresh concrete can reduced the water/binder ratio by around 4% (Sadrekarimi, 2004). When the pressure is released after final setting, this can further reduce the bulk porosity due to expansion of the decompressed aggregates (Richard & Cheyrezy, 1995).

While heat curing treatment only (B), the density increased by 4.8% and porosity decreased by 15.6%. These changes in properties are similar percentages to those of treatments C and D with an increase of 5.4% for density and a reduction of 17.6% for bulk porosity. Richard & Cheyrezy (1995) and Hong & Glasser (2004) all observed that heat curing can accelerate the pozzolonic reaction between amorphous silica and calcium hydroxide leading to rapid formation of CSH gels. Spherical pores (caused by air entrapment) in RPC are generally air-filled or with some calcium hydroxide crystals following standard curing, but can be filled with tobermorite (C-S-H) structures in the form of needle-like crystals when autoclave cured (Yazici et al., 2010). For curing at temperatures greater than 200°C, crystals of needle-shaped xonotlite are present together with plate-shaped tobermorite (Tam & Tam, 2012). This can lead to reductions in measured bulk porosity by pore filling with the CSH product and consequently alters the pore size distribution by reducing the median pore diameter (Cwirzen, 2007).

3.3 Effect of treatments on compressive strength

The 7-day compressive strengths of RPC samples for all treatments (A-D) are compared in Figure 3. The untreated mixture (A) produced a compressive strength of 78 MPa which, according to Neville (2010), is classed as a high performance concrete and was used as the reference mix. The static pressure-only (C) treatment increased mean compressive strength by 6% (see Figure 4), which was lower than the variance for the sample set and therefore insignificant. Heat curing increased the mean compressive strength by 60% when applied without pressure (C), and by 83% when applied with pressure (D). These significant increases are most likely related to an increase in the early-age formation of portlandite (from calcium silicate hydration) to feed the pozzolanic reaction, followed by significant temperature-induced acceleration of the latter. The hypothesis from Figure 3 is that high temperature curing, when preceded by static pressure application during setting, results in further matrix densification due to pore-filling by tobermorite/ xonotlite formation from the pozzolanic reaction. This would reduce macro defects and enhance the paste-aggregate bonding mechanism resulting in a higher measured compressive strength.
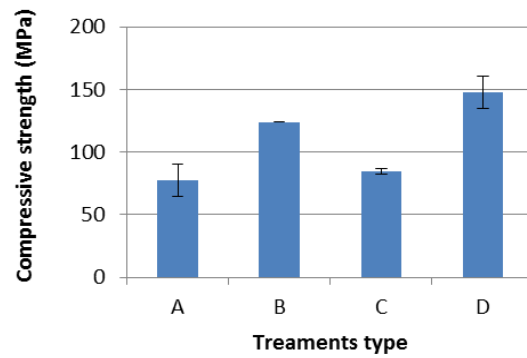


Figure 3 Effect of treatments on compressive strength at 7 days.

Neville (2010) presents the well-known correlation between bulk porosity (log scale) and compressive strength for plain concrete in Equation 1:

$$f_c = f_{c,0} (1 - \rho)^n \qquad \text{Eq. 1}$$

Where: $\rho$ = porosity, $f_c$ = compressive strength, $f_{c,0}$ = strength of paste at zero porosity, and $n$ = a scaling integer. This assumes that $f_{c,0}$ equals 500 MPa for cement paste at w/c = 0.45 (Neville, 2010). This results in scaling coefficients, $n$ of 40.5 (A), 38.8 (B), 103.7 (C), and 86.4 (D). Despite strong empirical correlations between bulk porosity and compressive strength for conventional concrete (Neville, 2010), the results here for RPC show that the correlation integer depends upon the treatment applied (Figure 4). When $f'/ \rho$ was plotted using $n$ = 40.5 (determined for treatment A by Eq.1), the curve intersects the data for treatment B, and where $n$ = 103.7 (determined for treatment C) the curve intersects the data for treatment D.
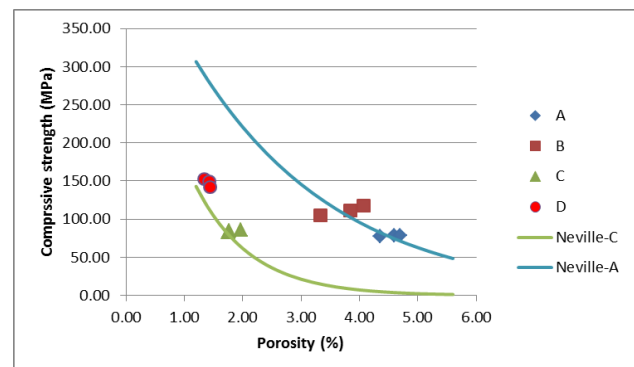


Figure 4 Correlation between porosity and compressive strength each treatment

The effect of both treatments concerning spherical entrapped air pores and pore-filling by crystalline phases can be illustrated using the simple conceptual model in Figure 5. It assumes that a uniformly hydrated matrix contains randomly distributed spherical pores with diameters varying between 10 and 300μm when cured under ambient temperature/ pressure conditions (Fig. 5a). When static pressure is applied to a fresh mixture, the total pore volume and the mode pore diameter both decrease (Fig. 5b). Heat curing accelerates the pozzolanic reaction in an

already densified cement matrix leading to pore filling by crystalline hydrate (tobermorite and/ or xonotlite) formation (Fig. 5c). Since heat curing increases the air pressure inside pores, the hydrated matrix outside of pores becomes dense. Further research is currently focusing on determination of the process-structure-property relationships within this conceptual model for RPC mixtures.
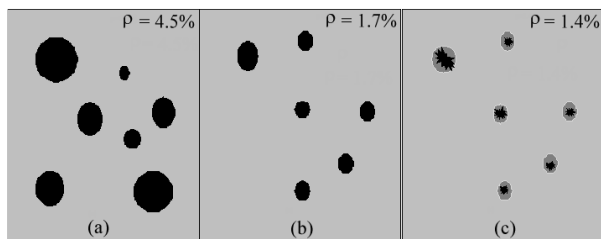


Figure 5 Conceptual model of curing treatment effects on spherical entrapped air pores in RPC: (a) standard curing, (b) pressurized setting, (c) pressurized setting followed by heat treatment during hardening

## 4. Conclusions

A heating rate of 50°C/hr during hardening and static pressure treatment at 8 MPa for 2 days was suitable for producing good quality RPC samples. The curing conditions were intended to increase early age strength by accelerating alite and belite hydration, and the pozzolonic reaction between amorphous silica (from SF and GGBS) and portlandite. The results appear to show that heat treatment during the hardening stage has a significant effect on the early age strength, whereas static pressure treatment during the setting stage has an insignificant effect. When heat treatment is applied after treatment by static pressing, the strength increases by a further significant amount. In an $f'/\rho$ plot, the integer, $n$ for non-pressure treated (A) and pressure-treated (C) samples were significantly different, but the measured compressive strength was not. When heat treatment was applied in both cases (A and B; C and D) the heat treated samples fitted the same $f'/\rho$ plot as the non-heat treated. This could be consistent with alteration of pore network geometry (resulting from entrapped air) resulting in (i) increased pore air pressure by reduction in mode pore diameter, (ii) reduction in microcracks within cement matrix, and (iii) change in macro defects stress distribution. For treatments B and D, this suggests that the heat treatment results in a significant macro pore filling effect maintaining consistency in the $f'/\rho$ relationship. This is supported by gravimetric data for bulk porosity and bulk density. Further research will test the conceptual model for different heat treatment rates and static pressures, supported by characterization of the pore network and mineralogy.

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