

## **Effects of Different Water and Super Plasticizer Amount, Pre-Setting and Curing Regimes on the Behavior of Reactive Powder Concrete**

**Dashti Rahmat Abadi, M.A.<sup>1\*</sup>, Haji Kazemi, H.<sup>2</sup> and Shahabian, F.<sup>3</sup>**

<sup>1</sup> Ph.D. Student, Department of Civil Engineering, Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran.

<sup>2</sup> Professor, Department of Civil Engineering, Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran.

<sup>3</sup> Associate Professor, Department of Civil Engineering, Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran.

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**ABSTRACT:** Reactive Powder Concrete (RPC) is an ultra high performance concrete which has superior mechanical and physical properties. The RPC is composed of cement and very fine powders such as crushed quartz (100–600  $\mu\text{m}$ ) and silica fume with very low water/binder ratio (W/B) (less than 0.20) and Super Plasticizer (SP). The RPC has a very high compressive and tensile strength with better durability properties than current high performance concretes. Application of very low water/binder ratio with a high dosage of super plasticizer, different heat curing processes and pre-setting pressure improve mechanical and physical properties of RPC. In this study, the RPC is composed of available materials in Iran. Two different mixing proportions, different water/binder ratios for preparation of samples, different super plasticizer dosages, five different (0, 25, 50, 100 and 150 MPa) pre-setting pressure and 7 different curing regimes were used in samples preparation and experiments. Results showed that appropriate water/binder ratio and super plasticizer dosage, higher temperature and pre-setting pressure increase the workability, density and compressive strength of compositions.

**Keywords:** Compressive Strength, Heat Curing, Pre-Setting Pressure, Reactive Powder Concrete, Workability.

### **INTRODUCTION**

Reactive Powder Concrete (RPC) is a general name for a class of cementitious composite materials developed by the technical division of Bouygues, S.A. in the early 1990s (Richard et al., 1994). It is characterized by extremely good physical properties, such as strength and ductility. The ultra-high performance of RPC in

mechanical properties, particularly in compressive strength, makes it of tremendous interest to construction practitioners. The RPC is a relatively new kind of ultra high performance concrete. Its main features include a high percentage of cement, very low water-to-binder (cement + silica fume) ratio, a high dosage of super plasticizer, and very fine crushed quartz and

\* Corresponding author E-mail: dashti@iauyazd.ac.ir

silica fume. Coarse aggregate is completely replaced by fine quartz sand.

The compressive strength of RPC is about 150–800 MPa, while its tensile strength changes between 25 and 150 MPa. Moreover, the specific gravity of RPC varies between 2.4–2.8 t/m<sup>3</sup> (Richard et al. 1994). Fine powders such as crushed quartz (100–600 µm) are used instead of coarse aggregates in order to increase the homogeneity of RPC. Adding silica fume to the RPC reduces the total pore volume of the cement paste as well as the average diameter of the pores (Zanni, 1996; Nassif, 2005).

Furthermore, it has been reported that the application of different heat cure processes improve the mechanical properties of RPC substantially after the application of 50 MPa pre-setting pressure to a fresh mixture (Cheyrezy et al., 1995; Sadrekarimi A., 2004; Glasser et al., 2003). The application of pressure to fresh RPC during setting phase for 6–12 hours can eliminate some amount of pores caused by shrinkage. Micro cracks in fresh RPC are improved after discharging the applied pressure (Cheyrezy et al., 1995; Zhanga et al., 2002).

Dugat et al. (1996) applied 60 MPa pre-setting pressure to samples of fresh RPC with heat curing at 90 °C and 250 °C. They reached compressive strength of about 500 MPa and static Young's modulus of 36,000–74,000 MPa for RPC. Bonneau et al. (1997) studied confinement behavior of the RPC in a steel tube. They reported that compressive strength up to 200 MPa could be achieved in hot water curing at 90 °C and in low-pressure steam chambers at the precast plant. Teichman and Schmidt (2004) studied structural properties of the RPC and its effects on strength and durability. They used steam curing for 2 days at 90 °C and heat curing for 7 days at 250 °C after 2 days of de-moulding. In

their study fresh RPC samples were exposed to pre-setting pressure of 50 MPa. The highest compressive strength as 487 MPa was reported.

Yazici (2007) replaced Portland cement in the RPC with fly ash and granulated blast furnace slag at percentages of 0, 20, 40, 60 and 80%, respectively. Three different curing methods (i.e. standard, autoclave and steam curing) were applied to the specimens. He achieved compressive strength up to 185 MPa when the cement content of these mixtures was only 340 kg/m<sup>3</sup>. Topçu and Karakurt (2005) applied pre-setting pressure of 2.5 MPa to the RPC mixture. These specimens were exposed to steam curing for 7 days at 250 °C and then kept in water for 7 days at 90 °C. Compressive strength up to 253.2 MPa and flexural strength up to 63.67 MPa were obtained.

Massidda et al. (2001) studied the effects of autoclaving on the physical and mechanical properties of the RPC reinforced with brass-coated steel fibers. Autoclaving improved flexural and compressive strength of the RPC. High pressure steam curing for 3 hours yielded flexural strength of 30 MPa and compressive strength of 200 MPa. Shaheen and Shrive (2006) investigated freeze–thaw resistance of the RPC. Test results showed that the RPC has excellent freeze–thaw resistance even up to 600 cycles (according to ASTM C 666). Rougeau and Borys (2004) showed that the RPC can be produced with fly ash, limestone microfiller or metakaolin.

In this study, RPC samples were made using local available materials in Iran. Excel computer software and basic principles were used to determine the mixture ratios. Compressive strength of the samples prepared according to these mixing ratios was determined and the sample with the highest compressive

strength value was selected. Some moulds and a compression machine were especially designed for application of pre-setting pressure process. The effects of different W/B ratio, SP dosage, curing regimes (water, hot water, steam) and pre-setting pressures (25, 50, 100 and 150 MPa) during setting phase to the RPC in order to improve its physical and mechanical behaviors were investigated.

**EXPERIMENTAL PROCEDURE**

**Materials**

The choice of cement is an important factor because the cement type has a very high effect on the performance of RPC. The ideal cement must have a high C<sub>3</sub>S and C<sub>2</sub>S (di- & tri-calcium silicate) and low C<sub>3</sub>A (tri-calcium aluminate) content. This is understandable because C<sub>3</sub>A has little intrinsic value as a binding agent and is primarily included in cement due to its role as a flux during the calcination process (Kejin and Zhi, 2003). The RPC considered here, is prepared by ASTM Type 2 Portland cement from Abadah Company in Iran which is formulated specifically for a low C<sub>3</sub>A content.

Silica fume (SF), which is an industrial product of the manufacturing and purification of silicon, zirconia and ferro-silicon alloys in submerged-arc electric

furnaces has been used for RPC. It fills micro voids and produces secondary hydrates by pozzolanic reaction. The SF was provided from Azna Company in Iran. The chemical compositions (%) of cement and silica fume are presented in Table 1.

Quartz sands (with a maximum particle size of 0.6 mm and 0.3 mm) and quartz powder (with a maximum particle size of 0.1 mm) with a density of 2.75 t/m<sup>3</sup> were used for preparing RPC mixtures. The very low water/binder ratios used in the RPC would be possible by using a large amount of high-quality super plasticizer (about 4% of cement amount). A polycarboxylate based super plasticizer Glenium-55p was used for this purpose. This super plasticizer had fluidity within time of fresh concrete and high strength in a short time.

**Sample Preparation**

The Mooney suspension model and Fuller method were used for RPC mixture design (Kejin and Zhi, 2003; Talebinejad et al., 2004; Larrard and Sedran, 1994; Mooney, 1951; Furnas, 1931). Two mixtures having the highest compressive strength were selected and used to prepare the samples. One mixture was applied to prepare non pre-setting pressurized specimens and the other was used for pre-setting pressurized specimens. The mix proportions of RPC are shown in Table 2.

**Table 1.** Chemical compositions of cement and silica fume (%).

Component	Cement	Silica Fume
CaO	62.10	1.02
SiO <sub>2</sub>	20.92	95.10
Al <sub>2</sub> O <sub>3</sub>	4.61	0.60
Fe <sub>2</sub> O <sub>3</sub>	4.16	1.10
MgO	2.75	0.60
K <sub>2</sub> O	0.59	-
Na <sub>2</sub> O	0.3	-
SO <sub>3</sub>	2.02	1.20
C <sub>3</sub> S	51.14	-
C <sub>2</sub> S	21.42	-
C <sub>3</sub> A	5.18	-
C <sub>4</sub> AF	12.66	-

**Table 2.** Mix proportions of RPC (kg/m<sup>3</sup>).

Material	Mix.1 (non pressurized)	Mix.2 (pressurized)
Cement	936	1037
SF	280	300
0-1 mm Quartz	374	410
1-3 mm Quartz	268	290
3-6 mm Quartz	268	290
Water	234	165
SP	28	42

High shear mixing can enhance flow properties. To increase the shear mixing, one method is to use a mixer with a higher speed. A mixer with a speed as high as 2000 revolutions per minute (rpm) should be used in order to effectively break down the agglomerated particles of very fine materials of RPC. A high speed mixer was designed as shown in Figure 1.



**Fig. 1.** High speed mixer.

For each type of the proposed RPC mixtures, dry ingredients (i.e. cement, quartz powders, quartz sand and silica fume) were first mixed for about 3 minutes at low and high speeds. Water and super plasticizer were added and re-mixed for about 5 minutes at a high speed.

### Test Method

High compression strength of the RPC limits the dimensions of specimens. Some special moulds and a compression mechanical jack were designed to apply the pre-setting pressure. The equipments are shown in Figure 2.



**Fig. 2.** Moulds and compression machine.

At the end of mixing period, the workability of mixtures was assessed according to the ASTM C 1437 "flow table test". The fresh RPC was compacted into 50 mm cube moulds by hand tamping in two layers. The different pre-setting pressures (i.e. 25, 50, 100 and 150 MPa) were applied to the fresh RPC using the special machine. The specimens were allowed to harden in their moulds for 24 hours at 21°C and 95% relative humidity, before being stripped and subjected to one of the different curing regimes.

Different curing regimes which were applied to the samples are presented in Table 3.

**Table 3.** Sample codes and curing regimes.

Sample Code	1 day	Cure Type		
		2 - 4 days	5 - 7 days	8 - 28 days
W20	in mould	20°C W	20°C W	20°C W
3W60	in mould	60°C W	20°C W	20°C W
6W60	in mould	60°C W	60°C W	20°C W
3W90	in mould	90°C W	20°C W	20°C W
6W90	in mould	90°C W	90°C W	20°C W
3S90	in mould	90°C S	20°C S	20°C W
6S90	in mould	90°C S	90°C S	20°C W

W = water curing; S = steam curing

Maximum and minimum temperatures were reached with increment or decrement of 10 °C per hour. Densities of the samples were calculated by weighing the samples after their specified curing times and dividing their weight by the measured volume of the sample and then compressive strength test was made on the sample. The compressive strength of the specimens was determined using an ELE 2000 KN capacity testing machine.

## DISCUSSION

### Effect of W/B on Workability

By visual observation, the fresh RPC mixtures are generally homogenous, cohesive, thick, viscous and sticky due to

the high volume of fines and low water content. They showed a little or no bleeding and segregation. When a suitable w/b ratio is used with an enough amount of super plasticizer, the RPC appears to be plastic flowing. The Slump test and Vebe time test would not be applicable. Thus, the workability of the RPC mix was assessed by flow table test according to the ASTM C 1437 as shown in Figure 3.

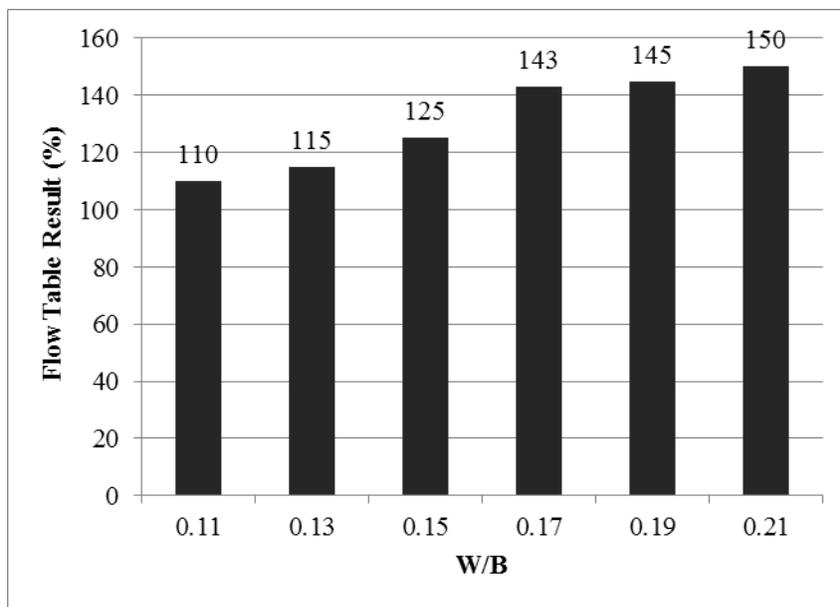


**Fig. 3.** Flow table test.

Water demand is the main parameter for assessing the quality of the granular mixture of RPC. The w/c ratio is usually replaced by the w/b ratio to account for the incorporation of silica fume and quartz powder to the strength development of RPC (Liu and Huang, 2008). Mixes with insufficient water were stiff and difficult to compact into moulds adequately so that this results in hardened concrete with entrapped air voids. Conversely, mixes with a higher w/b ratio were more susceptible to autogenous shrinkage. In this study, the RPC was prepared using different water/binder ratios including 0.11, 0.13, 0.15, 0.17, 0.19 and 0.21. Flow table test results are presented in Table 4 and Figure 4. As illustrated in Figure 4, a higher w/b or w/c ratio gives higher workability, but this increase is negligible in specimens with a w/b ratio more than 0.17.

**Table 4.** Sensitivity of workability to W/B ratio.

W/C	W/B	Workability (%)
0.15	0.11	110
0.17	0.13	115
0.2	0.15	125
0.22	0.17	143
0.25	0.19	145
0.27	0.21	150



**Fig. 4.** Effect of W/B ratios on flow table test results.

### Effect of Super Plasticizer Dosage on Workability

The production of a very low water-to-binder ratio of RPC is only possible through the use of high dosages of super plasticizer (SP). The very low w/b (cement + cement) ratio used in the RPC is only possible when the super plasticizer (SP) is

used. The Glenium-55p dosage of 2–4.5% liquid by the weight of cement was used for these applications. This is demonstrated in Table 5 and Figure 5, where the fairly consistent flow of a mix with a constant w/b ratio and varying super-plasticizer dosage is evident.

**Table 5.** Sensitivity of workability to SP/C ratio.

SP/C (%)	Workability (%)
2	113
2.5	115
3	125
3.5	123
4	124
4.5	120

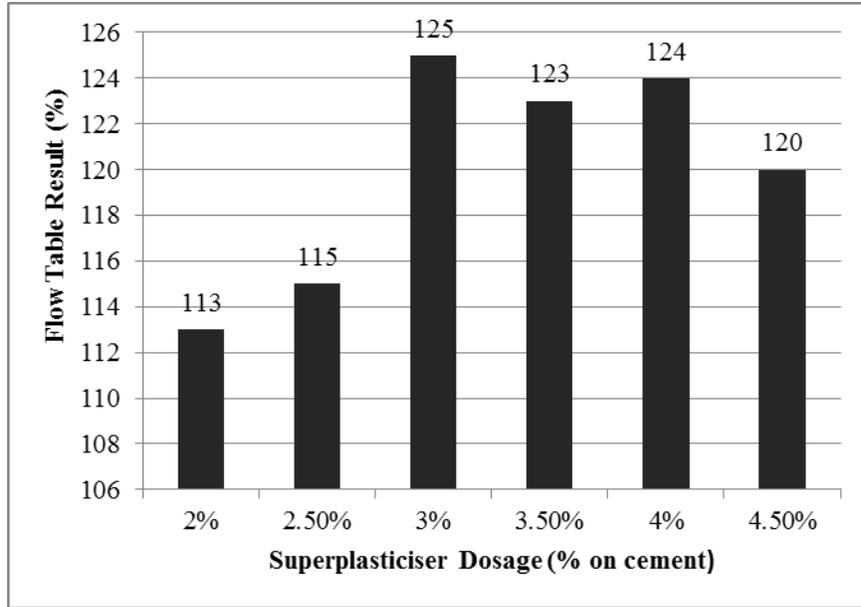


Fig. 5. Effect of SP/C ratios on flow table test results.

As seen in Figure 5, workability is highly affected by increasing the SP/C ratio up to 3%, but it decreases in specimens with the SP/C ratio more than 3%. In general, the particle size, shape and total water content appeared to be much more important controllers on workability.

**Effect of W/B on Compressive Strength**

The strength of concrete depends on the hydration reaction in which water plays a critical role, particularly the amount of water used. Therefore, an optimal w/b ratio is important in achieving high strength. Table 6 and Figure 6 show the effect of w/b ratio on the compressive strength at the age of 28 days.

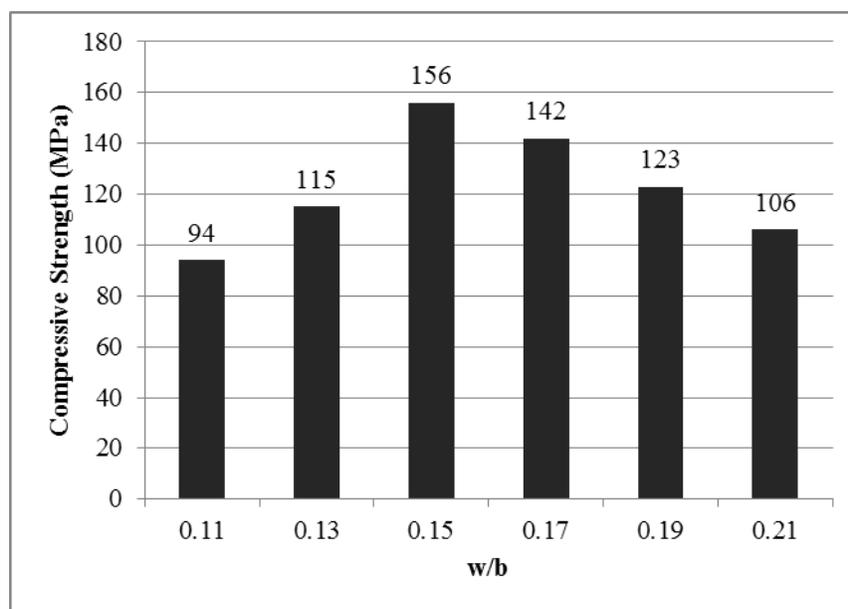
Figure 6 shows that there exists an optimal w/b ratio (i.e. 0.15) that gives the highest compressive strength of RPC (i.e. reaches 156 MPa in 28 days). Below the w/b ratio of 0.15, the lower compressive strength of RPC is experienced and above the w/c ratio of 0.15, the compressive strength decreases as w/b ratios increase. A 40% reduction in the compressive strength has been found when the w/b ratio is as low as

0.11. Such a reduction is mainly due to the lack of an adequate amount of mixing water in the RPC to ensure adequate compaction and proper hydration to occur.

Moreover, the pozzolanic reaction of silica fume is not complete due to the low water content. Beyond the optimal w/b ratio of 0.15, it was found that the compressive strength decreases as w/b ratios goes up. This is generally true as it has long been acknowledged that the lower the w/b or w/c ratio of a concrete mix, the higher the compressive strength of the concrete will be provided, Conversely the concrete with a higher b/w ratio gives lower compressive strength. This is because mixes with more fluid are more susceptible to entraining air bubbles due to the folding action of the mixing process. As a result, more voids are left in the matrix which increase the porosity and thus considerably reduce the compressive strength.

**Table 6.** Sensitivity of compressive strength to W/B ratio.

W/C	W/B	Compressive Strength (MPa)
0.15	0.11	94
0.17	0.13	115
0.2	0.15	156
0.22	0.17	142
0.25	0.19	123
0.27	0.21	106

**Fig. 6.** Effect of W/B ratios on compressive strength.

### Effect of Super Plasticizer Dosage on Compressive Strength

It has been demonstrated that the production of a cohesive and flowing RPC mix requires extremely large quantities of super plasticizer compared to high performance or self compacting concrete (Liu and Huang, 2008; Lee and Chisholm, 2005).

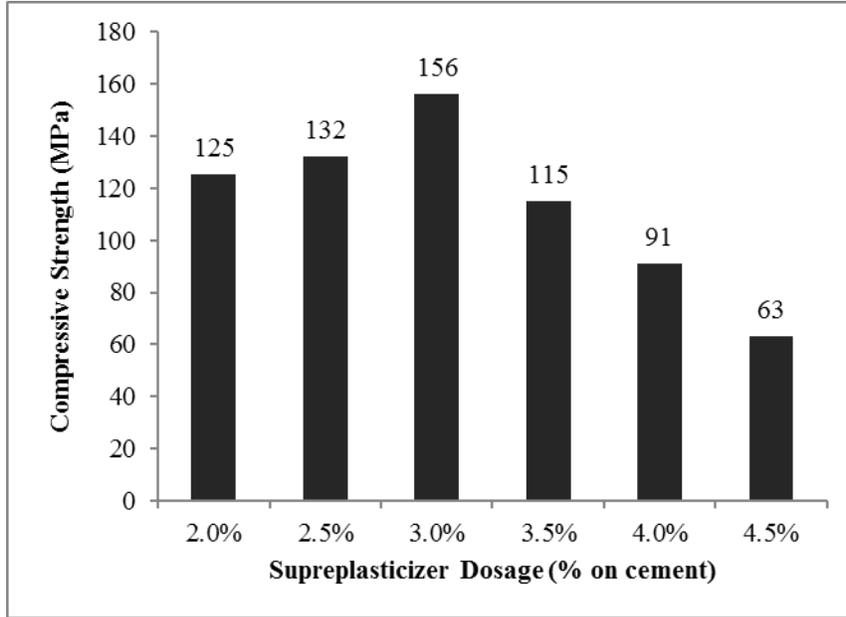
Table 7 and Figure 7 show the effect of SP dosage on compressive strength with a constant w/c ratio of 0.15. From Figure 7, the result clearly displays an optimal SP dosage that gives the highest compressive strength of RPC. The optimum SP dosage in this case is estimated to be 3% of the weight of cement content. Below and beyond the SP dosage of 3%, the results are associated with the lower compressive

strength of RPC. The application of the SP dosage of 2% has shown a 20% reduction in the compressive strength of RPC. The use of SP dosage of 4.5% has led to a 60% reduction in the compressive strength of RPC.

As the RPC contains many fine particles, the surface area increases dramatically compared to the normal concrete. The insufficient SP dosage reduces the workability due to the friction at the increased surface area. The cement, water and silica fume system tends to coagulate. In this state, the mixture becomes less mobile and the fine particles may easily get flocculated throughout the paste which leaving voids and consequently reducing the compressive strength.

**Table 7.** Sensitivity of compressive strength to SP/C ratio.

SP/C (%)	Compressive Strength (MPa)
2	125
2.5	132
3	156
3.5	115
4	91
4.5	63



**Fig. 7.** Effect of SP/C ratios on compressive strength.

**Effect of Temperature on Compressive Strength**

Table 8 and Figure 8 show the effect of temperature and heat-treatment duration on

the compressive strength of RPC. The compressive strength of specimens over a 28day period under different curing regimes is presented in Table 8 and Figure 8.

**Table 8.** Compressive strength of specimens at 28 days (MPa).

Sample Code	P = 0 MPa	P = 25 MPa	P = 50 MPa	P = 100 MPa	P = 150 MPa
W20	156	249.6	255.8	279.2	270.4
3W60	184	267	272	301.2	291
6W60	186.4	275	280.3	308.3	293
3W90	196	278	280.5	318.5	295.8
6W90	205	278.5	287.4	322	298.4
3S90	198	280	278.5	320	296.5
6S90	206	286	285.6	328.1	301.5

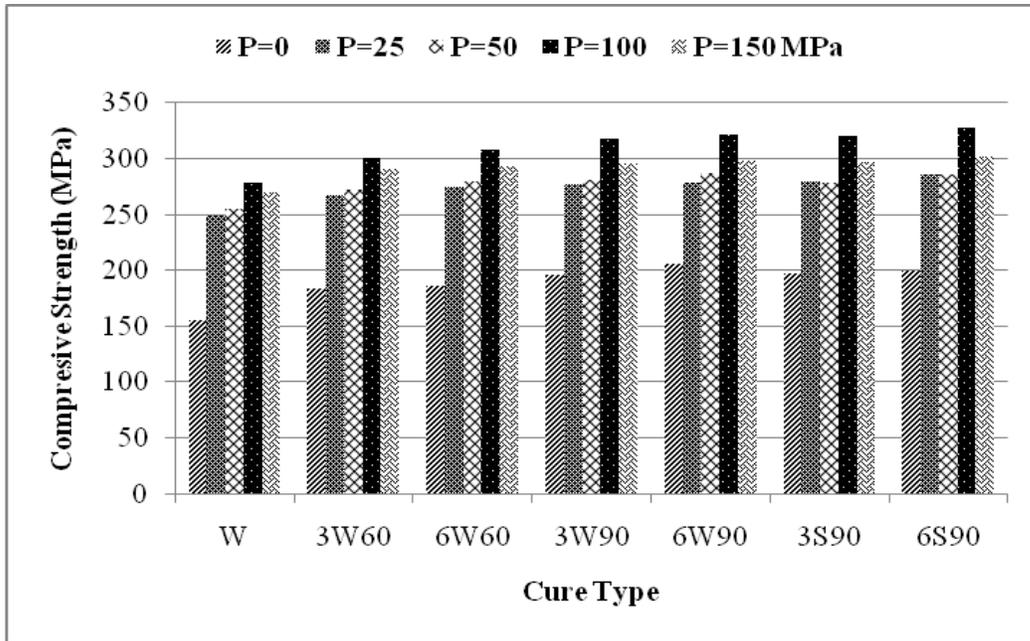


Fig. 8. Effect of cure type on compressive strength.

From Figure 8, it is found that the compressive strength of RPC increases rapidly when the temperature changes from 20 °C to 90 °C. When the RPC samples are heat-treated at temperature of about 90°C for 6 days (6W60 samples), the compressive strength of about 205 MPa are achieved in 28 days. This increase is about 32% when compared with that at room temperature of 20 °C (W20 samples). Heat-treatment temperature would lead to long C-S-H chains and this phenomenon could be attributed to the progression of cement hydration as well as the pozzolanic activity of both silica fume and crushed quartz (Zanni et al., 1996; Cheyrezy and Richard, 1995).

Figure 9 shows the effect of curing temperature on the compressive strength of RPC under pre-setting pressure (without pressure, 25 MPa and 100 MPa). The compressive strength of W20 samples for 25 MPa and 100 MPa pre-setting pressures are

increased by 14.5% and 17.5%, respectively. It is also observed that the increase in the compressive strength of the samples due to heat curing without any pre-setting pressure on specimens are more considerable than other mixes.

In heat and steam curing, the hydration process is activated and continued for a long time. The reactions of pozzolans are also accelerated by higher curing temperatures and it directly affects the compressive strength. The compressive strength of heat and steam cured specimens is higher than that of water cured specimens. In addition, C-H-S compositions change with temperature. In addition, pores of the microstructures of hot water and steam cure at 90 °C are less than the standard cures that can explain the compressive strength changes.

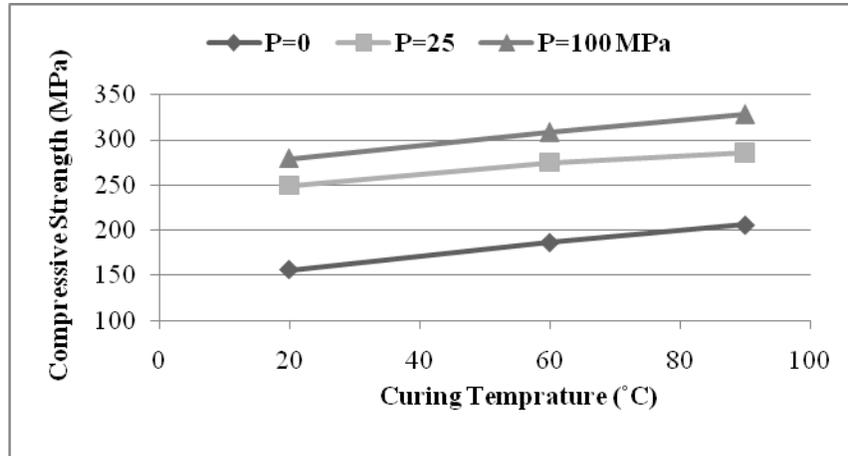


Fig. 9. Effect of curing temperature on compressive strength.

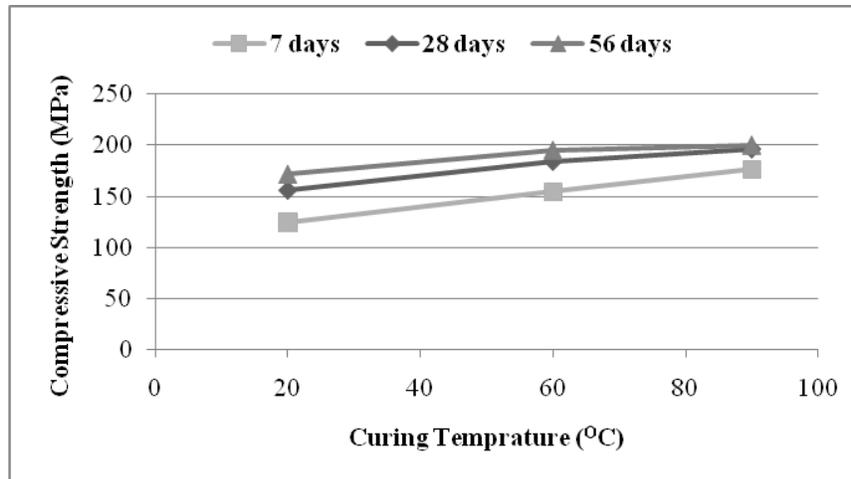


Fig. 10. Rate of changes in compressive strength at different ages.

The rate of changes in the compressive strength at different ages is presented in Figure 10. It can be found that the compressive strength of the samples slightly increased with increasing age. The increases observed in the compressive strength due to duration change from 7 days to 28 days are more than 28 days to 56 days. However, the compressive strength only increased slightly under hot steam curing after 7 days. It shows the accelerating effect of temperature on the hydration process of cement and Pozzolanic reaction of Silica fume in the early ages of specimens.

#### Effect of Pre-Setting Pressure on Density

The density of specimens under different pre-setting pressure is presented in Table 9 and Figure 11.

Figure 11 shows the mix that is pressurized obtained a higher density than others. The mix which is not pressurized obtained the lowest density. This confirms that the addition of pressure to the RPC decreases the amount of entrapped air and water.

The density of 6S90 sample at the 150 MPa pre-setting pressure with the highest ratio reached 2516 kg/m<sup>3</sup> while the density of this sample without pre-setting pressure was only 2401 kg/m<sup>3</sup>, implying a 4.7 % increment.

### Effect of Pre-Setting Pressure on Compressive Strength

Pre-setting pressure is applied for minimizing adverse effects of autogenous shrinkage as well as for removing water and air from the RPC. The compressive strength of specimens at 28 days of specimens under different pre-setting pressures is presented in Figure 12.

As illustrated in Figure 12, the compressive strength is highly affected by increasing pre-setting pressure up to 100 MPa. Table 5 shows the samples that are pressurized at 25 MPa their compressive strength reaches 286 MPa while the

samples without any pre-setting pressure have the compressive strength of 206 MPa. It can be observed in Figure 12 the samples which are pre-setting pressurized at 100 MPa have the maximum compressive strength of 322 MPa.

Pre-setting pressure up to 100 MPa is sufficient to force out air and free water from the samples. However, in cases with higher pre-setting pressure micro cracks are created due to the expansion of aggregates once the pressure is released. In addition, the water needed for hydration may be gone out, implying that materials may experience more deformations.

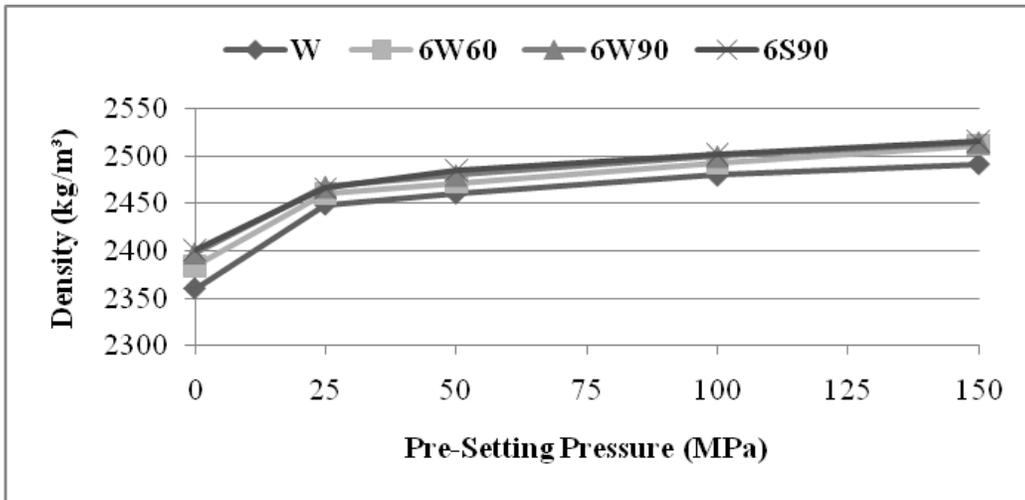


Fig. 11. Density in comparison to pre-setting pressure.

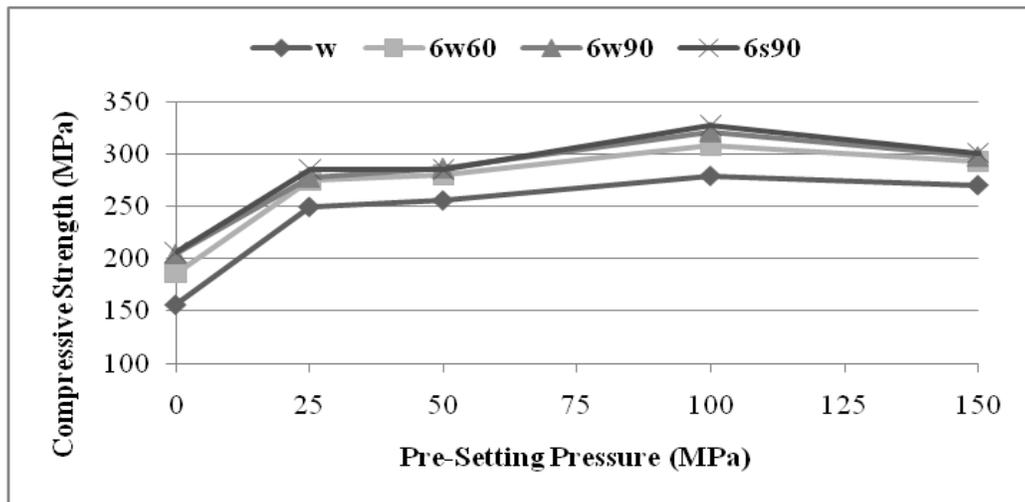


Fig. 12. Compressive strength in comparison to pre-setting pressure.

## CONCLUSIONS

The workability of the RPC mix was assessed using the flow table test according to the ASTM C 1437 for water/binder ratios of 0.11, 0.13, 0.15, 0.17, 0.19 and 0.21. A higher w/b or w/c ratio produced higher workability, but this increase was negligible in specimens with a w/b ratio more than 0.17.

The workability of RPC mixes containing 2, 2.5, 3, 3.5, 4 and 4.5% (liquid by weight of cement) was measured. The results showed that the workability is highly affected by increasing the sp/c ratio up to 3%. However the workability decreased in specimens with the sp/c ratio more than 3%. The effect of w/b ratio on the compressive strength over a 28-day period showed that the optimal w/b ratio is 0.15. The reduction of 40% in the compressive strength was experienced when the w/b ratio is as low as 0.11.

The effect of SP dosage on the compressive strength showed that the optimal SP dosage (for constant w/c ratio of 0.15) is 3% by the weight of cement content.

Curing temperature had a significant effect on the early strength development of RPC. An increase in the compressive strength was experienced at higher curing temperatures of 60 °C (compared to that of 20 °C) which was due to the rapid hydration of cement at higher temperature.

It was observed that when curing temperature increased from 20 °C to 90 °C, the compressive strength of RPC samples without pressure showed an increase of 32%.

By pressing the samples the density was increased. For W20 specimens, the density of pre-setting pressurized samples at 25 MPa reached 2440 kg/m<sup>3</sup> while the density of samples for samples without pre-setting pressure were 2360 kg/m<sup>3</sup>. The increase in

the density due to 50, 100 and 150 MPa pre-setting pressures was 4.3, 5.1 and 5.6%, respectively.

The compressive strength of W20 samples under 25 MPa pressure reached 249.6 MPa, while this value in samples with no pre-setting pressure was 156 MPa. It showed that pressurizing the samples could increase the compressive strength up to 60%. Other pre-setting pressure values were associated with lower rises in compressive strength. The maximum compressive strength of 328.1 MPa was observed under the 100 MPa pre-setting pressure.

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