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# The effect of autoclave pressure, temperature and duration time on mechanical properties of reactive powder concrete

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## HIGHLIGHTS

- ▶ Mechanical properties of RPC were investigated under autoclave curing.
- ▶ The effect of temperature, pressure and duration time was investigated.
- ▶ Mechanical performance of RPC increased considerably after autoclaving.
- ▶ There is an optimum autoclave duration time for each pressure and temperature level.
- ▶ SEM micrographs and mercury porosimetry results revealed dense microstructure of RPC.

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## ABSTRACT

The mechanical properties (compressive and flexural strength) of reactive powder concrete (RPC) have been investigated under autoclave curing and compared with standard water curing condition. Three significant parameters in autoclave curing have been investigated; autoclave temperature, pressure and time. The effects of silica fume and steel microfibers incorporation on mechanical performance were also studied. Test results indicate that; compressive strength of RPC increases significantly after autoclaving compared to the standard water curing. Autoclave time, temperature and pressure also influenced the mechanical performance of RPC considerably. It can be said that, there is critical duration time for each pressure and temperature conditions. The mechanical properties can be influenced negatively beyond this value. Also, SEM micrographs and mercury porosimetry results revealed the transformation of microstructure of RPC caused by autoclaving process.



## 1. Introduction

Reactive powder concrete (RPC) is new generation cement based composite and it was developed by microstructure enhancement techniques. The high compressive strength values of RPC over 200 MPa and its remarkably increased durability properties are mainly based on its dense microstructure. This structure is a result of high cement content, very low water cement ratio, the inclusion of highly reactive silica fume, proper granulometric adjustment of fillers, low $CaO/SiO_2$ ratios by addition of silica components, and incorporation of steel microfibers [1–4]. The low permeability, dense microstructure and superior mechanical properties (very high compressive strength, flexural strength, fracture energy, and toughness) classify RPC as an ultra high performance concrete [5]. Nowadays, RPC seems to be a promising material for special precast concrete production industry. This material can also be used in industrial and nuclear waste storage constructions [1–5].

Thermal curing has a strong effect on mechanical properties of concrete. Major effect is the development of a denser microstructure with the formation of calcium silicate hydrate (C–S–H) phases, which results in higher mechanical properties. Similar to conventional concrete heat curing or autoclaving of RPC generates an obvious change in the microstructure. Autoclave curing leads to the formation of tobermorite at temperatures below 200 °C. And xonotlite forms temperatures about 250 °C in pure heat treatment process without steam [6]. Owing to the very high cement content and low water/binder ratio, the majority of cement grains remain unhydrated after 28 days in RPC. The pozzolanic reaction resulting from the reaction between portlandite and silica fume is also not complete owing to the low water content that hinders the silica fume dissolution [7]. Under heat treatment the gradual silica fume dissolution and its reaction with the portlandite forms new hydrates, which is also C–S–H [8,9]. Cement paste consists of close networked crystal fibers with a length up to one micrometer in

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RPC samples cured at 200 °C and 15 bars [4]. Furthermore, dissolution processes around quartz grains in autoclaving was reported, which produced a better cohesion between the fillers and the fine crystalline cement paste [4]. On the other hand, autoclaved specimens that only contain Type I Portland cement were found to have very low compressive strength. In order to enhance the compressive strength, silicon compounds are required that can change the hydration reaction under autoclaving conditions [10–12]. Yazıcı et al. [13–16] showed that Portland cement and silica fume can be replaced with slag and/or fly ash at different proportions as potential silica sources.

The effects of pressure, temperature level and curing time on the mechanical properties of autoclaved RPC have been investigated in this study. Test results were compared with standard water cured control specimens. The effect of silica fume and brass coated steel fibers incorporation under autoclaving have also been studied. Findings were supplemented by the microstructural studies.

## 2. Experimental

RPC mixtures were prepared with: Ordinary Portland cement (CEM-I 42.5-R); quartz powder (0–0.075 and 0–0.4 mm) and quartz sand (0.6–1.2 and 1.0–3.0 mm, with a specific gravity of 2.65). Silica fume (SF), a polycarboxylate based superplasticizer (SP) in conformity with ASTM C 494-81 type F. Brass-coated steel microfibers (6 mm long, diameter of 0.16 mm, and aspect ratio, specific gravity and tensile strength of 37.5, 7.17 and 2250 MPa, respectively) were used in 2% by total volume. $SiO_2$ content of quartz aggregates was 99.65% (by weight) according to the results of XRF analysis. The physical, chemical and mechanical properties of cement and silica fume were presented in Table 1. Table 2 summarizes the compositions of RPC mixtures. As can be seen from Table 2, abbreviations were used for mixtures according to cement, silica fume and fiber content.

The ingredients (i.e. cement, quartz powder, quartz sand with/without silica fume) were initially dry mixed for about 5 min at low (140 rpm) and high speed (285 rpm) in a mixer. Half of the mixing water was added and re-mixed for about 3 min at low and high speed. The remaining water and super-plasticizer were added and re-mixed for about 5 min at high speed (up to 285 rpm). Finally, fibers were added and additional mixing was applied for about 5 min. The specimens were kept in the molds for 16 h at room temperature of about 20 °C in a high humid environment. After this period RPC specimens were removed from the steel molds. One group of RPC specimens were kept in water at 20 °C, the other group was autoclaved under different autoclave conditions (1 MPa–180 °C, 2 MPa–210 °C, 3 MPa–235 °C). The heating rate of autoclave cure treatment was 1.1 °C/m and the curing periods were chosen as 4 h, 6 h, 10 h, 12 h and 24 h. The specimens that were subjected to heat treatment were kept in laboratory conditions for cooling before testing.

Prismatic specimens (40 × 40 × 160 mm) were used to determine the flexural strength. The specimens were loaded from their mid-span and the clear distance between simple supports was 130 mm. The compressive strength test was performed following the flexural tests. The two broken pieces left from flexural test were subjected to compressive strength test. The loaded area under compressive load was 40 × 40 mm and the height of the specimens was also 40 mm. Each data presented here is the average results of three test specimens.

**Table 1**
Physical, chemical and mechanical properties of cement and silica fume.

| | Cement | Silica fume (SF) |
|---|---|---|
| *Chemical composition (%)* | | |
| $SiO_2$ | 20.10 | 92.26 |
| $Al_2O_3$ | 5.62 | 0.89 |
| $Fe_2O_3$ | 2.17 | 1.97 |
| CaO | 62.92 | 0.49 |
| MgO | 1.14 | 0.96 |
| $Na_2O$ | 0.30 | 0.42 |
| $K_2O$ | 0.85 | 1.31 |
| $SO_3$ | 2.92 | 0.33 |
| $Cl^-$ | 0.001 | 0.09 |
| Loss on ignition | 3.84 | – |
| Insoluble residue | 0.63 | – |
| Free CaO (%) | 0.52 | – |
| *Physical properties of cement* | | |
| Specific gravity | 3.13 | |
| Initial setting time (min) | 130 | |
| Final setting time (min) | 210 | |
| Volume expansion (mm) | 1.00 | |
| *Specific surface ($m^2$/kg)* | | |
| Cement (Blaine) | 380 | |
| *Compressive strength of cement (MPa)* | | |
| 2 days | 29.9 | |
| 7 days | 43.2 | |
| 28 days | 51.9 | |

**Table 2**
Mixture compositions of RPC.

| Material (kg/$m^3$) | C | C-f | SF | SF-f |
|---|---|---|---|---|
| Cement | 772 | 772 | 772 | 772 |
| SF | 0 | 0 | 179 | 179 |
| 1–3 mm Quartz | 736 | 709 | 622 | 596 |
| 0.6–1.2 mm Quartz | 294 | 284 | 249 | 238 |
| 0–0.4 mm Quartz | 294 | 284 | 249 | 238 |
| 0–0.075 mm Quartz | 147 | 142 | 124 | 119 |
| Water | 138 | 138 | 138 | 138 |
| SP | 29 | 29 | 35 | 35 |
| Steel fiber | 0 | 143.5 | 0 | 143.5 |
| Water/cement | 0.18 | 0.18 | 0.18 | 0.18 |
| Water/cement[a] | 0.20 | 0.20 | 0.20 | 0.20 |
| Water/binder | 0.18 | 0.18 | 0.15 | 0.15 |
| Water/binder[a] | 0.20 | 0.20 | 0.16 | 0.16 |

[a] Calculated with total water (water + water from SP).

## 3. Results and discussion

### 3.1. Flexural properties of RPC

Flexural strength values of mixtures after standard water curing were presented in Fig. 1. It can be seen that curing time, fiber reinforcement and SF contents are important parameters in terms of flexural performance. All mixtures have rather high flexural strength values (over 10 MPa) even at 1 day. Silica fume (SF) inclusion increased the flexural strength of RPC mixtures especially beyond 1-day. This increment is 8–15% parallel to the curing age for C and SF mixtures. Similar results were recorded (11–15%) for SF-f and C-f mixtures. Steel fibers (2% by total volume) have also improved the flexural performance significantly for both control (C) and SF mixtures. These increments are between 25% and 49% for C mixture and 29–48% for SF mixture. Furthermore, the strength ratio of 1-day/28-day is between 47% and 61% according to the mixture type after standard water curing.

Flexural strength values of mixtures after different autoclave regimes were presented in Fig. 2a–d. The strength values of mixtures before autoclaving have also represents 1 day strength values (0 h autoclave time). As can be seen from autoclave time, pressure and temperature influences the flexural strength. SF and fiber content were also significant parameters. Flexural strength increases with steel fiber incorporation about 41–59% and 36–63% for C and SF

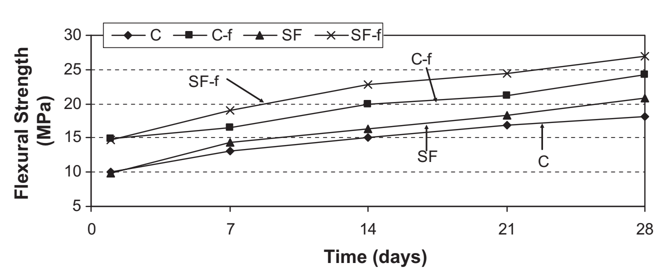


**Fig. 1.** Flexural strength gain of RPC mixtures after standard water curing within time.