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Properties and Microstructure of Roller Compacted Concrete with High Volume Low Quality Fly Ash

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The properties of roller compacted concrete (RCC) with high volume low quality fly ash are investigated, including strength, elastic modulus, impermeability and frost resistance, meanwhile the microstructure of the cement paste containing low quality fly ash and ground low quality fly ash are also studied by using X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetry-differential thermal analysis (TG-DTA) and Mercury Intrusion Porosimetry (MIP). The results indicate that the properties of RCC containing 60 % or more ground fly ash meet the design requirement. When incorporated with high volume (60 %) ground fly ash, the effect of the pozzolanic reaction was observed at 7 days by consumption of portlandite. At the hydration age of 90 days, a drastic pozzolanic reaction was observed by the depletion of CH and the formation of substantial secondary products. Moreover, the property development of the cement containing 60 % ground fly ash is much faster and enhanced than that compound with same content of raw fly ash. The incorporation of high volume (60 %) ground low quality FA not only refine the pore size but also ameliorate the pore size distribution of cement pastes, which greatly promotes the development of microstructure and the consequent strength and durability of RRCs.
Keywords: roller compacted concrete, low-quality fly ash, properties, microstructure.

1. INTRODUCTION

Cement consumption in China has accounted for more than half of the global production. As a result, the use of low-cost active industrial waste in concrete to partly replace cement and increase concrete properties has become an inevitable trend for the sustainable development of the concrete industry. Since 1970s, the dosage of fly ash used in concrete has increased greatly [1, 2]. The content of fly ash in roller compacted concrete (hereafter RCC) dam is often greater than 50%. In the past three decades, fly ash has always been used in concrete preparation. There has been a growing demand for fly ash as a mineral admixture [3 – 5]. China is rich in fly ash, and its comprehensive utilization has attracted nationwide attention. Therefore, the government is advocating for environmental protection and the economy growth through vigorous support for thermal power generation [6]. The China twelfth-five plan is intended to improve the comprehensive utilization of fly ash to 70 % by the year 2015, and will increase the waste utilization to 60 million tons.

Low quality fly ash (low quality FA) is rarely used since it cannot meet the requirement of the secondary fly ash, which results in severe environmental pollution [7, 8]. Moreover, the study of low quality fly ash is very limited and many problems are still under debate. For example, it is still unknown whether low quality fly ash has the same activity stimulation and hydration mechanism as normal fly ash. How to calculate the contribution proportion of the physical and chemical activity during the hydration process of cement-based materials is still not clear and the hydration mechanism requires further study [9, 10]. A laboratory investigation was carried out to evaluate the strength properties of high-volume fly ash roller compacted and superplasticised workable concrete cured at moist conditions which showed that high-strength concrete was possible with high-volume fly ash content. And it was concluded that high-volume fly ash RRC was an adequate material for both structural and pavement applications [11, 12]. RCCs with moderate cement and fly ash contents had lower values of permeability, absorption, and sorption and chloride diffusivity [13].

In this work, three kinds of RRC, which designed basing on the engineering requirement, are prepared with high volume low quality fly ash firstly. The properties including compressive strength, elastic modulus, permeability and frost resistance are investigated to test whether the properties of RRCs meet the demands. Some mechanisms are also studied with aid of micro-test including XRD, TG-DTA, SEM and MIP, the grinding technology is also used to improve the hydration properties of low quality fly ash.

2. EXPERIMENTAL

The chemical compositions of Lijiang 42.5 ordinary Portland cement and Madiwan fly ash are listed in Table 1. The proportion of coarse particles of raw fly ash expressed as 45 μm sieve residue is 49.8 %, which exceeds the limitation of the secondary grade. After ground for 45 min, the 45 μm sieve residue falls to 19.7 % from 49.8 % which

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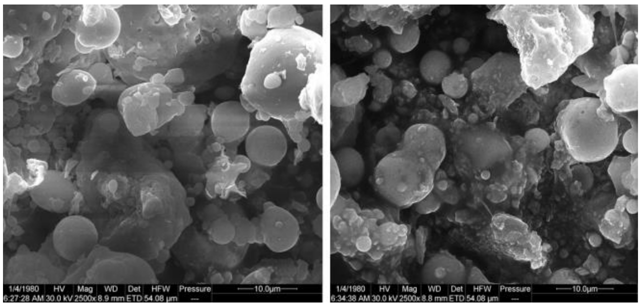
meets the requirements of secondary fly ash. The morphology of raw and ground fly ash is shown in Fig. 1.

Based on Lijiang 42.5 moderate heat Portland cement and Madiwan fly ash, three kinds of RRC, namely $C_{180}15W_{90}6F_{90}100$ (three-grade), $C_{180}20W_{90}8F_{90}100$ (three-grade) and $C_{180}20W_{90}8F_{90}100$ (two-grade), which named as R1, R2 and R3 respectively, are designed according to the requirements for engineering designs. The capital letters of C, W and F, and the subsequent numbers are on behalf of the compressive strength grades, impermeability and frost resistant grades, respectively. The subscripts are on behalf of testing ages. The mix designs of the three roller compacted concrete, R1, R2 and R3, are given in Table 2. All the tests are prepared according to DL/T 5433-2009.



a b

Fig. 1. SEM images: a – raw fly ash; b – ground fly ash

40 mm cubic pastes with different fly ash dosage (20 %, 40 % and 60 % by mass) and type (raw fly ash and ground fly ash) are prepared for the micro-tests, such as XRD, TG-DTA, SEM and MIP. The pure Portland cement (OPC) was also cast for comparison. Fly ash-cement with 20 %, 40 %, 60 % raw fly ash are denoted as R-20, R-40, R-60 for short, and those with 20 %, 40 %, 60 % ground fly ash are expressed as G-20, G-40, G-60 for short. The specimens were broken and the clean inner part of the specimen is collected and soaked in absolute ethanol to terminate the hydration. The granules are selected to test for MIP test, while the sample pieces are delivered for SEM test, the rest of the granules and pieces are ground in the agate mortar and dried in 60 ℃ for 2 hours to eliminate the carbonization for XRD test and dried in vacuum condition for TG-DTA test.

XRD experiments are measured by X-ray Diffraction Analyzer with D/MAX-RB models of target X-ray diffraction, using copper cathode and continuous scan, produced by a Japanese company. TG–DTA experiments are measured on the Diamond TG/DTA analyzer, produced by Perkin Elmer Instruments Plant (Shanghai), in the temperature range from 20 to 1.000 ℃, using platinum crucibles with approximately 0.004 g of sample, under dynamic N2 atmosphere (50 mL min^{-1}). Morphology of the products is investigated using a scanning electron microscopy with JSM-5610LV model of a Japanese company). An Auto Pore IV (9500) Mercury Injection Pore Apparatus was used to calculate pore sizes between 30 Å and 0.9 mm, and pressures between 0.20 and 60.000.00 psi (about 413.76 MPa to 1.38 kPa). Mercury Injection apparatus is Auto Pore IV (9500), the pressure ranges from 0.20 to 60000.00 psi (about 413.76 MPa to 1.38 kPa), and calculation of measuring pore size range is about 30 Å – 0.9 mm. The impermeability grade and frost resistant grade are tested in reference to Chinese standard SL 352-2006 Test code for hydraulic concrete.

3. RESULTS AND DISCUSSION

3.1. Properties

3.1.1. Compressive strength

The compressive strength of R1, R2 and R3 are shown in Fig. 2. The results indicated that compressive strength of RCCs is relative low at early age 7 days, while it tends to grow appreciably at later ages. At early age (before 7 days), the influence of fly ash on the hydration is mainly related to the "filler effect" [5], however, the positive effect of "filler effect" may not compensate the negative effect caused by sharp reduction of cement, therefore, strength at 7 days is relative low compare to that of later ages. From 7 days, the pozzolanic activity of fly ash was stimulated and pozzolanic reaction was dominant in the hydration of RRCs, thus the strength grows substantially at later stage. These results are consist with former researches [16 – 18].

Table 1. Chemical composition of cement and fly ash /Mass, %

Materials	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOI
Cement	18.97	5.18	4.01	61.58	4.24	0.52	0.23	3.26	0.87
Fly ash	53.41	4.28	25.79	2.60	2.79	1.38	0.42	0.30	3.67

Table 2. Mix design of the roller compacted concrete /$kg \cdot m^{-3}$

Sample	Water	Cement	Fly ash	Fly ash content	Sand	W/B	Gravel, mm			Superplasticizer (JM-Ⅱ(R1))	Air entraining agent(JM-YQJ)
							5-20	20-40	40-80		
R1	81	51	96	65%	730	0.55	455	606	455	0.887	0.066
R2	80	63	97	60%	716	0.5	456	609	456	0.962	0.072
R3	92	73	110	60%	794	0.5	705	705	/	1.100	0.082

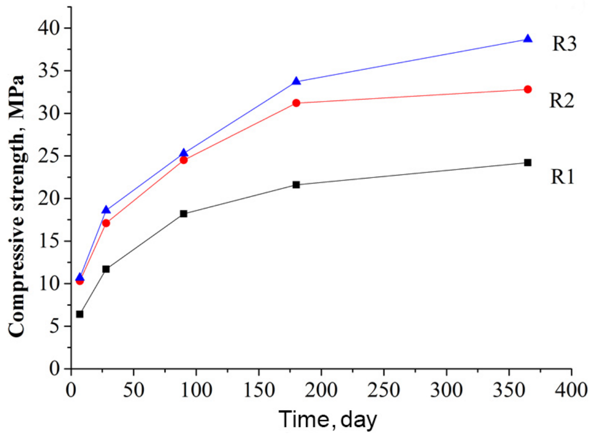


Fig. 2. Compressive strength of RCCs

It is also observed that the compressive strength of R1, R2 and R3 at 90 days are 18.2 MPa, 24.3 MPa, and 25.5 MPa respectively, which meet the design requirement. In addition, the fast strength development at later age after 90 days leads to the additional strength far exceeding the designed strength. This indicate that the RRC with high volume low quality fly ash (≥ 60 %) can completely meet the demands for engineering practice.

3.1.2. Static elastic modulus

Table 3 show the static elastic modulus and modulus to compressive strength ratio of RCC. The static elastic modulus of the 3 RCCs increases over curing age, while it almost becomes steady after 90 days. The elastic modulus of the 3 RCCs reduces as the order as R3, R2 and R1, which is the same as the trend for the strength. In this study, the elastic modulus of RCC with high volume fly ash is relatively low at early age. Though the elastic modulus increases over curing age, the strength increases faster, thus the modulus to compressive strength ratio reduces, which indicates that the 3 RCCs is characterized by a high crack resistance [2].

Table 3. Static elastic modulus /GPa and modulus to compressive strength ratio /$\times 10^3$ of RCCs

Sample	7 days	28 days	90 days	180 days	365 days
R1	12.6/1.97	20.1/1.72	31.2/1.71	34.5/1.60	35.1/1.45
R2	20.5/1.99	27.3/1.60	36.8/1.50	41.2/1.32	41.5/1.26
R3	22.9/2.41	30.1/1.62	39.4/1.56	41.8/1.24	42.0/1.08

3.1.3. Impermeability and frost resistance

Table 4 show the impermeability grade of RRCs. The impermeability grade of the 3 RCCs is examined by the maximum water pressure sustained on the standard samples at the age of 90 days, which meets the design requirements. In order to improve the frost resistant of RCC, air-entraining agent is added to control the air-content of the 3 RCCs higher than 4.5 %. After 100 circles of freeze-thaw, all 3 RCCs have a mass loss ratio of lower than 5 %, while the relative dynamic modulus is all higher than 60 % in Table 5, which indicates that the frost resistant grade reaches F100 and meets the design requirement. In addition to mechanical properties, the impermeability and frost resistance of RRCs with high volume low quality fly ash also satisfies the demands for engineering practice.

Table 4. Impermeability grade of RCCs

Sample	Impermeability grade	Seepage depth, mm
R1	≥W6	37.6
R2	≥W8	49.7
R3	≥W8	21.3

Table 5. Mass loss ratio and relative dynamic modulus of RCCs under different freezing-thawing cycles (C)

Sample	Mass loss ratio, %				Relative dynamic modulus, %			
	25 C	50 C	75 C	100 C	25 C	50 C	75 C	100 C
R1	1.26	1.38	2.38	2.84	92.3	91.1	85.4	76.9
R2	0.83	0.98	1.67	2.03	95.7	93.6	87.3	84.2
R3	1.14	1.22	1.96	2.61	97.1	94.3	91.8	85.0

3.2. Microstructure

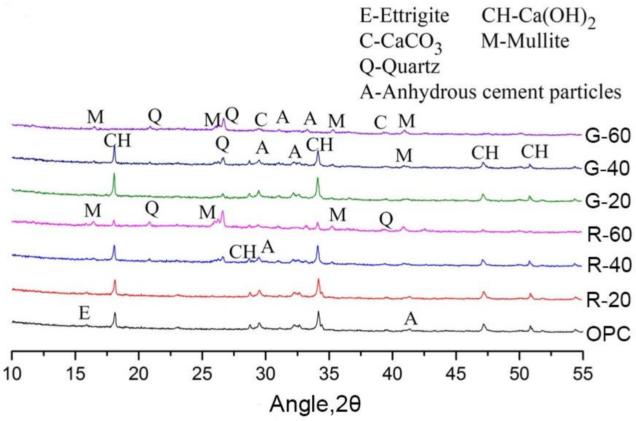
Concrete is composed of cement paste, aggregate and the interfacial transition zone (ITZ), so is RCC. It well accepted that the mechanical and durability of concrete depends mainly on the properties of cement paste, or as well as the coherent strength at the ITZ. Therefore, the investigation of hydration products and microstructure of cement-fly ash paste is conducive to understanding of the effect of high volume fly ash on properties of RCC. In the following study, the microstructure and hydration products of fly ash-cement paste compound with 20%, 40%, 60% raw low quality fly ash (R group) and same content of ground low quality fly ash (G group) are tested by XRD, TG-DTA, SEM, and MIP.

3.2.1. Hydration products

XRD patterns of both R and G group at curing age of 7 and 90 days are shown in Fig. 3. The identified crystalline phases of both R and G group are ettringite, portlandite, calcite, quartz, mullite and anhydrous cement particles, while only ettringite, portlandite and anhydrous cement particles crystals appeared in OPC sample. It illustrated that the types of hydration products are expanded due to the incorporation of fly ash.

With the increase of fly ash content, the diffraction intensity of CH weakened, which may be caused by both the decrease of cement dosage and the pozzolanic reaction of fly ash. With curing age proceeds, the CH characteristic peak of both R and G group decreased, especially for G group whose fly ash is ground. It indicates that pozzolanic reaction occurs during the curing age from 7 days to 90 days, besides, grinding can improve the activity of fly ash and enhance the pozzolanic reaction to consume more CH. When the content fly ash reaches to 60%, the characteristic peak of CH is very low even at 7 days and almost disappeared at 90 days, which indicates exactly that the pozzolanic reaction may occurs even at 7 days.

As CH with preferential orientation precipitates around aggregate and forms ITZ in RRC, which may be harmful to the strength and durability. However, fly ash consumes large quantities of CH to form low C/S ratio C-S-H, which is characterized by high strength and stability. In addition, the ground fly ash also contributes to the strength development of RCC. On one hand, the particle feature such as fineness and particle distribution may be improved by grinding, resulting in better morphology and filling effect of fly ash on the properties. On the other hand, grinding also increases the reactivity of fly ash effectively as previously mentioned, which is possibly the consequence of early pozzolanic reaction and high CH consumption compare to the raw fly ash-cement paste. As a result, fly ash, especially for the ground one, may greatly promote the properties of RRC, both early and long term properties.



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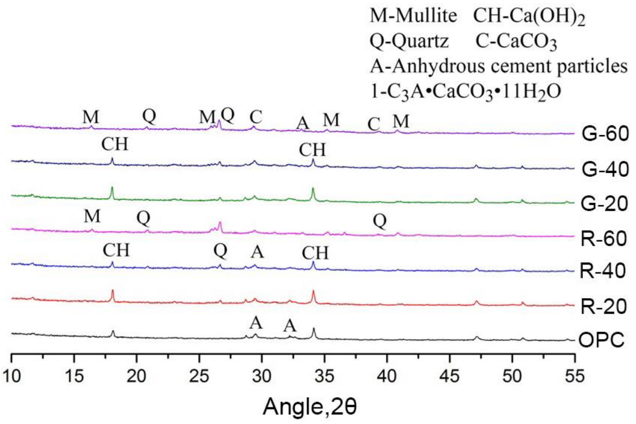
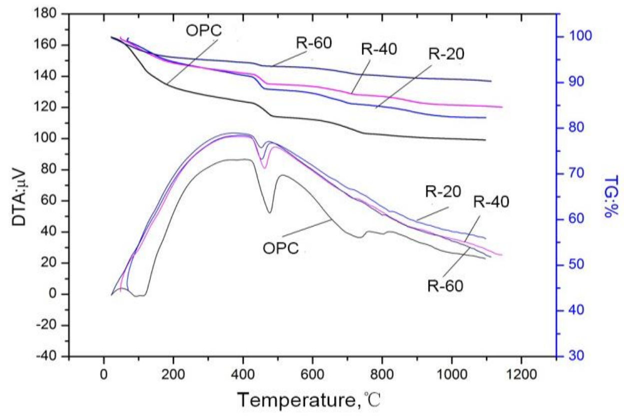


b

Fig. 3. XRD results of samples at: a – 7 days; b – 90 days

TG-DTA has been used to further study the effect of fly ash on hydration products of the system as shown in Fig. 4 (TG-up, DTA-down), based on which the CH content at 7 and 90 days are calculated in Table 6 (the TG-DTA curves at 90 days not present here due to limit space).

The secondary pozzolanic reaction of fly ash occurred under the excitation of hydrates of cement clinker, i.e., CH, which contributed to the strength. The hydration degree of fly ash can be reflected indirectly by the amount of calcium consumed. TG analysis can accurately determine the quantity of CH. The mass loss of CH and the heat absorption occur at 455 °C during the dehydration process. The amount of CH in the pure cement paste increases gradually with hydration time, which is in contrast to that of the fly ash-cement paste. It may be caused by the reduction of cement content and the secondary reaction of fly ash which consumes CH. Compared to the raw fly ash, the CH content of the ground fly ash-cement paste is very low, especially at 90 days, even no endothermic peak of CH is observed in the cement paste with 60 % ground fly ash at both 7 and 90 days, which is consistent with the results of XRD.



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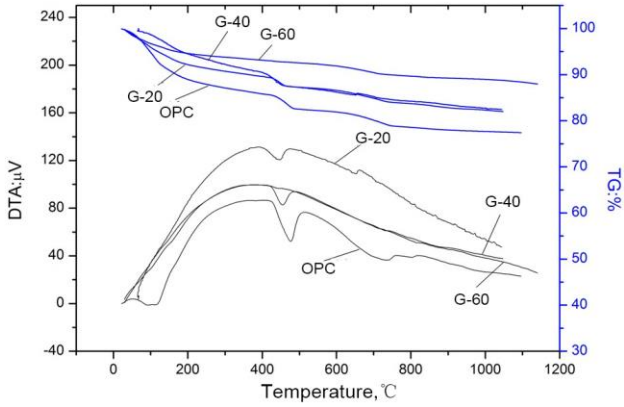


b

Fig. 4. TG-DTA curves: a – sample R-20, R-40, R-60, OPC; b – sample G-20, G-40, G-60, OPC at 7 days

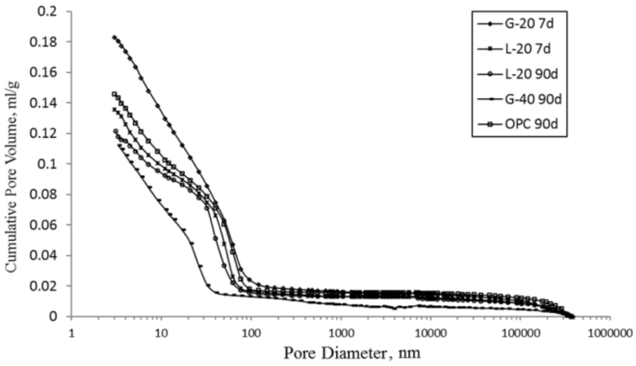
Grinding may arouse the pozzolanic activity of fly ash, and the pozzolanic reaction may even occurs at 7 days, and the CH formed by cement is almost consumed completely for the ground FA with the hydration prolonged. That is to say, less CH but more C-S-H may be formed in G group than in R group at same curing age, leading to improved strength as well as durability. The results again verify that the high volume ground fly ash are favorable to the properties of RRC.

Table 6. $Ca(OH)_2$ content in hydration products of the samples

Age	OPC	R-20	R-40	R-60	G-20	G-40	G-60
7 days	13.46	12.53	9.59	2.92	12.18	8.76	0
90 days	16.39	11.35	9.37	0.85	11.04	6.35	0

3.2.2. Pore structure

Porosity is one of the most important parameters to evaluate the properties of concrete which has a close relationship with strength and durability. In order to investigate the distinction between "physical effect", i.e., morphology effect and micro-aggregate effect, and pozzolanic reaction. Limestone powder, which is often works as inert admixture [5], is also used to partially replace the cement and in comparision with fly-ash cement regarding the porosity in this part. The cumulative and differential pore size distribution of cement with 2 0% ground fly ash (G-20), with 20 % limestone (L-20), with 40 % ground fly ash (G-40) and pure cement paste (OPC) are shown in Fig. 5. The relevant characteristic apertures are given in Table 7.



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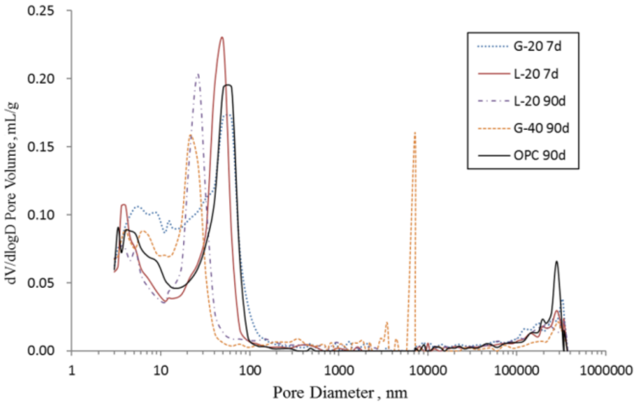


b

Fig. 5. a – Cumulative pore size distribution; b – Pore size distribution of the samples

In order to gain more insight into pore size distribution, the pore distributions present in Fig. 5 are divided into four size ranges [7]: gel micro-pores (< 4.5 nm), mesopores (4.5 – 50 nm), middle capillary pores (50-100nm) and large capillary pores (> 100 nm). The pore volume in each range are calculated and depicted in Fig. 6. At 7 days, the porosity of the paste with fly ash (G-20) is higher than that of paste with limestone powder (L-20) as well as OPC. At 90 days, despite that the total porosity of paste rises with the increase of fly ash [14], the porosity of sample G-40 is higher than that of G-20. The porosity of paste containing 40 % fly ash (G-40), however, is still lower than that containing 20 % limestone powder (L-20), and the porosity of both G-40 and L-20 is lower than that of the pure cement paste (OPC). Low quality fly ash plays a vital role in the later stage of hydration, leading to denser microstructure and higher durability of RRCs. Furthermore, low quality fly ash promotes the transformation of medium capillaries (50 – 100 nm) to interval pores (4.5 – 50 nm) significantly (Fig. 6). In general, the pores with the size lower than 50 nm do little harm to the concrete properties, which means the RCC containing high volume fly ash can show superior durability at later stage.

As shown in Table 7, the median pore (surface area) Rs is ranged from 5.1 nm to 6.4 nm, which is consistent with inter granular pores of C-S-H [15]. Therefore, the replacement level and hydration time have little impact on the median pore diameter of area. While a huge difference of the median pore (volume) Rv has been observed. Limestone powder can decrease Rv value slightly owing to the filling effect. In addition to the filling effect, the pozzolanic effect of fly ash also decreases the large pore size markedly, leading to Rv as low as 16.7, far less than that of OPC. Additionally, Rv reflects the fact that capillaries play an important role in porosity [16]. As a result, FA can effectively reduce Rv, which is favorable to the enhancement of strength and durability of RRC.

The threshold aperture, the most probable aperture and average pore diameter reflects the pore size and pore size distribution of pastes. It is clearly that limestone powder decreases these values more than FA at 7 days, but it appears contradictory trend at 90 days. These three values descend even more with increase of FA content. It again demonstrates that the incorporation of high volume ground low quality FA not only refines the pore size but also ameliorates the pore size distribution of RRC.

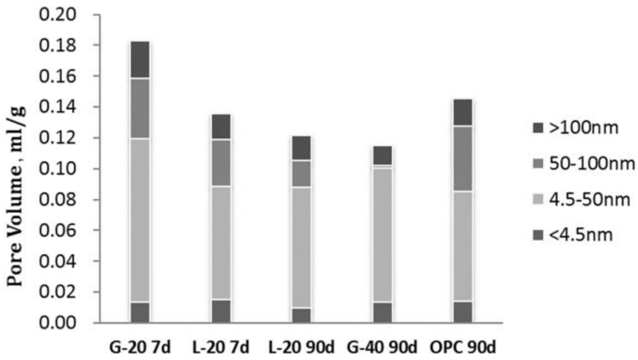


Fig. 6. Pore volume distribution

3.2.3. Morphology analysis

The morphology of hydration products of R-60 and G-60 are investigated and the SEM images are shown in Fig. 7. At 28 days, the fly ash has been found surrounded by a layer of hydration products, which is most probably formed by the hydration of OPC.

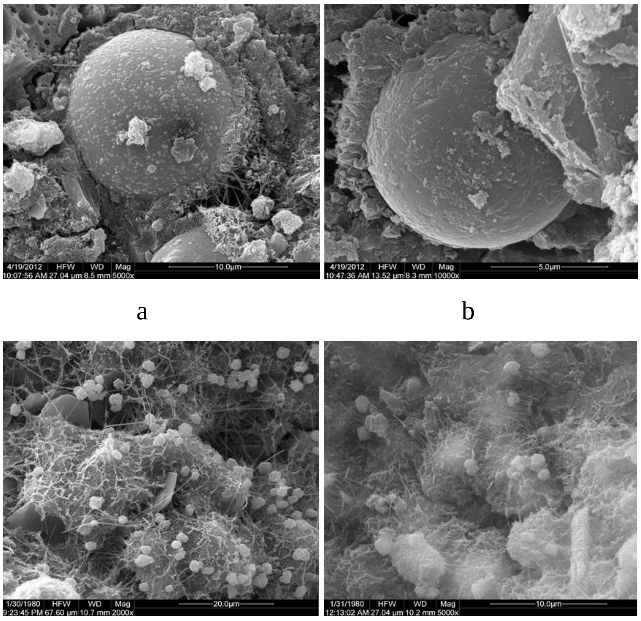
Crossing a seam bulk of C-S-H gel are also observed to be the outermost layer. Furthermore, small granular protrusion is found on the smooth surface of raw FA particles (Fig. 7 a). This granular protrusion may be nucleation and crystal growth sites of inner products, indicating the beginning of the pozzolanic reaction. Compare with raw FA, the surface of ground FA featured by more embryonic form (Fig. 7 b).

It may be due to the high pozzolanic reactivity of ground FA. At 90 days, the surface of FA particles are eroded badly, the spherical shape of FA particle as lots of inner products are formed on the remaining unreacted fly ash particles. These inner products are highly porous [17], but the inner products of G-60 are relative more than those of R-60. The fine fibers on the surface are presumably C-S-H. Additionally, flaky orthohexagonal CH can be observed in R-60 but not in G-60.

Table 7. Median pore diameter and average pore diameter /nm

Samples	Median pore diameter		The average pore diameter (4V/A, Ra)	The threshold pore size	The most probable pore size
	Pore volume (Rv)	Pore surface area (Rs)			
G-20, 7 days	28.4	6.4	14.0	102	60
L-20, 7 days	39.0	5.1	13.6	90	48
L-20, 90 days	36.3	5.8	15.3	80	34
G-40, 90 days	16.7	5.9	10.5	40	22
OPC, 90 days	43.9	5.8	16.1	100	60

The high pozzolanic reactivity of ground FA depletes the CH, which is consistent with the results of XRD and TG-DTA. To summarize, FA not only has a "filling effect" on the hydration of RRC, but also acts as nucleation site of hydration products for cement at early stages. Besides, a large amount of secondary hydration products is formed by the pozzolanic reaction of FA at later stage, which greatly improves the microstructure, and the consequent strength and durability gain of RRC.



a b

c d

Fig. 7. SEM images of samples: a – 60 % raw fly ash, 28 days; b – 60 % ground fly ash, 28 days; c – 60 % raw fly ash, 90 days; d – 60 % ground fly ash, 90 days

4. CONCLUSIONS

1. Three RCCs with high volume fly ash, i.e., $C_{180}15W_{90}6F_{90}100$ (three-graded) and $C_{180}20W_{90}8F_{90}100$ (three-graded, two-graded), are prepared and meet the designed engineering requirements.
2. The effect of high volume (60%) raw low quality fly ash at early age (<7d) are mainly related to filling effect and the effects of the pozzolanic reaction were observed at 28 days. When incorporated with high volume ground fly ash, the effect of the pozzolanic reaction was observed at 7 days by consumption of portlandite based on XRD and TG-DTA test. At 90 days, a drastic pozzolanic reaction was observed by the depletion of CH and the formation of substantial secondary products based on SEM, TG-DTA and SEM tests. It combines with the "filling effect" of fly ash, and they are the main contributors for the RRC properties.
3. The incorporation of high volume ground low quality FA not only refine the pore size but also ameliorate the pore size distribution of cement pastes, which greatly promotes the development of microstructure and the consequent strength and durability gain of RRCs.

Acknowledgements

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