

Research article

Autoclaved reactive powder concrete: the effects of steel microfibers and silica fume dosage on the mechanical properties

Ahsanollah Beglarigale, Çağlar Yalçınkaya*, Halit Yazıcı

Department of Civil Engineering, Dokuz Eylül University, Izmir, Turkey

Received 27 December 2013 Revised 20 February 2014 Accepted 24 June 2014

Abstract

Reactive Powder Concrete (RPC) is a type of ultra-high performance cement based composite with high strength and ductility. RPC was developed in the 1990s by Bouygues' laboratory in France. It is a special type of concrete which has properly optimized micro grain, binder phase and steel micro-fibers. RPC can achieve compressive strength values between 150–800 MPa, while traditional concrete which is used in current structures usually has 20–50 MPa compressive strength. In addition, its high performance under flexural loads is the most important advantage of RPC in the field of civil engineering. RPC has the potential to compete with steel from the point of aesthetics and structural capability. One of the curing methods to enhance the strength of this composite material is autoclaving. Autoclave curing needs additional SiO₂ source to fill micro pores and strengthen hydration products. In the scope of this study, the effect of volume fraction of steel micro-fibers and silica fume dosage as SiO₂ source on mechanical properties of RPC under autoclave curing was investigated. High performance cementitious composites were produced with 0%, 1%, and 2% volume fractions of steel micro-fibers. Nine mixtures with three different silica fume dosages were produced. Workability of fresh state and flexural-compressive strengths of hardened specimens were determined. In addition, fracture energies of the mixtures under

©2014 Usak University all rights reserved.

Keywords: Reactive powder concrete, silica fume, steel fiber, autoclave

1. Introduction

Reactive Powder Concrete (RPC) is an ultra-high-strength composite material with advanced mechanical properties and enhanced ductility. The material was developed in the 1990s by Bouygues' laboratory in France. RPC has very low water-to-binder ratio, high amount of binding and inert powders, and micro steel-fibers. The concept of reactive powder concrete was first developed by P. Richard and M. Cheyrezy. The researchers have developed two different classes of RPC, known as RPC 200 and RPC 800 [1].

In parallel with developments in the materials and chemical industry, the concrete technology has also been advanced. For most routine uses, the 20 MPa to 50 MPa concrete is often used in the modern concrete structures. RPC achieves compressive

©2014 Usak University all rights reserved.

^{*}Corresponding author: Tel: +90 – 232 – 3017042 E-mail: caglar.yalcinkaya@deu.edu.tr DOI: 10.12748/uujms.201416495

strength level in the range of 200–800 MPa (2000-8000 kg/cm²) and tensile strength in the range of 25–150 MPa. As a result of very dense microstructure, the RPC has greater durability properties compared to the conventional concrete.

The application of high strength concrete in practice is severely limited due to its brittle behavior. This negative behavior can be overcome by adding steel-micro fibers. The fiber reinforcement improves ductility of concrete under mechanical loads. In addition, crack propagation can be limited owing to the micro reinforcement effect of fibers. Very low water-to-binder ratio of the paste phase of RPC generates a powerful fiber-matrix interface. Therefore, RPC has better mechanical properties compared to high strength steel fiber reinforced concrete.

Yazıcı *et al.* investigated the mechanical properties of reactive powder concrete containing mineral admixtures under different curing regimes [2]. Their test results showed that the RPC containing high volume mineral admixtures had a satisfactory mechanical performance. Yazıcı *et al.* emphasized that although the cement and silica fume contents of these mixtures were importantly lower than the conventional RPC, the compressive strength exceeded 200 MPa after standard water curing. They also showed that the autoclave and steam curing seems very effective ways to increase the compressive strength of RPC. This finding of the study was attributed to the improvement of hydration process under heat curing regimes. In this case the compressive strength values were higher than 234 and 250 MPa after the steam and autoclaving curing, respectively.

In the scope of this study, it is aimed to produce a cementitious composite with a high workability and a compressive strength higher than 200 MPa. The effects of volume fraction of steel micro-fiber on the mechanical properties were studied. In addition, the influence of silica fume on the properties of RPC was investigated by using three different percentages of silica fumes. The compressive strengths and the flexural performance including fracture energies of RPC were evaluated.

2. Materials and Method

The RPC considered here is prepared by the following ingredients: an ordinary Portland cement (CEM-I 42.5-R produced by Denizli Cement Company); quartz powder (0–0.4 mm) and quartz sand (0.5–1.0, with a specific gravity of 2.65), silica fume (SF), a polycarboxylate-based superplasticizer (SP) in conformity with ASTM C 494-81 type F, and brass-coated steel micro-fibers (6 mm long with the diameter of 0.15 mm, the aspect ratio and tensile strength of the fibers is 37.5 and 2000 MPa, respectively). The physical and chemical properties of cement and silica fume are presented in Table 1. Silica fume was used as secondary cementitious material.

It is aimed to produce self-compacting RPC with a compressive strength higher than 200 MPa. Cement was replaced with 0%, 10%, and 20% (by weight) silica fume in the mixture which has 0.18 water-to-binder ratio. In addition, the effect of steel micro-fiber reinforcement was evaluated by incorporating 0%, 1%, and 2% volume fractions of fiber. A Hobart mixer was used to prepare fresh mixtures. All dry particles and fibers were mixed for 1 min. Then, water-one quarter SP solution was added to dry mixture. The RPC was mixed for 5 min at low rotation speed and 10 min at high rotation speed with remaining SP. After mixing, slump flow tests were immediately applied with using a mini slump cone which has 10 cm diameter. After the workability test, fresh RPCs were cast into $40 \times 40 \times 160$ mm³ prismatic moulds without any compaction. After that, a short time

vibration (10 sec) was applied to reduce entrapped air content of the mixtures. All moulds were kept in a cabinet with $20\pm1^{\circ}$ C and $97\pm2\%$ relative humidity during the first 24 hrs. Then, all prismatic specimens were demoulded and kept in autoclave. The specimens were autoclaved under 2 MPa pressure for 8 hrs (210°C). Temperature and pressure reached to their maximum values in 2.5 hrs. Mix proportions was presented in Table 2. In the mixture names: the dosage of silica fume was abbreviated as SF and volume fraction of steel micro-fiber was abbreviated as F. For example, SF10-F2 mixture has 10% silica fume replacement and 2% steel micro-fiber volume.

Table 1

Physical and chemical properties of cement and silica fume

Chemical composition (%)	Cement	SF	Physical Properties		
CaO	64.25	0.51	Cement		
SiO ₂	18.52	92.25	Specific surface (cm ² /g)	3860	
Al_2O_3	4.70	0.88	Specific gravity	3.15	
Fe ₂ O ₃	3.24	1.98	SF		
MgO	0.93	0.96	Specific surface (cm ² /g)*	210800	
Na ₂ O	0.35	0.45	Specific gravity	2.2	
K20	0.80	0.12			
SO_3	3.03	0.33			
Loss on ignition	3.17	3.0			
Free CaO	1.9	-			

*nitrogen adsorption method

Table 2Mix proportions

	Mixtures										
Materials	SF0-	SF0-	SF0-	SF10-	SF10-	SF10-	SF20-	SF20-	SF20-		
(kg/m ³)	FO	Fl	FΖ	FO	F1	FΖ	FO	F1	FΖ		
Water	174	174	174	174	174	174	174	174	174		
Cement	981	981	981	883	883	883	785	785	785		
SF	-	-	-	98	98	98	196	196	196		
0.5-1.0mm*	726	710	694	704	689	673	684	668	652		
0-0.4 mm*	483	473	462	469	459	448	455	445	434		
Fiber	-	71.7	143.4	-	71.7	143.4	-	71.7	143.4		
SP	35	35	35	35	35	35	35	35	35		
Design Parameters											
water/cement	0.18	0.18	0.18	0.20	0.20	0.20	0.22	0.22	0.22		
water/binder	0.18	0.18	0.18	0.18	0.18	0.18	0.18	0.18	0.18		
water/cement [†]	0.20	0.20	0.20	0.22	0.22	0.22	0.24	0.24	0.24		
water/binder [†]	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20		

*quartz aggregate

[†]calculated with total water (water + water from SP)

All the flexural strengths and load-deflection graphs were determined with carrying out three-point bending tests by an electro mechanic closed-loop testing system (loading rate: 0.2 mm/min). After autoclaving, the specimens were loaded from their mid-span, and the clear distance between simple supports was 130 mm (Fig. 1). The beam specimens have the same notch depth for all series, equal to one sixth of beam height (\approx 6.5 mm). The fracture energy was determined using with the load-deflection curves of the three point bending test results. The fracture energy was calculated by dividing the area under the load-deflection curve to effective cross-section area for each specimen. In this study, small prismatic specimens were used in flexural loading. So, the weight of the specimens (\approx 600–700 g) was neglected in the calculation of fracture energy.

The compressive strength tests were applied on two pieces ($40 \times 40 \text{ mm}^2$ loading area) that were left from the flexural tests with a 2400 N/s loading rate.



Fig. 1 Schematic presentation of three-point bending test

3. Results and Discussions

An increase in silica fume (SF) dosage decreased slump-flow diameters. While the mixture without SF has a 250 mm slump-flow value, each 10% SF addition caused 15 mm reduction in flow values. This may be attributed to high specific surface of SF. In spite of self-compacting ability of the mixtures, these sticky mixtures have a tendency for air entrapment. Thus, a short time vibration (10 sec) was applied to decrease entrapped air content of the mixtures.

Load versus mid-span deflection curves of the mixtures with different fiber volumes and SF replacement ratios are presented in Fig. 2. As can be seen from Fig. 2, the unreinforced RPC demonstrated relatively brittle behavior. The stress decreased rapidly with the increase of mid-span deflection after peak load. Steel micro-fibers changed that behavior completely. The peak loads, ductility, and toughness values were increased with an increase in fiber volume and SF dosage.

Steel micro-fibers in RPC bridge micro-cracks and therefore control their growth and delay its propagation. Brass-coated steel micro-fibers can achieve a powerful adherence to the cement paste with a low water-to-binder ratio. Moreover, the tensile strength of the brass-coated steel micro-fibers is higher than the strength of the steel macro fibers. The number of fibers crossing a cracked section of concrete increases as the size of the used steel fiber decreases. The bridging effect of fibers in a cracked specimen subjected to the flexural loading can be seen in Fig. 3. Depending on visual observations, an increase in SF dosage resulted in an increase in the number of ruptured fibers. In other words, SF enhanced the fiber-matrix bond characteristics. Transition zone in a cementitious composite is quite porous and also filled with CH in direct contact with the fiber surface. In this study, the density of this weak zone was increased by silica fume which has a great pozzolanic activity and a high specific surface area.



Fig. 2 Load vs. mid-span deflection curves of RPC specimens



Fig. 3 Crack bridging of steel micro-fibers

The flexural strengths and fracture energies of RPC mixtures are presented in Fig. 4. The flexural strength of plain mixture without fibers was 5 MPa, while the flexural strengths of 1% fiber reinforced mixture reached to 12 MPa for SF0, 12 MPa for SF10, and 18 MPa for SF20. The flexural strengths were 17 MPa for SF0, 21 MPa for SF10, and 30 MPa for SF20 with using 2% fiber volume. Positive effect of fiber reinforcement on the fracture energies was more pronounced compared to the flexural strengths. The fracture energies of 1% and 2% steel fiber reinforced RPC with 20% SF were about 77 times and 121 times higher than the plain RPC without fibers, respectively. These significant increases in fracture energy decreased dramatically with a decrease in SF dosage. The brittle cement paste was highly sensitive to the micro cracks. In addition, lack of coarse aggregate in RPC may reduce the effectiveness of the aggregate phase on crack bridging. Cracks in RPC may run through the aggregates since the mechanical properties of the powerful matrix of RPC and aggregates were quite similar. It is well known that notches have a negative influence on flexural strengths of cementitious materials. This behavior is called as notch sensitivity in the literature [3]. Thus, the mixtures without fiber reinforcement exhibited a flexural performance less than expected. It can be said that the fiber reinforcement decreases the notch sensitivity of RPC.



Fig. 4 Flexural strengths and fracture energy values

The compressive strengths of the mixtures after autoclaving are presented in Fig. 5. Compressive strengths were increased importantly with an increase in fiber volume and SF dosage. Target compressive strength of 200 MPa was achieved by inclusion of 1% fiber in SF20 mixture. This target strength value was exceeded by fiber volume of 2%. The mixtures with a SF dosage under 20% did not achieve strength value of 200 MPa. Each 10% SF addition caused a compressive strength gain of 15%–20% for the plain RPC without fibers. When SF replacement level increased from 10% to 20%, the strength gain reached 25% for fiber volume of 1%. In the case of 2% fiber volume, the compressive strength gain due to SF replacement is similar to its non-fiber condition. The relative compressive strengths for each SF dosage are presented in Fig. 6. It can be said that the positive effect of fiber inclusions on compressive strength increased slightly with a decrease in SF dosage. In other words, weakness of autoclaved cementitious matrix due to lack of SF may be compensated by fiber inclusion. Rapid formation of hydration products under autoclave curing may cause weak and porous matrix due to absence of SF as a SiO₂ source. In addition, porous CH structure surrounding the fiber can be modified with using SF.



Fig. 5 Influence of steel micro-fiber volume and SF dosage on compressive strength



Fig. 6 Relative compressive strengths for SF dosage

The hydration products formed under autoclave curing differ essentially from those formed at temperatures below 100°C [4]. In the hydration of C_3S and C_2S , crystalline α -dicalcium silicate hydrate (α -C₂SH) forms instead of a usual amorphous C–S–H phase in the absence of a SiO_2 source. The mechanical properties and bonding characteristics of this phase is rather unfavorable [5]. Secondary electron imaging was applied on fractured samples to study the morphology of products and to understand the reason of the strength loss in the absence of SF under autoclaving (Fig. 7). It was found that α -C₂SH may form in the absence of SF in RPC mixtures (SF0). Energy dispersive spectroscopy (EDS) analysis showed that these products have a high Ca/Si ratio (1.5 to 2.2). In the presence of finely ground SiO_2 sources under autoclaving, a pozzolanic reaction takes place, yielding crystalline 1.1 nm tobermorite ($C_5S_6H_5$), as the main product of reaction. The low Ca/Si ratio (0.8 to 1) of these products improves the mechanical properties [5-6]. Foliaceous and fibrous structure of tobermorite with a Ca/Si ratio of 1 in SF10 mixture can be seen in Fig. 7. When SF dosage was increased to 20%, Ca/Si ratio decreased significantly (0.92). These findings showed evidence of transformations of α -C₂SH to tobermorite structure.



Fig. 7 SEM photographs of hydration products in Autoclaved RPC with different SF dosages

4. Conclusions

A cementitious composite with a compressive strength of 213 MPa and a flexural strength of 30 MPa can be produced by inclusion of 2% steel micro-fiber and 20% SF. Two important influences of SF were discussed in this study: strength enhancement in matrix without fibers (modifications on C-S-H structure) and the improving interface properties. Test results indicated that the RPC has a good mechanical performance after autoclaving in a short curing time. However, the hydration products should be designed carefully on micro-scale by using optimum amount of SiO_2 source to achieve desired performance. In addition, SF usage is still one of the most effective methods of improving fiber-matrix interface characteristics. It is recommended that further research be undertaken in the following areas: strengthening of the fiber-matrix interface for practical applications of RPC in the construction industry, and producing a special type of cement for RPC.

Acknowledgement

This study was supported by the Scientific and Technological Research Council of Turkey (TUBITAK, Project code: 110M691). The authors would like to thank BASF (Mr. Cevdet KUMAŞ), Bekaert, Denizli Cement, Pomza Export (Mr. Ümit ÜRÜN) firms for supplying materials.

References

- 1. Richard P and Cheyrezy M. Composition of reactive powder concretes. Cement and Concrete Research, 1995; 25: 1501 1511.
- 2. Yazıcı H, Yardımcı MY, Aydın S and Karabulut AŞ. Mechanical properties of reactive powder concrete containing mineral admixtures under different curing regimes. Construction and Building Materials, 2009; 23: 1223 1231.
- Wong YL, Lam L, Poon CS and Zhou FP. Properties of fly ash modified cement mortar-aggregate interfaces. Cement and Concrete Research, 1999; 29: 1905 – 1913.
- 4. Neville AM. Properties of concrete. John Wiley & Sons, New York, 1973.
- 5. Odler I. Hydration setting and hardening of Portland cement. Lea's chemistry of cement and concrete, 4th edition, Elsevier Science & Technology, UK, 2004.
- 6. Yazıcı H, Deniz E and Baradan B. The effect of autoclave pressure, temperature and duration time on mechanical properties of reactive powder concrete. Construction and Building Materials, 2013; 42: 53 63.