

# Effect of Preparation Technologies on Properties of Reactive Powder Concrete with Nano-zirconia

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**Abstract :** Reactive powder concrete filled with 3% content of nano-zirconia (NZ) are fabricated to investigate the effect of preparation technologies on the mechanical strength. The preparation technologies involve internal (NZ is added in RPC and replaced cement)/external mixing (NZ is added in RPC but not replaced cement), ultrasonic time, high mixing speed, saturated lime water/high temperature curing media (curing in water at 90°C). The influencing mechanisms of processing method are revealed through X-ray powder diffraction (XRD) and thermogravimetry (TG) analysis, scanning electron microscope observation. Experiment results showed that high mixing speed and high temperature curing media can improve the mechanical strength obviously. The compressive strength of NZ filled reactive powder concrete with high mixing speed increase 49.9%. The compressive strength, flexural strength and splitting strength of reactive powder concrete with NZ under high temperature curing media increase 35%, 15% and 17% respectively compared with control concrete.

**Keywords:** Concrete; Nano-zirconia; Preparation technologies; Mechanical strength;

## 1. Introduction

Beacuse of the resistance to water, plasticity and availability, cement and concrete have been the most widely used construction materials. However, cement-based material is a quasi-brittle material according to the macroscopic mechanical behavior. There exist many defects interiorly and on its surface. Its tensile, shear and impact resistance are weak. Reactive powder concrete (RPC) was known as a type of ultra-high-performance concrete with excellent granular compactness[1-3]. The compressive strength and elastic modulus of RPC are between 200 and 800 MPa, 50 and 75 GPa, respectively, while the flexural strength can reach 140 MPa[4-6].

Nanotechnology is an emerging field related to the understanding and control of materials at nanoscale. Recent developments of nanotechnology show significant promise in addressing many of the challenges in various areas. To date, applications and advances of nanotechnology have injected new vitality into cement and concrete materials. Nano-ZrO<sub>2</sub> (NZ) considers as a reinforcement filler with high strength, high toughness and good dispersion, which has attracted considerable attention from many researches.

Soleymani[7] investigated the flexural strength of cement paste at 28 d, with NZ filled. Compared to the increase of 20.2% in 1.5% NZ content, the strength value has increased 65.9% in saturated limewater curing, with 2.0% NZ. With the increase of nanoparticles, harmless and few-harm pores in concrete enhance, while harmful and multi-harm pores reduce. However, both of the extent decrease and the improvement on the pore structure of concretes is weakening. Shekari[8] et al found that the compressive strength of NZ filled ordinary Portland cement paste with 15% metakaolin can increase 20.2% at curing age of 28 d in water when NZ content is 1.5%. Nazari[9-11] et al researched the mechanics properties and workability of NZ filled ordinary Portland cement paste. Results show that the flexural, compressive and splitting strength at curing age of 28 d in water increase 31.8%, 18.5% and 83.3%, respectively when NZ content is 1.0%. But the workability decrease when the NZ was added. Han<sup>[12]</sup> et al reported that NZ can obviously improve the mechanical properties of RPC. The compressive, flexural and splitting strength of RPC increase 16.3%, 36.6% and 34%, respectively, compared with pure RPC.

Through the literature survey of the authors there are few published studies on the influence of different preparation technologies on composites. However, previous studies mainly focus on the influence of NZ on ordinary Portland cement paste. Few research has been done on the preparation technologies effect on mechanical strength of RPC with NZ. In this study, RPC with 3.0% NZ was fabricated by different preparation technologies and the flexural, compressive and splitting strengths are investigated at curing age of 28 d. Scanning electron microscope (SEM), TG with theoretical calculation and XRD were used to analyze the reinforcing mechanisms of NZ in RPC.

## 2. Experiment

## 2.1 Materials

The materials used in this paper include water, cement, silica fume, fly ash, quartz sand, superplasticizer and NZ. The NZ is monoclinic crystal with average particle size of 20 nm, which is provided by Nanjing Haitai Nano-materials Co. Ltd. in China and used as filler. Its SEM image was shown in Fig. 1. The P.O 42.5R cement provided by Dalian Onoda Cement Co. Ltd. in China was used as binder. Silica fume is provided by Shanghai Tian Kai Silicaon Fume Co. Ltd. China. Fly ash is product of Dalian Daokete Building Materials Co. Ltd. China. Silica fume and fly ash were used as mineral admixture. Quartz sand with the size range from 0.12 to 0.83 mm was used as aggregate. The type of superplasticizer is RHEOPLUS 411 (BASF), which was used to adjust the workability of the concrete mixtures and assist NZ dispersion.

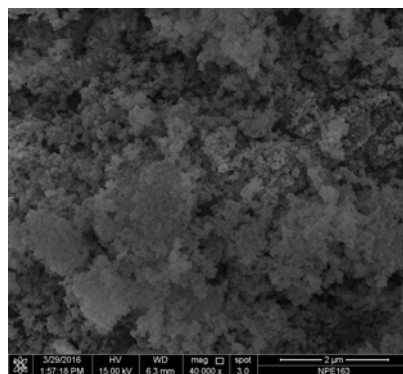


Fig. 1 SEM image of NZ

## 2.2 Preparation

The water to binder ratio was fixed at 0.24 for all the mixture. The content levels of NZ is 3.0% by weight of cement (our previous experiments prove the optimal content of NZ contributed to the mechanical property of RPC is 3% [12]). The detail mix proportions are shown in Table 1. In order to mix NZ uniformly and keep workability of the mixture, superplasticizer was first put into the water containing NZ. The progress of fabricating NZ filled RPC is presented in Fig. 2. The details of specimens of codes 0 W, B and R are as following: (1) Water, superplasticizer, NZ, cement, silica fume, fly ash and sand were weighted as mix proportions. (2) Water, NZ and superplasticizer were mixed in cement paste mixer (MSD-E1000 Type, Shanghai Muxuan Experimental Co., Ltd., China) at low speed for 10 s. (3) The silica fume were put into the suspension and mixed at low speed for 60 s. (4) The cement and fly ash were put into the mixing pot and mixed at low speed firstly for 120 s and then at high speed for 120 s. (5) The sand was put into the suspension and mixed at low speed firstly for 60 s and then at high speed for 240 s. (6) The mixture was poured into the oiled mould (40 mm×40 mm×40 mm and 40 mm×40 mm×160 mm) and the mould was put on the electric vibrator to eliminate bubbles. (7) The specimens were cured at temperature of 20.0°C in 95% relative humidity for 24 h before demold. Then specimens of codes 0 and W were cured in water at 20±1°C for 27 d. Specimens of code B were cured in saturated lime water at 20±1°C for 27 d. Specimens of code R were cured for 48 h in water at 90°C.

Table 1 Mix proportions of NZ filled RPC

Sample codes	Cement	Fly ash	Silica fume	Sand	Water	Superplasticizer	NZ
0	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
W	0.8	0.2	0.25	1.1	0.3	1.86%	3.0%
C2	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
C5	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
C10	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
J	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
B	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%
R	0.776	0.2	0.25	1.1	0.3	1.86%	3.0%

The preparation process of series C is same as that of code W except for steps (2), in which the mixture of water, NZ and superplasticizer were sonicated in SCIENTZ-1200E (provided by Ningbo Xin Zhi Biotechnology Co., Ltd. in China) for 2min, 5min and 10min, respectively. The preparation process of code J is as following: (1) Water, superplasticizer, NZ, cement, silica fume, fly ash and sand were weighted as mix proportions. (2) Water, NZ and superplasticizer were mixed at speed of 3000 r/min for 120 s. (3) The silica fume were put into the suspension and mixed for 120 s. (4) The cement and fly ash were put into the mixing pot and mixed for 120 s.

Steps (5), (6) and (7) are same as that of code W. The samples for TG and XRD test were cement paste, which mix proportions and preparation technologies are same as above except adding sand. The reason why do not add sand is that the weight of dry sand does not decrease as temperatures increase and the amount of sand in samples is random. The other hands, the very strong XRD characteristic peaks of sand will deteriorate the conspicuous of XRD peaks of cement hydration products. After curing for 48 h or 28 d, the cement pastes were crushed, milled and sieved at 80  $\mu\text{m}$ . Then the samples were dried at 50°C for 24 h to expel the absorbed water. The samples for SEM are fragment of the composites after testing flexural and compressive strength.

### 2.3 Measurement

The tested properties of the specimens include flexural strength, compressive strength, and splitting strength. The flexural strength was measured by a mortar folding meter DKZ-5000 at age of 48 h and 28 d. All the specimens were loaded to failure at constant loading rate of 0.5 mm/min. The average value of flexural strengths of 3 specimens in each group was recorded as the final flexural strength if the maximum or the minimum value is 10% less than the average value. The compressive strength was also measured according to (Method of testing cements-determination of strength-ISO) GB/T17671-1999 of China. The specimens of 40 mm  $\times$  40 mm  $\times$  160 mm were broken in two near the middle after flexural test. The two pieces were measured by using compression testing machine YAW-2000D provided by China Jinan Era Assay Testing Machine Co. Ltd. and using test fixture with contact area of 40 mm  $\times$  40 mm. All the specimens were loaded to failure at constant loading rate of 1.2 mm/min. The average value of compressive strengths of 6 types of specimens in each group was recorded as the final compressive strength if the maximum or the minimum value is 10% less than the average value. Splitting strength was also measured by the universal material testing machine WDW-200E. The specimens were put on the center of platen and the steel bearing plate and the wooden cushion were put between the upper platen and the lower platen (as shown in Fig.3). Field Emission Scanning Electron Microscope (Nova Nano SEM 450, American FEI Ltd.) was used to observe the microstructures of the composites. Environmental Scanning Electron Microscope (Quanta450, American FEI Ltd.) was used to observe the distribution of NZ in RPC. TG analysis was performed by using a METTLER TOLEDO STARE system to get the amount of CH and other hydration products. The condition of TG analysis was under nitrogen atmosphere at a heating rate of 10 °C/min up to 1000°C. XRD (Bruker D8 Advance, Bruker German) was applied for testing the change in tendency of calcium hydroxide (CH) crystal inside the composites caused by NZ.

## 3. Result and discussion

### 3.1 Flexural strength

Fig. 2 shows relative increase rate of flexural strength of the composites at curing age of 48 h and 28 d. It can be seen that the flexural strength of specimens made by different preparation technologies have grown. The relative increase rate of flexural strength is 14.4% on the code W. When the water, NZ and superplasticizer were treated by ultrasonic, the relative increase rate increase firstly and then decrease with the ultrasonic time increase (series C). The maximum relative increase rate is 28.2% at the ultrasonic time of 5 min. As a contrast, the value can reach 49.9% on the code J. Ref[7] make a comparison on the flexural strength of NZ filled Ordinary Portland Cement (OPC) with different curing condition (water/saturated lime water). When the content of NZ is 2.0%, the flexural strength of NZ filled Ordinary Portland Cement (OPC) cured in saturated limewater increase by 44.7%. However, the increase rate of flexural strength of 3% content NZ filled RPC cured in saturated limewater is only 9.7%. Compared with code 0 cured in water, composites cured in water at 90°C have a 35.0% increase on flexural strength.

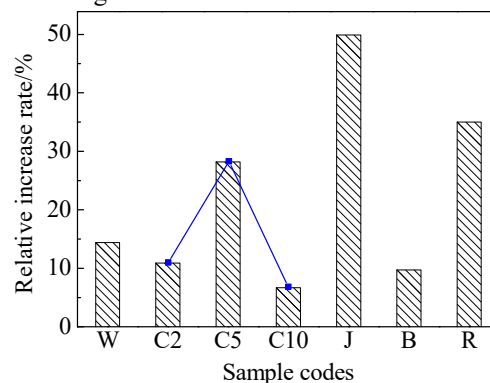


Fig. 2 Relative increase rate of flexural strength of the composites at curing age of 48 h and 28 d

Table 2 shows the relative standard deviation of flexural strengths of RPC with NZ at curing age of 48 h and 28 d. It can be seen flexural strength of the composites is discrete, which ranges from 0.01 to 0.08. This indicates that the dispersion of NZ in RPC is relatively homogeneous, and flexural strength of the composites in each group are relatively stable.

Table 2 The relative standard deviation of flexural strengths of 3% NZ filled RPC with different preparation technologies at curing age of 48 h and 28 d /%

Age	Sample codes						
	W	C2	C5	C10	J	B	R
48 h and 28 d	0.08	0.05	0.01	0.05	0.06	0.04	0.05

### 3.2 Compressive strength

Fig. 3 shows the relative increase rate of compressive strength of the composites, at curing age of 48 h and 28 d. As shown in Fig. 3, the compressive strength of code W and series C decreased obviously, the minimum relative decrease rate is 0.5% presented in code C5. What's more, the low relative increase rate of code J (1.4%) was also not high enough to generate a statistically significant effect on the compressive strength. From a curing condition standpoint, curing in saturated lime water at  $20\pm 1^\circ\text{C}$  for 28 d has no effect of reinforcement on the compressive strength of composites. But the significant increase of compressive strength appeared on the composites cured in water at  $90^\circ\text{C}$  for 48 h., and the relative increase rate is 15%.

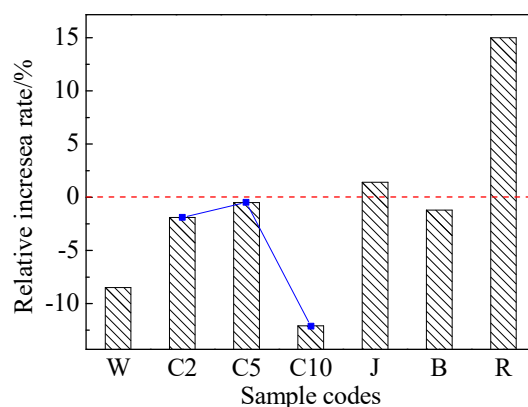


Fig. 3 Relative increase rate of compressive strength of the composites at curing age of 48 h and 28 d

Table 3 shows the relative standard deviation of compressive strengths of RPC with 3% NZ at curing age of 48 h and 28 d. It can be seen that the discreteness of compressive strength of NZ filled RPC at curing age of 48 h and 28 d are in the range from 0.04 to 0.06. It indicates that the dispersion of NZ in RPC is relatively homogeneous and compressive strength of the composites in each group is relatively stable.

Table 3 The relative standard deviation of compressive strengths of 3% NZ filled RPC with different preparation technologies at curing age of 48 h and 28 d /%

Age	Sample codes						
	W	C2	C5	C10	J	B	R
48 h and 28 d	0.06	0.04	0.04	0.05	0.04	0.04	0.04

### 3.3 Splitting strength

Fig. 4 shows relative increase rate of splitting strength of the composites at curing age of 48 h and 28 d. It can be seen that the splitting strength of code W also decreased. The splitting strength of series C decreased first and then increase with the ultrasonic time increase. Unlike flexural and compressive strength, the relative decrease rate of splitting strength of code C5 is the maximum, and the splitting strength of code C10 has grown slightly. However, the drop in splitting strength of code J is quite big, whose relative decrease rate is 35.6%. Compared with curing in water at  $20\pm 1^\circ\text{C}$ , both conditions of saturated lime water curing at  $20\pm 1^\circ\text{C}$  and water curing at  $90^\circ\text{C}$  make contribution to the splitting strength. The relative increase rate is 4.2% and 17%, respectively. Table 4 lists the relative standard deviation of splitting strengths of 3% NZ filled RPC with different preparation technologies at curing age of 48 h and 28 d. It can be seen that the discreteness of splitting strength of the composites at curing age of 48 h and 28 d are from 0.02 to 0.18. It means that the dispersion of NZ in RPC is relatively homogeneous and splitting strength of the composites in each group is relatively stable.

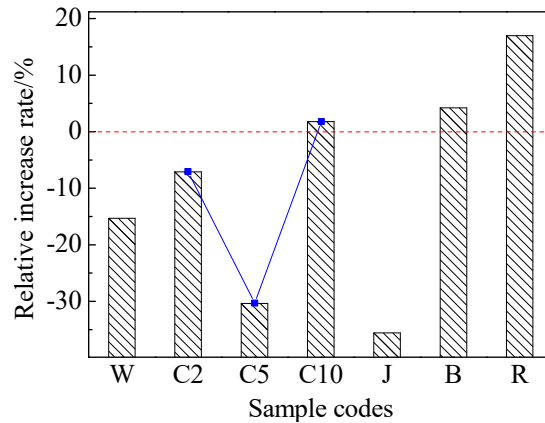


Fig. 4 Relative increase rate of splitting strength of the composites at curing age of 48 h and 28 d

Table 4 The relative standard deviation of splitting strengths of 3% NZ filled RPC with different preparation technologies at curing age of 48 h and 28 d/%

Age	Sample codes						
	W	C2	C5	C10	J	B	R
48 h and 28 d	0.04	0.08	0.11	0.08	0.02	0.18	0.02

### 3.5 Reinforcing mechanisms

TG and DTG diagrams of the composites at curing age of 48 h and 28 d are shown in Figs. 5, 6 and 7, respectively. The mass loss between 50-300°C is due to the dehydration of C-S-H gel, Aft and physically-bonded water[13]. The mass loss step in 400-550°C is resulting from the decomposition of calcium hydroxide (CH) (as shown in Table 6). In addition, cement hydration degree can be gotten based on the TG results and the calculation method according Ref. [14]. Furthermore, the water required is 0.23 g for full hydration of 1 g cement[15]. The ratio of cement to binder is 0.64 in this paper. Based on the above-mentioned, the cement hydration degree in this paper can be calculated and is shown in Table. 5. The XRD patterns of composites at curing age of 48 h and 28 d are shown in Figs. 8, 9, and 10. Crystal face peak intensity of CH can be obtained and then the CH orientation can be calculated according to the calculation given in Ref.[16]. The calculation results are listed in Table 7.

It can be seen from Figs. 5, 8 and Tables 5, 6, 7 that the cement hydration degree increase and the mass of CH and the CH orientation both decrease when NZ was added in RPC but NZ not replaced cement. The above factors could make contribution to the flexural strength but no contribution to the compressive and splitting strength. As shown in Fig. 11, though the mass of CH and the CH orientation both decrease, cracks and pores can be seen obviously in the matrix, which may lead the compressive and splitting strengths decrease.

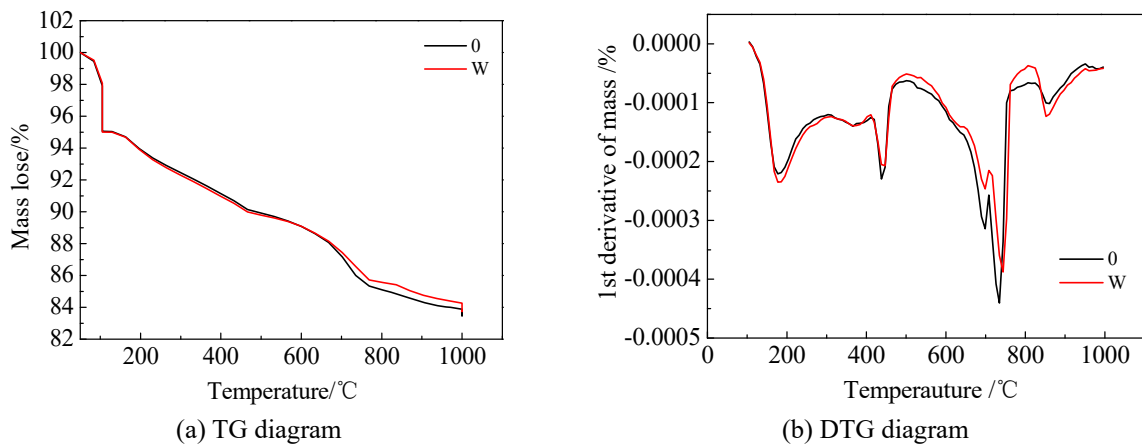
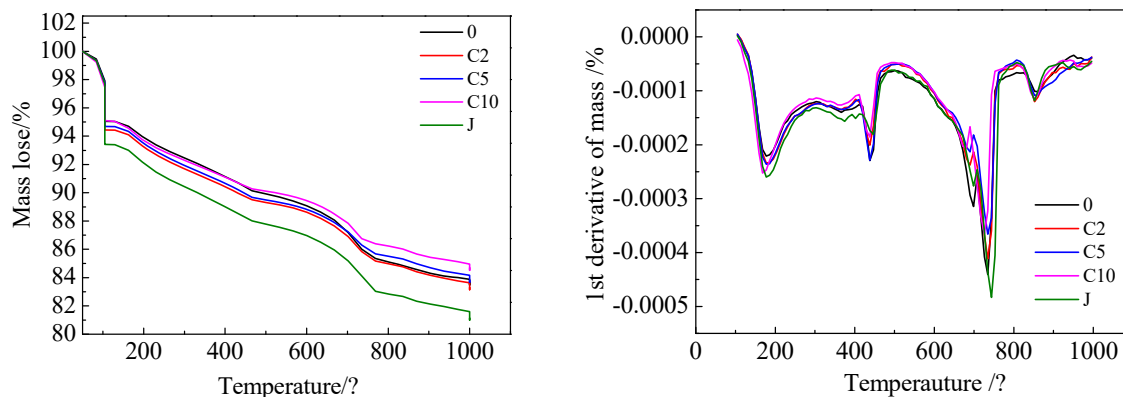


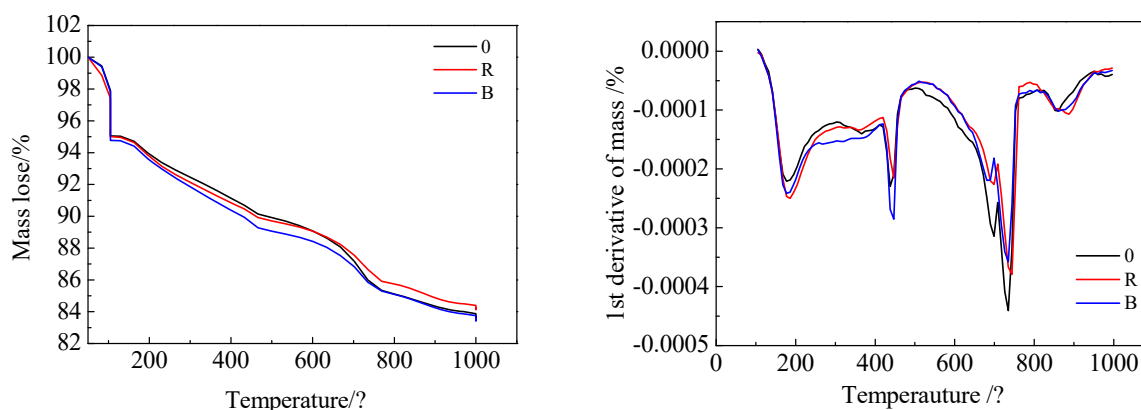
Fig.5 TG diagram (a) and DTG diagram (b) for 0 and W at 28 d



(a) TG diagram

(b) DTG diagram

Fig.6 TG diagram (a) and DTG diagram (b) for 0, series C and J at 28 d



(a) TG diagram

(b) DTG diagram

Fig. 7 TG diagram (a) and DTG diagram (b) for 0 R and B at 48 h and 28 d

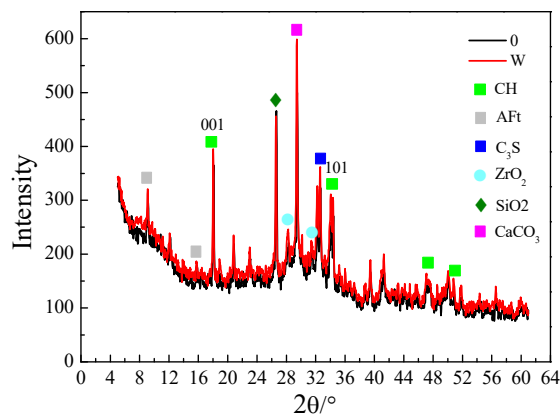


Fig. 8 XRD patterns of 0 and W at curing age of 28 d

As shown in Fig. 6 and Table 5, the cement hydration degree increase when ultrasonic time is less than 5 min. But when ultrasonic time is greater than or equal to 5 min, the cement hydration degree decrease. It was possible that the longer the ultrasonic time is, the more uniformly the NZ was dispersed (as shown in Fig. 12), which lead to the water absorbed on the surface of NZ increase. In the process of hydration, the water absorbed on the surface of NZ release slowly. Therefore, the cement hydration degree decrease. As shown in Fig. 9 and Table 7, the CH orientation increase when ultrasonic time is less than 5 min. When the ultrasonic time is greater than or equal to 5 min, the NZ was dispersed more uniformly. The filling effect of NZ makes the matrix denser, which limits the growth of CH. Thus, the mass of CH is also decrease (as shown in Table 6). Because the composite is a complex and multiphase material, different factors have different effects on the composite in different

conditions of ultrasonic time. Combined, the contribution of NZ sonicated for 5 min to the strength of composite is larger than those for 2 min and 10 min. The flexural and compressive strength both increase when materials were mix at speed of 3000 r/min. Moreover, the increase rate of both flexural and compressive strength of code J is higher than those of series C. It can be seen from microscopic analysis that the cement hydration degree increase and the CH orientation decrease. As shown in Fig. 13 (a), the disorientation of CH can prevent the extension of cracks, which can make contribution to the strength of composites. It can be seen from Fig. 13 (b), the matrix became denser when materials were mix at high speed. Maybe materials were mixed more uniformly at high-speed, which promote the cement hydration and make the matrix denser to limits the growth of CH.

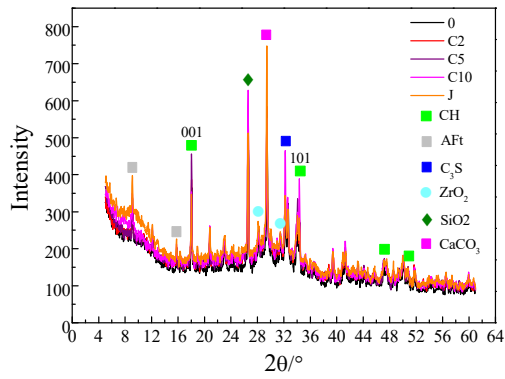


Fig. 9 XRD patterns of 0, series C and J at curing age of 28 d

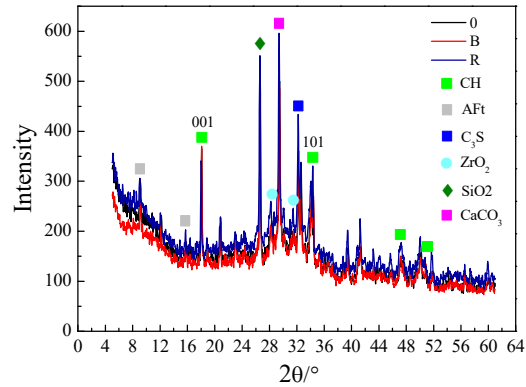


Fig. 10 XRD patterns of 0, B and R at curing age of 48 h and 28 d

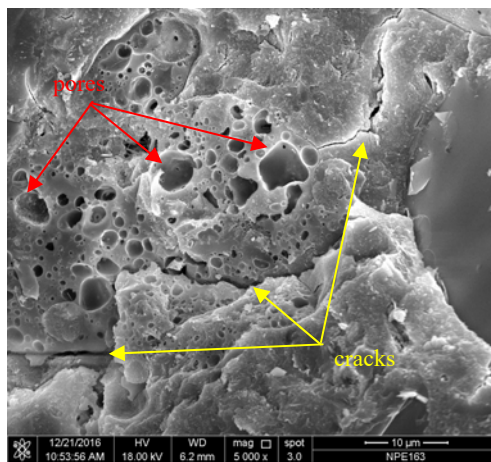


Fig. 11 SEM images of code W

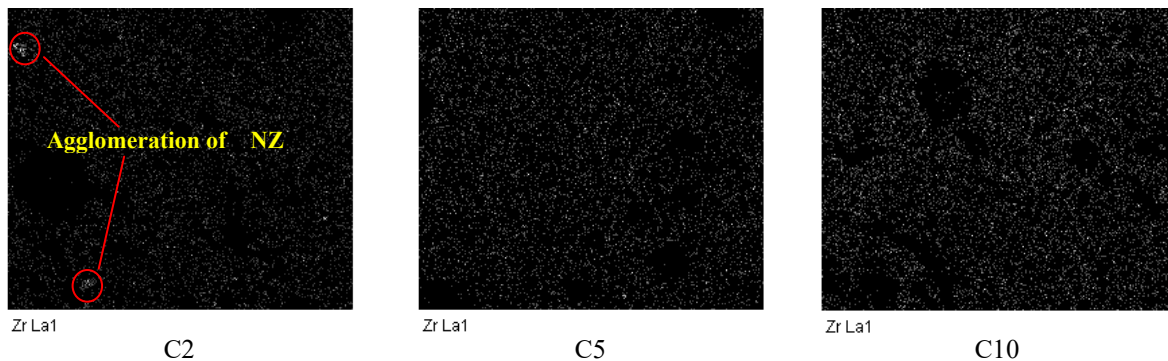


Fig. 12 The distribution of NZ inside RPC (200×)

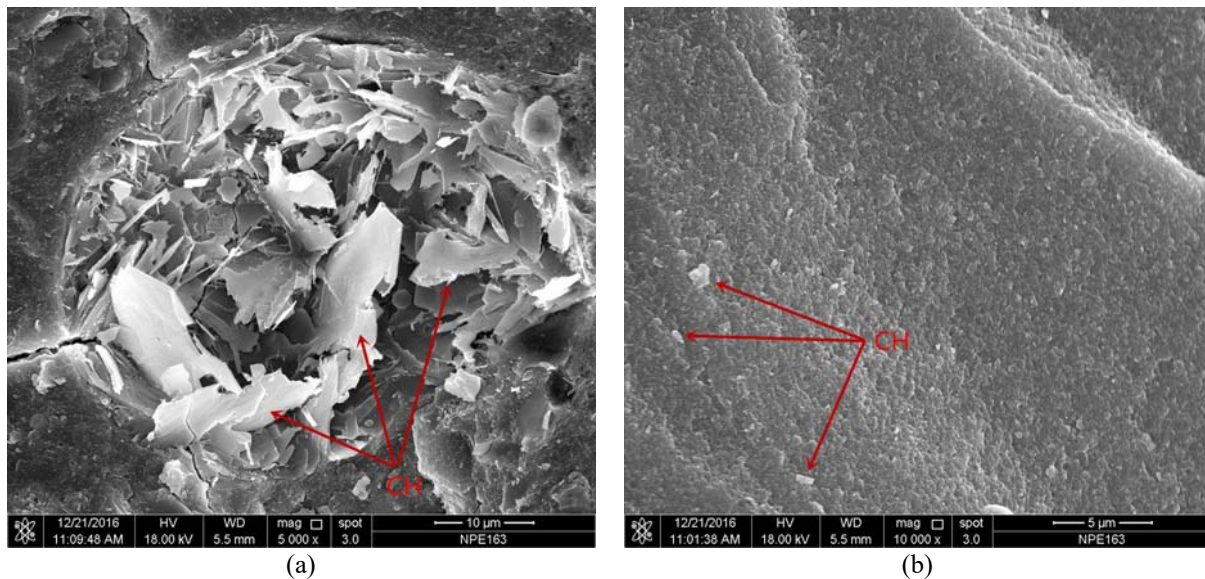


Fig. 13 SEM images of code J

Table 5 Cement hydration degree at 48 h and 28 d/%

Age	Sample codes							
	0	W	C2	C5	C10	J	B	R
48 h and 28 d	44.5	47.5	46.0	40.1	44.2	49.9	45.9	49.5

Table 6 The mass loss at 400-550°C /%

Sample codes	411 °C	501 °C	Mass of CH
0	91.159	89.918	5.102
W	90.994	89.791	4.942
C2	90.425	89.318	4.551
C5	90.666	89.487	4.847
C10	91.117	90.110	4.140
J	89.021	87.785	5.081
B	90.860	89.706	5.492
R	90.402	89.066	4.744

Table 7 Diffraction intensity and orientation of CH at 48 h and 28 d

Sample codes	(001)CH	(101)CH	Orientation of CH
0	365	271	1.818
W	394	305	1.744
C2	387	264	1.979
C5	456	335	1.838
C10	354	388	1.232
J	346	284	1.645
B	370	262	1.906
R	341	332	1.387

It also can be seen from Table 5, 6 and 7 that the cement hydration degree of code B increases. But the mass and orientation of CH increase when the composites were curing in saturate lime water, which are not beneficial to the strength. However, the strength of composite cured in water at 90 °C increase. The reason is that the increase in temperature accelerated the hydration reaction of cement, the mass of CH and CH orientation both decrease and heating curing can accelerate the secondary reaction of fly ash and improve the interface of RPC. The compactness of matrix became very high, and internal moisture was difficult to evaporate. Therefore, the strength of concrete are promoted to increase[17].

#### 4. Conclusion



NZ is employed as filler to modify RPC. The effect of different preparation technologies on mechanical properties of RPC are investigated.

The flexural strength of RPC with NZ added externally increase 14.4%, the compressive strength and splitting strength of that decrease 8.5% and 15.3%, respectively, through the mass and orientation of CH, decrease. NZ with ultrasound treatment is beneficial for the flexural strength of composites, but decreases the compressive strength of the composites. The splitting strengths don't increase obviously, either. However, the flexural and compressive strength of composites produced by high mixing speed of 3000 r/min both increase, while the splitting strength decrease 35.6%. Curing in saturated lime water can improve the flexural and splitting strength, which contrary to compressive strength. High temperature curing media make contribution to the flexural, compressive and splitting strength, the increase rates are 35.0%, 15.0% and 17.0%, respectively. Preparation technologies of high temperature curing media, high mixing speed and NZ added external can increase the hydration degree of cement and decrease the mass and orientation of CH. However, saturate lime water curing media not only would increase hydration degree of cement, but also would increase the mass and orientation of CH which are bad for strength of RPC. Preparation technologies of ultrasound can decrease the mass of CH, but the CH orientation increase when the ultrasound time is 2 min and 5 min. The cement hydration degree of RPC decreases with the increasing ultrasound time.

Because the RPC is a complex and multiphase material, different factors have different effects on the composite in different conditions of preparation. In summary, high temperature curing media makes a greater contribution to the strength of composites. However, the effects of same preparation technology on flexural and compressive strength are different, which still requires for further study.

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