

Article



Comparing Permeability and Drying Shrinkage of the Concrete Containing Mineral Admixtures under the Equal Strength Grade

Yufeng Fan¹, Qiang Zhao² and Dengquan Wang^{3,*}

- ¹ China Construction Second Bureau Installation Engineering Co., Ltd., Beijing 100176, China
- ² Beijing Urban Construction Group Co., Ltd., Beijing 100082, China
- ³ Department of Civil Engineering, Tsinghua University, Beijing 100084, China
- * Correspondence: wangdq16@mails.tsinghua.edu.cn; Tel.: +86-188-1135-1597

Abstract: Fly ash (FA) and ground granulated blast-furnace slag (GGBS) are the most widely used mineral admixtures in engineering. However, their roles in concrete under the equal strength grade, a common comparison method in engineering, were seldom reported. This study investigated the chloride ion permeability and drying shrinkage of concrete samples containing FA or GGBS under an equal strength grade. The samples' strengths and slumps maintained the same levels by adjusting the water-to-binder ratios and superplasticizer dosages. The results show that both FA and GGBS can promote the resistance to chloride ion penetration and decrease the chloride diffusion coefficients, especially at late ages, due to the hydraulicity of GGBS and pozzolanic activity of FA. Compared with FA, GGBS presents a greater reduction in the concrete permeability due to its higher reactivity. Forty percent replacement levels of FA and GGBS can decrease the penetration level from "high" of plain cement concrete to "moderate" and "low", respectively. In addition, FA and GGBS can decrease the drying shrinkage of concrete at high replacement levels (30% and 40%). This decrease is more significant in the FA-containing concrete, with the shrinkage decreasing from approximately 400 $\mu\epsilon$ to 350 $\mu\epsilon$ at a 40% replacement level. The findings can provide scientific guidance for applying FA and GGBS in practical engineering.

Keywords: fly ash; ground granulated blast-furnace slag; equal strength grade; chloride ion permeability; drying shrinkage

1. Introduction

Concrete is the most widely used construction material of humankind due to its high workability, low cost, high ultimate strength, and good durability [1]. In practical engineering, however, the service lives of concrete structures are severely shortened by the chloride ion penetration and drying shrinkage [2–5]. Chloride ions can cause the corrosion of reinforcements in concrete structures, especially in coastal and saline soil areas [6,7]. In addition, drying shrinkage is one of the main causes of concrete cracking [2,3]. These concrete "diseases" deteriorate the concrete performance and degrade the structure durability.

Mineral admixtures, e.g., fly ash (FA) and ground granulated blast-furnace slag (GGBS), can promote the concrete performances and improve the durability [8–12], which have been widely applied in engineering for decades [9,13,14]. FA is a by-product of coal-fired power plants. Despite decreasing early-age strength, FA improves the late-age strength and has positive impact on the durability of concrete [8]. It shows very high resistance to chloride ion penetration at late ages compared with plain concrete [5]. Liu et al. [15] found that concrete obtains the lowest drying shrinkage with 10% FA replacement and shows increased drying shrinkage if the FA content exceeds this value.

GGBS is a ground powder of stone-like industrial by-product produced in the ironmaking process, which shows high hydraulicity [11,13]. Ozbay et al. [16] reported that GGBS enhanced concrete's long-term compressive strength and elastic modulus of concrete.



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). In addition, GGBS could improve the durability of concrete, including reducing creep, drying shrinkage, chloride ion permeability, and inhibiting sulfate and alkali-silica reactions [16]. Qureshi et al. [17] found that chloride ion penetration resistance was significantly enhanced with GGBS incorporation in both plain and hook-ended steel fiber-reinforced concrete. Compared with ordinary concrete, the drying shrinkage of concrete containing GGBS as a cement substitute is significantly reduced, and a 30% GGBS substitution results in a 20% reduction [15]. In addition, due to the filling effect of GGBS, it can reduce the porosity of concrete and improve the microstructure [7]. Li et al. [18] also exhibited that GGBS could significantly decrease pore volumes and reduce the most probable pore size from 10–100 nm to the range of <10 nm. The high-performance concrete shows a continuous decrease in drying shrinkage with the substitution of 5%–20% GGBS in the first 28 days; the optimal content of GGBS is 20% [19]. However, some studies showed that the creep and shrinkage of concrete containing GGBS are more significant than those of plain concrete because concrete strength development occurs more slowly [20].

In short, there are many in-depth studies on the properties of concrete containing FA and GGBS. However, in practical engineering, concrete formulation is designed according to the 28-day strength. Therefore, the strength index must be first met. Existing research on fly ash and GGBS seldom considers the design of concrete formulation in engineering. Therefore, it is crucial to understand the roles of fly ash and GGBS on the properties of concrete under an equal strength grade. This study investigated the chloride ion permeability and drying shrinkage of the concrete samples with FA and GGBS as partial substitution of the cement under equal 28-day strength (strength grade: 30 MPa). The roles of FA and GGBS on concrete properties were compared systematically. The study aimed to provide scientific guidance for the application of FA and GGBS in practical engineering.

2. Materials and Methods

2.1. Raw Materials

Ordinary Portland cement (OPC), FA, GGBS, crushed limestone, and river sand were used in this study. The OPC (Type I 42.5) conformed to the Chinese National Standard GB 175–2007 (Common Portland Cement). The low-calcium FA conformed to the requirements of Chinese National Standard GB/T 1596–2005 (fly ash used for cement and concrete). GGBS met the requirements of Chinese National Standard GB/T 18046-2000 (ground granulated blast furnace slag used for cement and concrete). The specific surface areas (Blaine method) of OPC, FA, and GGBS were 380 m²/kg, 355 m²/kg, and 415 m²/kg, respectively. The chemical (SHIMADZU Corporation, Japan, XRF-1800) and mineralogical (XRD, Bruker D8, Germany, CuK α , 45 kV, 40 mA) compositions of the materials are shown in Table 1 and Figure 1, respectively. The river sand complied with the Chinese Standard GB/T14684 (sand for construction). Its fineness modulus was 2.66, and its bulk density was 1470 kg/m³. The crushed limestone conformed to the Chinese Standard GB/T14685 (pebble and crushed stone for building), and it had a continuous gradation from 5 mm to 25 mm. In addition, the commercial polycarboxylic superplasticizer was used to adjust the concrete workability.

Table 1. Chemical compositions of the raw materials (wt%).

Composition	CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	SO ₃	MgO	P_2O_5	TiO ₂	Na ₂ O	K ₂ O	LOI
OPC	65.84	18.13	4.30	3.30	3.08	1.95	0.07	0.23	0.17	0.68	1.36
FA	10.23	56.10	17.74	7.59	-	0.92	0.61	1.05	2.02	2.42	1.26
GGBS	36.44	31.76	14.84	0.60	1.94	9.08	-	-	0.56	-	1.09



Figure 1. XRD patterns of cement, FA, and GGBS.

2.2. Mix Proportions and Preparation of Concrete with an Equal Strength Grade

Seven types of concrete samples were designed, as shown in Table 2. The concrete samples were denoted as C1, F20, F30, F40, B20, B30, and B40, corresponding to the plain cement concrete and the concrete at cement replacement levels of 20%, 30%, and 40% by FA and GGBS. For all samples, the binder-to-aggregate ratio was 0.2. The water consumption per cubic meter of concrete was constant, but the water-to-binder (w/b) ratio was varied. After mixing, concrete samples ($100 \times 100 \times 100 \text{ cm}^3$) were prepared and cured at $20 \pm 1 \text{ °C}$ and more than 95% relative humidity.

Sample	OPC	FA	GGBS	Sand	Crushed Limestone	Water	Superplasticizer	w/b
С	340	0	0	816	1016	166	5.9	0.49
F20	284	71	0	801	1014	166	6.7	0.47
F30	255.5	109.5	0	790	1015	166	6.9	0.45
F40	228	152	0	768	1015	166	7.1	0.44
B20	276	0	69	809	1016	166	6.1	0.48
B30	248.5	0	106.5	798	1017	166	6.3	0.47
B40	216	0	144	790	1020	166	6.6	0.46

Table 2. Mix proportions of concrete (kg/m^3) .

The designed slump and strength grade of concrete were 19–21 cm and 30 MPa (28 days), respectively, as shown in Figure 2. The compressive strengths of all samples were between 35 MPa and 40 MPa, which met the original design requirements of this study. Generally, FA and GGBS substitutions for cement reduced the concrete strength if the w/b ratio was constant [8,9,13,21]. Therefore, lower w/b ratios should be applied to the concrete at higher FA and GGBS replacement levels to obtain similar compressive strengths. However, the reduced w/b ratios could decrease the concrete workability, even though the filling effect of FA and GGBS could result in the release of free water from flocculates, and the "ball bearing" effect of FA could provide lubricating functions, which were beneficial for the workability [22]. As a result, increased superplasticizer amounts were needed in the concrete containing FA and GGBS [22,23].



Figure 2. Compressive strengths and slumps of plain cement concrete and concrete containing FA and GGBS.

2.3. Test Methods

Corresponding paste samples were also prepared, which maintained the same mixing ratios except for adding fine and coarse aggregates. The selective dissolution method was employed to determine the reaction degrees of FA and GGBS in hydrated cement pastes at 3, 28, 56, 90, and 360 days based on the European technical report CEN/TR 196-4 (Methods of testing cement—Part 4: Quantitative determination of constituents). In cement-GGBS and cement-FA hardened pastes, the hydration products and the unhydrated cement components are soluble in certain solutions, but FA and GGBS cannot dissolve in such solutions. Accordingly, the amount of unreacted GGBS and FA can be calculated. The hardened pastes, including the plain cement paste, FA-cement paste and GGBS-cement paste, were ground into powder and oven-dried at 105 °C to constant weights. Then corresponding solutions were applied to the powdered samples. The FA-cement sample, as well as the plain cement sample (as a reference), was dissolved using an acid mixture, which was produced using 50 g of salicylic acid and 41 mL of hydrochloric acid (37 wt.%), made up to 1 L with methanol. Then, 200 mL of the acid mixture was used for the dissolution of 2 g of the sample with stirring for 30 min. The insoluble residue was dried at 105 °C for 2 h to determine its weight. The reaction degree of FA (α_{FA}) can be calculated:

$$\alpha_{FA} = \frac{100f - R_b(1 - 1.813 \times S_b) + R_{PC}(1 - f)(1 - 1.813 \times S_{PC})}{100f} \times 100\%$$
(1)

where *f* is the mass fraction of FA in the initial dry binder; R_b is the mass of the residue from the FA-cement composite paste (in g/100 g anhydrous binder); R_{PC} is the mass of the residue from the plain cement paste (in g/100 g anhydrous binder); S_b is the mass of SO₃ in FA-cement composite paste residue (in g/g of residue); and S_{PC} is the mass of SO₃ in the plain cement paste residue (in g/g of residue). The SO₃ content of the residue was measured using XRF.

The GGBS-cement sample and plain cement sample were dissolved using an acid mixture, which was produced using 250 mL of triethanolamine, 93 g of ethylenediamine tetraacetic acid, and 173 mL of diethylamine, with water up to 1000 mL. Then, 50 mL of the solution was diluted with water to approximately 800 mL, and 0.5 g of the sample was placed in the diluted solution, stirring for 2 h. The insoluble residue was dried at 105 °C for 1 h and weighed to determine the residue amount. The reaction degree of GGBS (α_{GGBS}) can be calculated:

$$\alpha_{GGBS} = \frac{100f - R_b + R_{PC}(1 - f)}{f(100 - 2.35M_s)} \times 100\%$$
⁽²⁾

where *f* is the mass fraction of GGBS in the initial dry binder; R_b is the mass of the residue from the GGBS-cement composite paste (in g/100 g anhydrous binder); R_{PC} is the mass of the residue from the plain cement paste (in g/100 g anhydrous binder); and M_s is the mass of MgO in GGBS (in g/100 g GGBS). The MgO content in GGBS was measured using XRF. More details of the method can be found in the reference [24].

In addition, the scanning electron microscope (SEM, HITACHI SU8220) was employed to directly observe the pastes containing FA and GGBS (F30 and B30) at 90 days.

A slump cone test was performed to measure the slump of fresh concrete immediately after mixing according to Chinese Standard GB/T 50080-2016 (standard for test method of performance on ordinary fresh concrete). Fresh concrete samples were placed into the mini-slump instrument in three layers, and then the instrument was lifted quickly. The height difference between the concrete and the instrument was recorded as the slump. The compressive strengths were tested according to the Chinese National Standard GB/T 50081-2011 (Standard for test method of mechanical properties on ordinary concrete) at the ages of 28 days. A constant loading rate of 0.8 MPa/s was applied [25].

The chloride ion permeabilities of the concrete samples at 28 days and 56 days were measured in accordance with ASTM C1202 (Standard test method for electrical indication of concrete's ability to resist chloride ion penetration). The samples of $100 \times 100 \times 50 \text{ mm}^3$ were used to measure the current passing through the concrete with an electron flux determinator [26]. In addition, six concrete cylinders ($\Phi = 100 \text{ mm}$) for each formulation were prepared for rapid chloride migration (RCM) testing in accordance with NT Build 492 to obtain the chloride diffusion coefficients [27,28].

The drying shrinkage tests of concrete samples ($100 \times 100 \times 515 \text{ mm}^3$) were carried out according to Chinese Standard GB/T 50082-2009 (standard for test methods of long-term performance and durability of ordinary concrete). The test was conducted at a fixed temperature of 23 °C and a relative humidity of 45%–60%. The measurements started at 3 days (marked as 0 day of the shrinkage curve) after standard curing. The shrinkage test lasted for 360 days, and the changes of sample lengths were measured using a dial gauge with an accuracy of ± 0.001 mm.

3. Results

3.1. Reaction Degrees of FA and GGBS

FA is a widely used mineral admixture presenting pozzolanic reactivities [13]. The silicaaluminum glass in FA reacts with calcium hydroxide, one of the main products of cement hydration, to form calcium silicates (aluminates) hydrates. The reaction can be described as follows:

$$SiO_2-Al_2O_3 glass + Ca(OH)_2 + H_2O \rightarrow C-(A)-S-H$$
(3)

Different from FA, GGBS shows hydraulic reactivities [13]. Its reaction does not consume $Ca(OH)_2$, and $Ca(OH)_2$ only acts as an alkaline activator for the calcium-silicaaluminum glass of GGBS, as described below:

$$CaO-SiO_2-Al_2O_3 glass + OH^- + H_2O \rightarrow C-(A)-S-H$$
(4)

FA and GGBS in the cement pastes cannot reach complete reactions, even over a long hydration time, due to the kinetic barrier. GGBS commonly presents higher reactivities than FA in the composite cement paste. Figure 3 shows the hydration degrees of FA and GGBS in the composite cement pastes that correspond to the concrete with an equal 28-day strength grade. GGBS shows much faster reaction rates and obtains higher hydration degrees than FA. In addition, for both GGBS and FA, higher replacement levels cause lower hydration degrees over the whole ages due to the lower alkalinity in the pastes. Figure 4 shows the direct observation on the hydration of FA and GGBS in F30 and B30 at 90 days, respectively. Both F30 and B30 present dense microstructures. A small number of hydration products (mainly C-S-H gel) precipitate on the surfaces of FA particles, while a massive C-S-H gel

forms around the GGBS surfaces. It further supports the result that GGBS shows higher reaction degrees than FA under an equal strength grade.



Figure 3. The hydration degrees of FA and GGBS in the composite cement pastes corresponding to the concrete with an equal 28-day strength grade.



Figure 4. SEM observation of (**a**) FA and (**b**) GGBS in the hardened pastes (90 days) corresponding to the concrete with an equal 28-day strength grade.

3.2. Chloride Ion Permeability

Figure 5 presents the charge passed and permeability grade of the concrete under the condition of an equal 28-day strength grade. The charge passed of concrete at 56 days is lower than that at 28 days due to the increased hydration degrees and the denser microstructure [11]. Replacing OPC with FA and GGBS reduces the charge passed of concrete, especially at the later age of 56 days. Incorporating 20% FA can decrease the concrete permeability grade from high permeability (above 4000 C) to moderate permeability (2000–4000 C) at 56 days, but cannot cause this decrease at 28 days. At higher replacement levels with FA (30% and 40%), concrete can obtain a moderate permeability grade at both 28 days and 56 days. It indicates that FA can improve the resistance to chloride ion penetration of the concrete, especially at high replacement levels, which is consistent with the references [8,21]. This improvement can be attributed to the pozzolanic reaction of FA, which can consume Ca(OH)₂ to form C-S-H gel, improve the interfacial transition zone of concrete, and decrease the connectivity of the pores [29]. Compared with FA, GGBS presents a greater improvement in the concrete permeability. A 40% GGBS incorporation can decrease the high permeability grade to low at 56 days. It is because GGBS shows high hydraulicity and hydrates to form additional C-S-H gels, which can better improve the interfacial transition zone of concrete and decrease the connectivity [29]. In addition, the GGBS-cement concrete may also show better chloride ion-binding capacity than FA-cement concrete [30].





Apart from the charge passed, the chloride diffusion coefficient can also characterize the resistance to chloride ion penetration of concrete [6]. Therefore, the chloride diffusion coefficients of concrete with an equal 28-day strength grade are used to further support the roles of FA and GGBS, as shown in Figure 6. The results show similar trends with the charge passed of concrete, which means that the two methods have good consistency and can effectively characterize the resistance to chloride ion penetration of concrete. Based on the results in Figures 3 and 4, it can be concluded that FA and GGBS have positive effects on the resistance to chloride ion penetration of the concrete, especially at a high replacement level (40%), and the ability of GGBS to resist the chloride ion penetration is stronger than that of FA under an equal strength grade.



Figure 6. The chloride diffusion coefficients of the concrete containing FA and GGBS under an equal 28-day strength grade.

3.3. Drying Shrinkage

Figure 7 shows the drying shrinkage of the concrete with an equal strength grade within 1 year. It can be observed that the shrinkages increase rapidly during the initial 12 days. This is due to the rapid loss of moisture from hardened concrete to the environment, which causes a volume reduction of the concrete by the negative capillary pressure [31]. The shrinkage curves slow down after 12 days and become flat after 180 days. It is because with hydration, the denser microstructures make the moisture harder to transfer. The plain cement concrete shows high dry shrinkage values at both the initial and late ages. The high values at the initial stage could be attributed to the high w/b ratio, which makes the

water easier to evaporate. At late ages, the high shrinkage is most likely due to the refined pores, which can produce higher capillary pressure when losing the same amount of water. The substitution of cement by FA and GGBS, especially at high replacement levels, slightly decreases the drying shrinkage at the initial stage due to the reduced w/b ratio. At late ages, high replacement levels (30% and 40% for FA, and 40% for GGBS) can significantly decrease the drying shrinkage. Generally, the concrete shrinkage arises from the capillary pressure generated by the surface tension of the pore solution [32]. This pressure σ_c can be theoretically calculated according to the Young-Laplace Equation [33]:

$$\sigma_c = \frac{2\gamma cos\theta}{r_c} \tag{5}$$

where γ is the surface tension; r_c is the Kelvin radius; and θ is the contact angle. From Equation (5), it can be found that finer pore radius results in greater capillary pressure. Incorporation of a large proportion of FA and GGBS is known to reduce the drying shrinkage [15,34,35]. It is because replacing cement with FA or GGBS increases the pore diameter, as revealed by many researchers. Based on the results from the mercury intrusion porosimetry (MIP), Wang et al. found that the microstructure of cement-FA composite paste (50% replacement level) was coarser than the plain cement paste at 90 days, with the most probable aperture increasing from approximately 40 µm to 70 µm [36]; Park et al. [37] found that replacing cement with 50% FA increased the volume fractions of large pores (50–100 nm and above 100 nm) at 28 and 90 days. Using 2D and 3D imagining, Yio et al. [38] also found that FA replacement (23 wt.%) increased the large pore volume at 90 days. Compared with FA, GGBS is well-known to be more reactive, whose reaction degree can achieve approximately 50%–65% at 90 days, much higher than the approximately 15%–30% of FA [13]. Therefore, low GGBS content tends to refine the microstructure of hardened pastes, but high-volume GGBS can also increase the pore diameter. It was found that a 60% GGBS replacement level increased the volume of large pores at a water-to-binder ratio of 0.45 [38]. Alderete et al. [39] also found that 40% and 60% GGBS incorporation increased the pore volume of mesopores at 28 days from the dynamic water vapor sorption (DVS) data. The discussion on the pore structure is consistent with the shrinkage results in Figure 7 that sample B20, with refined pore structure, shows a higher drying shrinkage value than sample C, while F30, F40, and B40 show lower shrinkage values.



Figure 7. The drying shrinkages of the concrete containing FA and GGBS under an equal 28-day strength grade (dashed line: the shrinkage at 28 days).

4. Conclusions

This study investigated the roles of FA and GGBS on chloride ion permeability and drying shrinkage of concrete under an equal strength grade (30 MPa). The conclusions are summarized as follows:

- (1) The strength of all samples can maintain the same grade of 30MPa at 28 days by decreasing the w/b. The increased superplasticizer dosage is applied to compensate for the loss of slump to keep it at the same level of 29–31 cm.
- (2) GGBS shows much higher reaction degrees than FA at both early and late ages under the condition of an equal 28-day strength grade.
- (3) Under the equal strength grade, replacing OPC with FA and GGBS decreases the charges passed and the chloride diffusion coefficient. FA and GGBS have positive effects on the resistance to chloride ion penetration. This effect of GGBS is stronger than that of FA due to the higher reactivity of GGBS. Forty percent replacement levels of FA and GGBS can decrease the penetration level from "high" of plain cement concrete to "moderate" and "low", respectively.
- (4) The drying shrinkages of concrete decrease with the contents of FA and GGBS. High FA and GGBS replacement levels have positive effects on the drying shrinkage because they reduce the capillary pressure. The ability of FA to decrease dry shrinkage is much stronger, with the shrinkage value decreasing from approximately 400 $\mu\epsilon$ to 350 $\mu\epsilon$ at a 40% replacement level.
- (5) At 30% and 40% cement replacement levels with FA or GGBS, concrete can obtain significantly improved resistance to chloride ion penetration and decreased drying shrinkage.

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