

MICROSTRUCTURAL AND PHYSICAL ALTERATIONS OF REACTIVE POWDER CONCRETE AFTER HIGH TEMPERATURE EXPOSURE

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ABSTRACT

Reactive powder concrete (RPC), which contains no coarse aggregate and has a higher proportion of fine particles (<600 μm), is categorized as an ultra-high performance concrete (UHPC). This paper presents the effect of high temperature exposure on RPC performance in terms of compressive strength and microstructural alteration. Our results indicated that the exposure progressively decreased the compressive strength of RPC and correlated to both the shrinkage and thermal characteristics of the aggregates and the matrix. At a temperature of 400 °C, the exposure induced mass loss from the hydrates by dehydroxylation, coarsening of the capillary pores, and induced micro cracks between unhydrated cement grains, hardened paste and aggregate. Exposure at 500 °C further increased mass loss and developed the micro cracks in terms of length and width. At 800 °C, hydrate mass loss sharply increased by 84 %, the C-S-H was substantially degraded, and extensive micro cracking appeared throughout the specimens including within the fine quartz aggregates.

Keywords: Reactive powder concrete, durability, high temperature, pore coarsening.

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1. INTRODUCTION

The durability of concrete generally correlates with its ability to withstand the deleterious effects of ambient exposure, including interaction with liquids, gases and heat [1-2]. There are two factors influencing the durability: (i) mechanical damage relating to the loss of integrity of concrete, and (ii) chemical damage related to degradation of its constituents leading to strength loss over time [3]. High temperatures may be applied to concrete during the setting process or curing periods, or during post-hardening exposure. All have significant effects on the physical and chemical properties of concrete. Many scholars have found that concrete can often maintain adequate residual strength even after several hours of high temperature

exposure [4-13]. The residual strength is mainly affected by cement paste chemical composition, aggregate properties, and the applied temperature [10]. The mechanisms of property alteration are complex and correlate with chemical and physical processes occurring within the hardened cement paste, and in the interfacial transition zone (ITZ) [12].

In general, research has shown that the microstructure was altered in the following ways such as mass loss, appearance and colour, spalling, and composition change. Poon *et al.* [6] and Abid [13] studied fibre reinforced high-performance concrete after high temperature exposure and using two different fibre types: steel and polypropylene. They found that steel fibre significantly contributed to the residual compressive strength. Chen & Liu [9] used three types of fibres (steel, polypropylene and carbon) and found that fibres with a high modulus of elasticity can bridge thermally-induced cracking more effectively.

Reactive powder concrete (RPC) containing no coarse aggregate and a higher proportion of fine particles (<600 μm is categorized as high performance concrete (HPC). The performance of RPC often is developed by controlling three main variables: composition, pressure during the setting period, and post-set heat curing [14]. The properties of RPC relating to the bonding between fiber and paste have been examined by [15] and the use of industrial waste as cement replacement by [16]. Reference [7] studied the effects of fire duration at a fixed level of 500 ± 50 °C at 30, 60, 90 and 120 minutes of exposure. They concluded that the residual compressive strength of RPC was significantly higher (about 20%) than other concretes. Tai *et al.* [8] evaluated the mechanical properties of RPC by placing samples in a high temperature electric furnace and heating them at a rate of 2 °C/minute, then maintained the temperature for 30 minutes every 100 °C and then increased incrementally up to 800 °C. They summarized that compressive strength increased gradually and linearly with temperature until 300 °C, then dropped sharply by 80% at 800 °C. At a temperature of 800 °C, RPC becomes brittle and contains more pores and large cracks and the colour became a darkish brown. Pore coarsening is believed to be an important factor in causing the decrease of strength of RPC, as observed by other scholars [17-19].

Reference [16] studied the composition of RPC mixtures using GGBS as partial cement replacement by 40% and resulted in compressive strength >200 MPa. This study is interesting because it removed the problem of a high cement content in RPC due to early shrinkage and hardened performance. Therefore, it is necessary to reproduce their work with some modifications. These may include by applied static pressure during the setting period to get a denser matrix, applying heat curing in an oven after hardening and using carbon fibre to reinforced at the micro scale.

High temperature can change the chemical composition and physical structure of hardened concrete, which also relates to the durability of concrete structures. It appears that study on this area is open for further exploration mainly due to its complex factors, which differ from those of common concrete. Lots of other people have studied this already but we are going to study it as well. Although the properties of RPC under elevated temperature have been studied by several scholars, it is still necessary to quantify the effect of high temperature on the microstructural properties of RPC containing different amounts of ultrafine powder and carbon fibre. As this mixture incorporates a large proportion of ultrafine powders resulting in very high density, it was hypothesized that the residual compressive strength would also be high. In addition, the use of carbon fibre in RPC mixture can also increase the residual compressive strength.

2. MATERIALS AND METHOD

The cement type used in this study was CEM I 52.5 (Cemex, UK). Admixture powders were condensed micro silica grade 940-D (Elkem, Switzerland) and ground granulated blast furnace slag (Hanson, UK). The fine aggregate was quartz sand and mixed in a ratio of 2:1:1 (by mass) from three grades of A (2.36 mm - 1.18 mm), C (0.60 mm - 0.30 mm) and E (0.15 mm - 0.09 mm), respectively (David Ball Ltd., UK). Admixture liquid was used to reduce water content and to increase workability based on a polycarboxylate polymer (Grace Construction product Ltd., UK). The high performance carbon fibre had the following properties: grade L; diameter = 7 μm ; tensile strength = 4.9 GPa; tensile modulus = 230 GPa; elongation = 2.10%; density = 1.8 g/cm^3 (Soficar S.A., France). The fibre was originally in a thin bundle and was cut to 12 mm lengths.

The composition (by mass) of the RPC mix adopted from previous study [20] with additional carbon fibre is presented in Table 1 which was mixed in several steps. Initially, all dry materials were put in a bowl, mixed at a speed of ~ 120 rpm for 2 minutes and then increased to ~ 450 rpm for another 2 minutes. Half of the volume of required water and SP were then added and mixed for about 5 minutes until the mixture became consistent. The carbon fibre was added incrementally into the mixture at a lower speed. The rest of the water was then added and the mixing speed increased to ~ 450 rpm for a total 12-16 minutes. After the mixture became consistent, it was cast into an oiled steel molding for casting into three prisms with dimension: 40 x 40 x 160 mm, then vibrated on a table for 3 minutes. After casting, a static pressure of 8 MPa was applied directly onto the surface of the specimens for 5 hours, followed by de-molding after 2 days. Then heat curing cycle was applied as follows: preheated at 40 $^{\circ}\text{C}$ for 2 hours before increasing to 240 $^{\circ}\text{C}$ at a rate of 50 $^{\circ}\text{C/hr}$; cured at 240 $^{\circ}\text{C}$ for 8 hrs; decreased at a rate of 50 $^{\circ}\text{C/hr}$ to 40 $^{\circ}\text{C}$. After this step, heat-cured samples were stored in water at 20 $^{\circ}\text{C}$ prior to high temperature exposure testing. After 27 days, RPC specimens were lifted from the curing water and dried at ambient temperature. High temperature exposure of the prisms was achieved by placing them in an oven at 400 $^{\circ}\text{C}$, 500 $^{\circ}\text{C}$, and 800 $^{\circ}\text{C}$ (at 28 days) following the process in [8]. The 40 mm cube specimens were used to determine compressive strength in accordance with BS EN 196-1:2005. All strength data presented is the mean average for three representative samples.

Microstructural characteristics were analyzed by mercury intrusion porosimetry (MIP) using a Micromeritics Autopore IV 9500 with a maximum pressure of 60,000 psi (enabling the measurement of pore diameters from 360 – 0.005 μm) and thermo-gravimetric analysis (TGA) using a TA Instruments SDT Q600. MIP samples were irregular sub-samples (approx. 3-4 g) which were cleaved from specimens after exposure and placed in a 5cc solid penetrometer. The Hg contact angle and surface tension parameters were taken as 140 $^{\circ}$ and 0.485 N/m^2 , respectively. TGA samples were extracted from the inner core of cube specimens after compression testing and ground using a ceramic mortar & pestle. 20–30 mg of powder was placed in Ti pans and heated to 1000 $^{\circ}\text{C}$ at a constant heating rate of 20 $^{\circ}\text{C/min}$ in a 100% N_2 atmosphere.

Table 1 Mass composition of RPC mix per m^3

PC (kg)	SF (kg)	GGBS (kg)	Quartz (kg)			Water (kg)	SP (l)	Carbon fiber (kg)
			A	C	E			
498	208	332	488	244	244	200	55	1.8

3. RESULTS AND DISCUSSION

3.1. Alteration of Porosity

The high temperature exposure induces the coarsening of pore size and then influences the properties of concrete, such as strength, durability, and permeability [17]. The effect of exposure on pore coarsening development can be analysed by MIP in terms of pore size distribution and total porosity [18]. Table 2 shows the changes of porosity and average pore diameter pore which indicates the coarsening of RPC after exposure. Regarding to the median pore diameter of reference sample, it increased slightly at 400 °C, almost 3 times at 500 °C, and by about 18 times at 800 °C. It suggests that the exposure enlarges the measured pores size due to the formation of micro cracks. In addition, the total porosity also increased which may correlate with crack progression and decomposition of hydration products such as, i.e. ettringite at 110 °C, C-S-H at 180 °C, and Ca(OH)₂ at 450 °C. The release of water molecules from these hydrates is likely to induce secondary porosity within the hardened paste.

Figure 1 shows that the high temperature exposure has significantly changed the measured pore volume and size distribution. The total porosity at 400 °C slightly increases due to the enlargement of capillary pores and corresponding reduction pores with a diameter less than 0.05 µm. The volume of pores greater than 0.1 µm in diameter increased due to crack propagation, observed chiefly in the paste and ITZ region. Morsy *et al.*, [19] found that both paste and quartz aggregate expand significantly by 500 °C but at different rates to one another, whilst quartz can partially decompose at temperatures of 580 °C. Differential thermal expansion is therefore the most likely cause of the increased cumulative pore volume for these two exposure temperatures micro cracking on the surface of aggregates during the cooling process [21-22]. After exposure at 800 °C, the changes in pore diameter for the ranges <0.1 µm and >10 µm were quantified as 28.3 % and 43.2 %, respectively. It supposes relating to have many pores and a rough, grainy surface, and also the increase of cracks number [11].

Table 2 Alteration of pore properties after high temperature exposure

Properties	Unit	20°C	400°C	500°C	800°C
Total intrusion volume	mL/g	0.024	0.025	0.030	0.100
Median pore diameter (volume)	µm	0.012	0.018	0.031	2.054
Average pore diameter (4V/A)	µm	0.010	0.011	0.014	0.101
Bulk density at 0.51 psia	g/mL	2.221	2.205	2.130	2.017
Apparent (skeletal) density	g/mL	2.352	2.331	2.280	2.258
Porosity	%	5.377	5.571	6.586	20.208

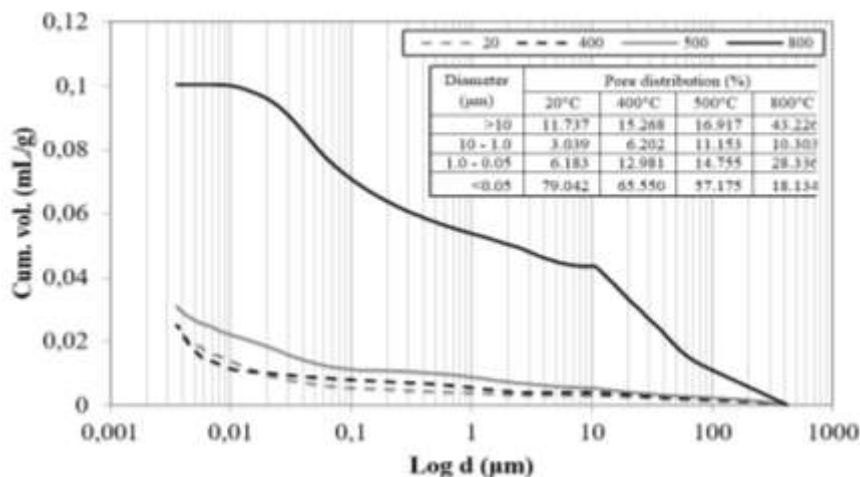


Figure 1 Distribution and proportion of pores in different temperature of exposure

3.2. Alteration of Hydrates Composition

The results of thermogravimetric analysis for both pre- and post-exposure samples is shown in Figure 2 which indicates the changes in the composition of hydrate phases. Mass loss between temperature 30 °C and 105 °C corresponds to evaporable (capillary) water and not considered part of the decomposition process. At higher temperatures, mass loss occurs in three distinct stages [12]. Firstly, between 110 and 300 °C mass loss occurs due to dehydroxylation of several hydrates, *i.e.*, decomposition of ettringite, loss of water from aluminate hydrates, and decomposition of the C-S-H. It also may be due to the evaporation of capillary water from macro capillary pores and evaporation of gel water from gel pores of cement paste [10]. Secondly, at temperatures between 450 and 500 °C mass loss corresponds to the dehydroxylation of portlandite. Thirdly, at temperatures above 600 °C mass loss occurs due to dissolve of crystal water and cement hydrates with the appearance a large amount of rose bush-like structures which is similar to monosulfate aluminate [12]. The alteration of hydrate at 600-750 °C is due to the dehydration of residual portlandite and C-S-H and also decomposition of calcite [10]. The calcite decomposition is at temperature above 800 °C due to a crystallization process of wollastonite [10, 12, 22].

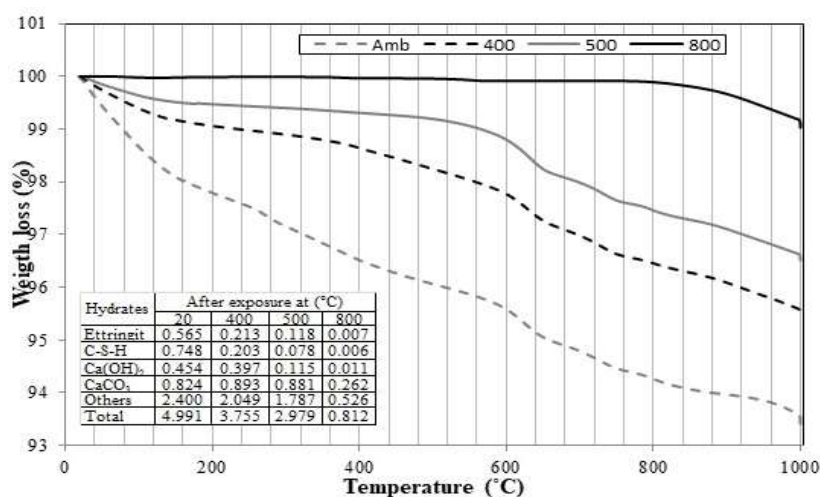


Figure 2 Mass loss of main hydrate in RPC after high temperature exposure

The analysis of mass loss for three high temperature exposure were also based on these three regions. The mass loss in first region is by 0.416 %, 0.196 % and 0.001 % for temperature of 400 °C, 500 °C, and respectively. The last value at 800 °C indicates that exposure of the RPC at this level caused the total evaporation of water in ettringite and C-S-H. In second region, the amount of portlandite decrease by 0.397 % for temperature of 400 °C, by 0.011 % for temperature of 500 °C and by 0.115 % for temperature of 800 °C which is due to dehydroxylation reaction. The curves indicate that the mass loss after exposure at temperature 400 °C and 500 °C are similar to the ambience's curve, however it decreases sharply to 70 % after exposure at 800 °C. It indicates that exposure RPC at 800 °C caused the decomposition of most hydrates. In last region, the total mass loss is by 25 %, 38 % and 84 % for temperature 400 °C, 500 °C and 800 °C respectively.

3.3. Alteration of Surface Colour

High temperature exposure has an effect on both the crack morphology and the surface colour of concrete. The colour changes of RPC specimens after high temperature exposure were captured by a digital camera as shown in Figure 3. The colour change was recorded immediately after exposure and cooling to ambient temperature. Cross-sectional images were recorded of post-failure fracture surfaces following compression testing.

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Some macro defects were observed on the specimen surfaces prior to exposure in the form of entrapped air voids and fine crazing. The colour in cross section shows that part of the specimen appeared to have a lighter grey colour towards the surface and to a depth of approximately 1-2 mm. The lighter grey was supposed due to decomposition of ettringite, loss of water from aluminate hydrates, and decomposition of the C-S-H as mentioned in TGA test. This sequence can result a more porous and weaker structure in hardened cement paste [23]. The curing treatment at 240 °C mostly had no effect on the quartz aggregate colour; yellowish brown, most likely because quartz material chemically will transform at temperatures of about 500 °C [8].

The colour of the RPC specimens at 400 °C exposure changed to a lighter grey colour both on the surface and inside due to dehydroxylation of portlandite. Some smooth cracks appeared on the surface occurred due to the contrast in thermal expansion coefficients between quartz aggregate and cement paste [8]. The lighter colour outer specimen slightly increased by 3-4 mm of thickness in cross section and the damage gradient was also different. It supposes that this happened due to the increase in temperature gradient between the surface and the inside of the concrete [24].

At 500 °C, the colour of specimen became a darkish grey and the cracks on surface sharply increased in both width and length when it was compared to the ones at 400 °C. The exposure at this temperature induced deeper colour alteration in the outer part of the cross section of specimen in about 4 - 5 mm. It is suggested due to furtherance of the same chemical alterations described above at 400 °C. In addition, the colour of quartz aggregates became to a darkish brown due to chemically transformation of quartz material. Furthermore, micro cracks appeared on the surface of the aggregate due to difference of thermal expansion coefficient between quartz and paste which then induced paste spalling on the specimen surface.

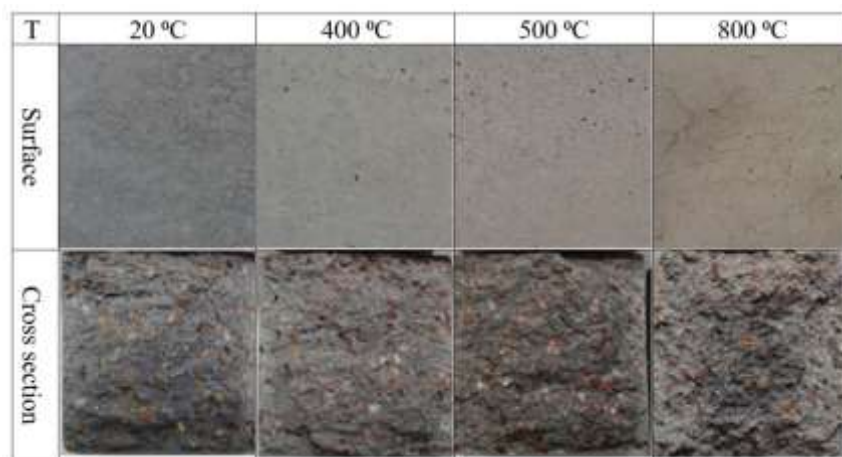


Figure 3 The colour change on the surface and cross section of RPC specimens

When the temperature exposure increased to 800 °C, the colour of the surface turned to brownish grey. The cracks appeared on the specimen surface and increased in terms of width, length and number. The surface appeared some large pores and turned to friable due to spalling of thin paste around quartz aggregate. This condition probably correlated to the formation of an impermeable wall for gases, which then induced small ‘explosions’ inside the pores due to pressurization of the voids from thermal expansion of the gas [25]. Furthermore, the micro cracks formed at 500 °C near the surface of the specimen induced the cement paste to detach easily after exposure to 800 °C. In addition, this exposure turned the matrix colour in cross section to a light grey with a depth around 12-15 mm.

3.4. Residual Compressive Strength

The durability properties of RPC after exposure to fire commonly are indicated by residual compressive strength [7]. The compression test results for the mean of three specimens at all levels of temperature exposures are presented in Figure 4. The mean compressive strength for the control samples was 138.68 MPa, i.e. for specimens set under a static load of 8 MPa for 48 h then cured at 240 °C for 48 h.

The compressive strength decreased by 6.71 % and 8.91% following exposure at 400 °C and 500 °C, respectively. It is suggested due to the dehydroxylation of the hydrates, *i.e.* ettringite, C-S-H and CH. However, the compressive strength decreased by 74.33 % after exposure at 800 °C. It is supposed due to decomposition process which breaks down the C-S-H [3]. It can be noted that temperatures exposure up to 500 °C have decomposed the ettringite, C-S-H and portlandite but slightly influences the compressive strength. While exposure of RPC at 800 °C it decreases compressive strength sharply due to coarsening of pores in the paste and degradation of the cement chemical bonding that lead to separate the finest aggregate and cement [7, 26].

Strength loss due to high temperature exposure occurs in three stages: (i) evaporation of water within the concrete starting at 105 °C which induces shrinkage and increased pore pressure leading to cracking and spalling; (ii) differential expansion between the cement paste and aggregate starting at c. 500 °C resulting in thermal stresses and cracking within the ITZ; and (iii) breakdown of hydrates (decomposition) starting at c. 700 °C [3]. It is correlated to two mechanisms; shrinkage and thermal expansion behaviors [27]. High temperature exposure may also result in partial transformation of the C-S-H gel phase to tobermorite and/or xonotlite.

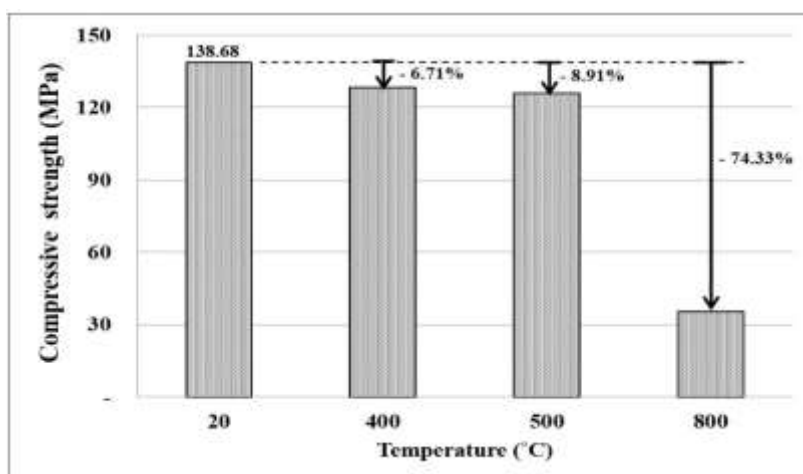
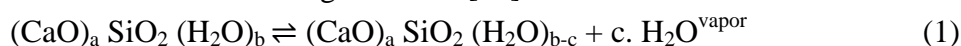


Figure 4 Residual compressive strength of RPC after high temperature exposure

The rapid evaporation of capillary water can generate high pore pressure resulting in explosive spalling of concrete. This mechanism is dominated by the dehydroxylation of C-S-H and CH with the following reactions [28]:



The observed relationship between residual compressive strength and exposure temperature are in close agreement with those of previous studies for both fibre reinforced concrete and RPC [6, 8-9], as shown in Figure 4. The trend line for the results in this study are described by Equation 3, where f'_c represents the compressive strength for RPC before exposure and f'_{cT} for those after exposure. [8]. The use of carbon fibres in RPC mixtures may

be advantageous due to the very small diameter of carbon fibres (7 μm) and good fibre dispersion to strengthen all parts of the paste uniformly. In addition, carbon fibres may provide a bridging mechanism to hold the spread of micro cracks in the paste without yield up to 800 $^{\circ}\text{C}$ during high temperature exposure [7-9].

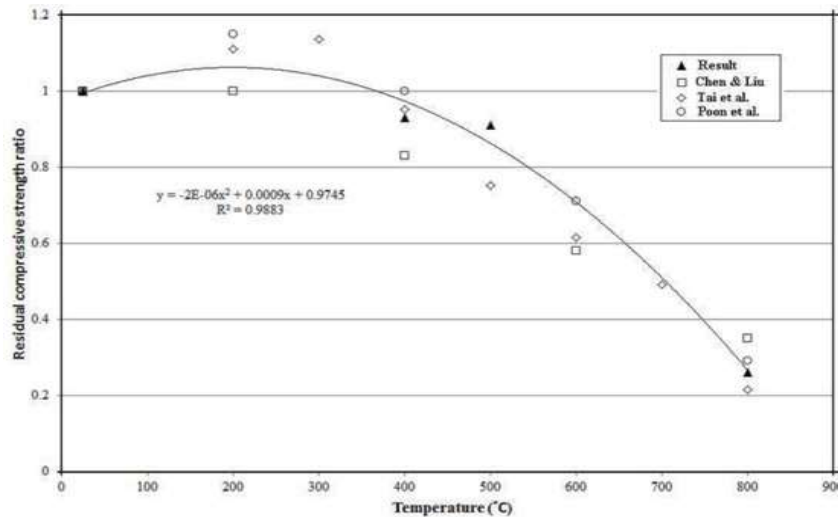


Figure 5 Ratio of residual compressive strength compared to other scholars

$$\frac{f'_{cT}}{f'_c} = 2 \times 10^{-6} T^2 + 9 \times 10^{-4} T + 0.9745 \quad (3)$$

Where the early application of heat resulted in a significant increase in compressive strength, it is hypothesised that this occurred due to acceleration of the pozzolonic reaction, as well as transformation of tobermorite to the denser xonotilite phase [8].

4. CONCLUSION

The high temperature exposure strongly influenced the microstructural and physical properties of RPC due to the mechanism of crack progression, hydrate decomposition and thermal expansion behaviours of the aggregates and matrix. At temperature of 400 $^{\circ}\text{C}$, the exposure reduced total mass loss of hydrates by 25 % through dihydroxylation; appeared the coarsening of the capillary pores; and induced micro cracks between unhydrated grains, paste and aggregate. At temperature of 500 $^{\circ}\text{C}$, the total mass loss of hydrates increased by 38 % with further extensions of micro cracks in terms of length and width. At the highest temperature (800 $^{\circ}\text{C}$), the hydrates loss sharply increased by 84 %; cement bonding degraded in the C-S-H; micro crack appeared in specimen entirely including within the quartz aggregates. High temperature exposure decreased the compressive strength of RPC by between 6 and 9 and 74 % at temperatures of 400 $^{\circ}\text{C}$, 500, and 800 $^{\circ}\text{C}$, respectively. The presence of ultrafine powders and carbon fibre in RPC mixture have less effects on the residual compressive strength

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