

Article

Influence of Lightly Burned MgO on the Mechanical Properties and Anticarbonization of Cement-Based Materials

Lin Wang ¹, Chao Li ², Chunxue Shu ¹, Han Yong ¹, Jianmin Wang ³ and Hui Wang ^{3,4,*}

¹ School of Civil and Transportation Engineering, Beijing University of Civil Engineering and Architecture, Beijing 100044, China; wanglin1@bucea.edu.cn (L.W.); shuchunxue@bucea.edu.cn (C.S.); yonghan@bucea.edu.cn (H.Y.)

² China Railway Second Bureau Fourth Engineering Co., Ltd., Chengdu 610306, China; caoli@bucea.edu.cn

³ School of Civil and Environmental Engineering, Ningbo University, Ningbo 315000, China; wangjianmin@nbu.edu.cn

⁴ State Key Laboratory of Materials-Oriented Chemical Engineering, Nanjing 210000, China

* Correspondence: wanghui4@nbu.edu.cn

Abstract: This study aims to study the influence of a lightly burned magnesium oxide (LBMO) expansion agent on the rheological properties (the slump flow, plastic viscosity and variation of shear stress) of cement-based materials. Four different mass contents (i.e., 0%, 3%, 6% and 9%) of LBMO were selected. The following compressive strength and expansion value of the corresponding cement concrete were tested. Cement concrete with two strength grades of 30 MPa and 50 MPa (C30 and C50) was selected. Results indicated that the addition of LBMO can effectively decrease the fluidity and increase the plastic viscosity of fresh cement paste. An optimum dosage (3%) of LBMO is the most advantageous to the compressive strength of cement concrete. The addition of LBMO can increase the expansion rate of cement concrete, thus preventing inside cracks. Moreover, the incorporation of LBMO led to a reduction in the fluidity of the cement paste and an increase in plastic viscosity. The addition of LBMO can increase the expansion rate of cement concrete, thus preventing inside cracks. It can be found that little difference exists in the compressive strength and the expansion rate of cement concrete with strength grades of 30 MPa and 50 MPa. Finally, the increased dosage of LBMO, curing age and compressive strength led to improving the carbonization resistance of cement concrete.

Keywords: lightly burned magnesium oxide; rheological properties; compressive strength; expansion value; carbonization resistance



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1. Introduction

Concrete cracking has always been a complex problem in the engineering field. Cracks in concrete seriously affect buildings' safety, shorten the service life and even lead to enormous economic loss [1–5]. Concrete shrinkage is one of the main reasons for concrete cracking [6,7]. Therefore, reducing the shrinkage of concrete is effective to prevent concrete cracking, which is effective to improve the durability of modern concrete [8,9]. Adding an expansive agent into concrete is one of the most effective methods to compensate for the expansion value of concrete, thus preventing the propagation of cracks. Based on these reasons, the use of an excellent expansive agent in concrete is very important for restraining concrete cracking [10]. For the past 20 years, calcium oxide and ettringite expansive agents have been the two most widely used expansive agents. Due to the considerable expansion energy and fast hydration rate, both expansive agents provide a specific anticracking effect if appropriately used in engineering [11,12]. However, calcium oxide and ettringite are expansive agents. The expansion speed is too fast when they are applied in cement-based materials, and an expansive agent with a fast hydration rate may not be able to compensate for the shrinkage effect in the later stage [13,14]. These properties of calcium oxide and

ettringite restrict their wide application in modern concrete structures. In recent years, magnesia-based expansion agents have been used in water conservancy projects, showing good anticracking performance [15,16]. Compared with the expansion agents of calcium oxide and ettringite, the expansion agent of lightly burned magnesium oxide (LBMO) has the advantages of less water demand for hydration, a moderate expansion rate, a long expansion period, delayed microexpansion and a stable hydration product, which makes it more applicable for modern concrete [17–19].

It is well known that concrete with a high cement content results in higher carbon emissions and is susceptible to cracks. Due to the rapid development and progress of high-efficiency water-reducing technology, the water–cement ratio of concrete has been dramatically reduced, which makes it possible to apply low-content clinker cementitious systems [20,21]. The low-clinker cementitious system is the development direction of concrete in the future. Lightly burned magnesia expansive agent is also a new type of expansive agent. Moreover, this lightly burned magnesia expansive agent can be produced with less energy consumption and pollution, which is consistent with the theme of a low-carbon and environmental protection society [22]. Therefore, it is very meaningful to study its application in concrete.

Concrete may deteriorate when exposed to the environment of carbon dioxide. Compared with calcium oxide and ettringite expansive agents, cement-based materials with a lightly burned magnesia expansive agent may show stronger carbonation resistance [23,24]. Additionally, the rheological properties are very critical for the concrete construction quality; however, little research about the rheological properties and the carbonation resistance of calcium oxide and ettringite has been reported.

In this study, the slump flow, plastic viscosity and variation of shear stress with time of cement paste with lightly burned magnesium oxide were determined. Additionally, the following compressive strength and expansion values of the corresponding concrete were tested. Finally, the carbonation depth of concrete with lightly burned magnesium oxide was determined. This research will provide a new idea for the application of eco-friendly crack-resistant cement-based materials.

2. Experimental Section

2.1. Raw Materials

In this study, the LBMO expansion agent produced by Wuhan Sanyuan Special Building Materials Co., Ltd. (Wuhan, China) was used. Its oxide compositions provided by the supplier are shown in Table 1. To prepare the specimens, cement with a strength grade of 42.5, Type II fly ash (FA) and S95 blast furnace slag (BFS) produced by Yushuzhuang Component Factory (Shanghai, China) were selected. River sand with a fineness modulus of 2.35 and crushed gravel with a maximum size of 20 mm and a crushed index of 4.8% were used as fine and coarse aggregates, respectively. Polycarboxylate superplasticizer with a water reduction rate of about 25% was used as the water-reducing agent. The specific surface areas of FA, BFS and cement in this study were 387 m²/kg, 421 m²/kg and 391 m²/kg, respectively.

Table 1. Mass percentage content of each component of the lightly burned magnesia expansion agent (%).

Types	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	MnO	Fe ₂ O ₃
LBMO	79.12	0.44	4.36	0.13	0.11	0.07	0.27	10.80	0.49	4.26
Cement	0	5.47	20.86	0	2.66	0	0.48	62.23	0	3.94
BFS	0	14.74	34.06	0	0.23	0	3.51	35.93	0.74	0.83
FA	0	13.99	32.95	0	0	0	0	7.66	0	2.91

2.2. Samples Preparation

To evaluate the influence of LBMO on the rheological properties of fresh cement paste, LBMO was added into the cementitious paste with respect to the total weight of the binder material. In this study, four LBMO contents, including 0%, 3%, 6% and 9%, were used

to prepare the testing samples. Table 2 shows the mixing proportion of the fresh cement paste. To prepare the fresh paste, cement, fly ash, slag powder and magnesia were mixed in proportion, and then the binder material mixture and water were added to a cement slurry mixer and stirred slowly for 120 s. After a pause of 15 s, the mixture continued to be stirred rapidly for 120 s. The slump flow and plastic viscosity of the fresh cement paste were measured. The slump flow test was carried out by a steel cone with a bottom diameter of 60 mm, an upper diameter of 36 mm and a height of 60 mm according to Chinese Standard GB/T8077-2012 [?].

Table 2. The mixing proportion of the fresh cement paste (g).

Group	Cement	Fly Ash	BFS	Magnesia Expansion Agent	Water
MgO-0%	80	60	60	0	76
MgO-3%	78	58	58	6	76
MgO-6%	75	56	56	12	76
MgO-9%	73	55	55	18	76

Table 3 shows the mixing proportion of concrete with a strength grade of 30 MPa and 50 MPa (C30 and C50) according to Chinese Standard. The cement ratio to fly ash or granulated blast furnace slag was 1:1.33. Water–binder ratios in this study were 0.44 and 0.33, respectively. The LBMO content ranged from 0% to 9% by the total weight of the binder materials.

Table 3. The mix ratio of C30 and C50 concrete (kg/m³).

Group	Cement	Fly Ash	BFS	MgO	Total Binder Material	Water	Sand	Coarse Aggregate
MgO-C30-0	148	111	111	0	370	163	833	1017
MgO-C30-3	143.56	107.67	107.67	11.1	370	163	833	1017
MgO-C30-6	139.12	104.34	104.34	22.2	370	163	833	1017
MgO-C30-9	134.68	101.01	101.01	33.3	370	163	833	1017
MgO-C50-0	194	145.5	145.5	0	485	155	673	1098
MgO-C50-3	188.18	141.14	141.14	14.55	485	155	673	1098
MgO-C50-6	182.36	136.77	136.77	29.10	485	155	673	1098
MgO-C50-9	176.54	132.41	132.41	43.65	485	155	673	1098

The process of concrete mixing is shown as follows:

Firstly, cement, sand and coarse aggregate were added to the concrete mixer and stirred at a low speed of 60 rpm for 1 min, and then water was added and stirred for another 2 min. Finally, all the fresh mixture was poured into oiled molds to form testing specimens.

The specimens with a size of 100 mm × 100 mm × 100 mm were fabricated to measure the compressive strength of concrete. The specimens with a size of 100 mm × 100 mm × 400 mm were used to measure the free expansion value rate of concrete. The molding specimens were sealed by plastic sheets for 2 days, cured at room temperature (20 ± 2 °C) and then demolded. All demolded specimens were cured in a standard curing room with a temperature of 20 ± 2 °C and relative humidity above 95% for 26 days. Experimental space with a constant temperature of 20 ± 2 °C and relative humidity of (60 ± 5)% was provided for the measurement of the compressive and the free expansion value. The microstructure and crystal types of specimens were determined by scanning electron microscopy. Before the experiments, the samples were dried in a vacuum drying oven at a temperature of 60 °C. The dried samples were then sprayed with gold in a vacuum environment.

2.3. Measurement

An RST-CC rheometer manufactured by Brookfield Company (Toronto, ON, Canada) was used to measure the rheological performance parameters (plastic viscosity and shear stress) of the fresh paste with different magnesia expansion agents. In the determination of rheological parameters, the shear rate was increased from 5 rpm to 250 rpm and then

gradually decreased from 250 rpm to 5 rpm. The Bingham model was used to fit the rheological curve to obtain the rheological parameters of the fresh paste [25,26]. Figure 1 shows the process of manufacturing and the rheological measurement of cement paste.

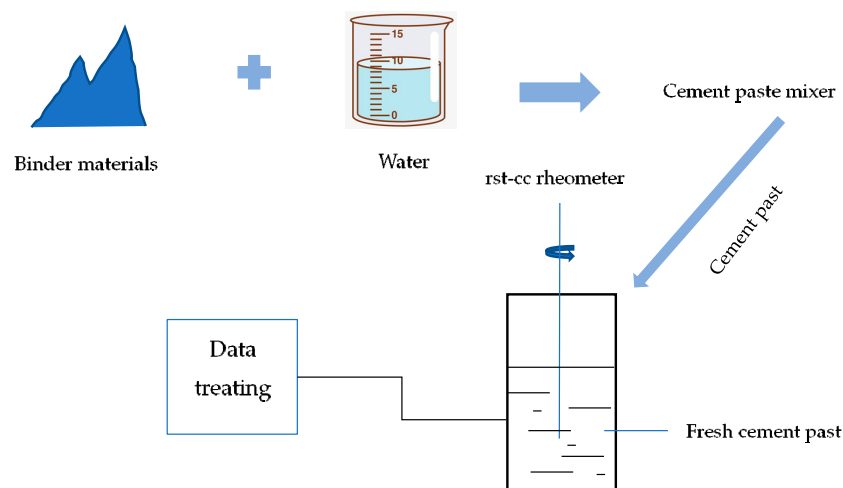


Figure 1. RST-CC rheometer.

Figure 2 shows the schematic diagram of the free expansion value rate of concrete mixed with a magnesium oxide expansion agent. The probe of the dial indicator is fixed at two sections of the test specimen. The shrinkage rod of the dial indicator supports the middle of one end of the rectangular specimen. When the length of the specimen changes, the dial indicator reads out the value of the length change. Through this method, the expansion value is measured.

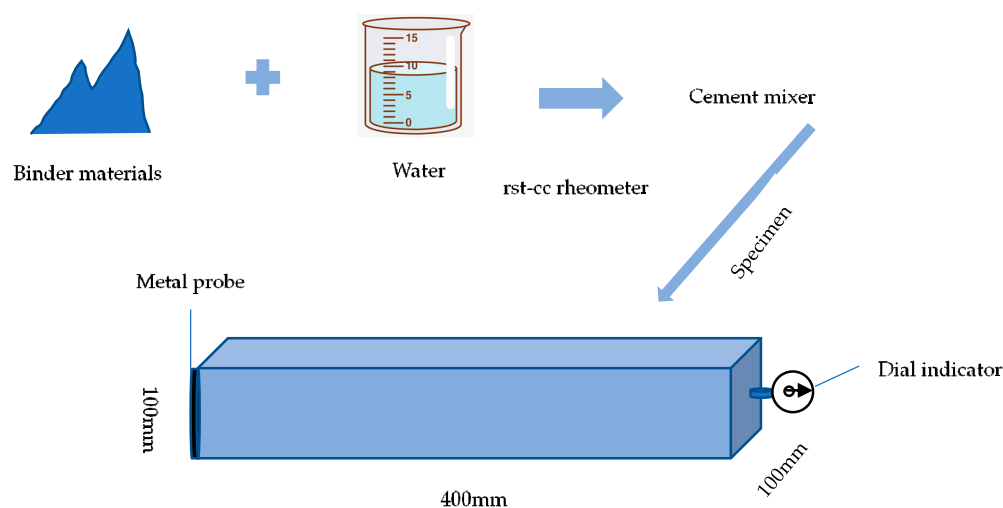


Figure 2. Measurement of the expansion rate of specimens.

Specimens with a size of 100 mm × 100 mm × 100 mm were applied to the experiment of the carbonization experiment. Every specimen was cured for 28 days and then placed in the CCB-70F automatic concrete carbonization test box produced by Tianjin Deste Instrument Technology Co., Ltd. (Tianjin, China), with a 20% CO₂ by the mass ratio of the total mass of gas for 30 days and 60 days, respectively. After curing, all specimens were dried in a vacuum drying oven (Shanghai Yi Heng Instrument Co., LTD DZF-6020, Shanghai, China) at 60 °C for 48 h. When dried in the oven, the sides were sealed with paraffin; however, the two bottom surfaces were not. After carbonization, the carbonation depth was measured according to Chinese Standard GB/T 50082-2009. The microstructure

and crystal types of hydration products were determined by the JSM-6360LV scanning electron microscope (Japan Electron Optics Laboratory, Tokyo, Japan).

3. Results and Discussion

3.1. The Rheological Properties of Fresh Cement Paste

Figure 3 shows the slump flow of the fresh cement paste with different contents of LBMO at different times. It can be observed that the fluidity of all fresh cement pastes with different LBMO content gradually decreases with the extension of time, but the decreasing rates are different. At the initial stage, four groups of fresh cement paste show almost the same initial fluidity, but with time, the paste with higher LBMO exhibits a larger slump flow loss. When the amounts of magnesium oxide are 3% and 6%, the fluidity at a different time gradually decreases, but the decreasing extent is not significant. However, when the content of LBMO reaches 9%, the slump flow of the fresh paste decreases significantly with time. It can be found that at the initial stage, the slump flow is about 240 mm; however, the slump flow drops to 190 mm after 30 min. After 2 h, the fresh paste reduced to about 165 mm. These results show that the LBMO affects the fluidity of the fresh paste, and the influencing degree increases with the increase in the content of the expansion agent. The partial replacement of cement by LBMO may cause a decrease in cement paste fluidity due to the water absorption characteristics of MgO. The reaction equation of magnesium oxide and water is $\text{MgO} + \text{H}_2\text{O} \rightarrow \text{Mg}(\text{OH})_2$. At the initial stage, due to the slow reaction between MgO and water, there is little crystal formation. Therefore, the fluidity loss is caused by the water absorption of MgO [27].

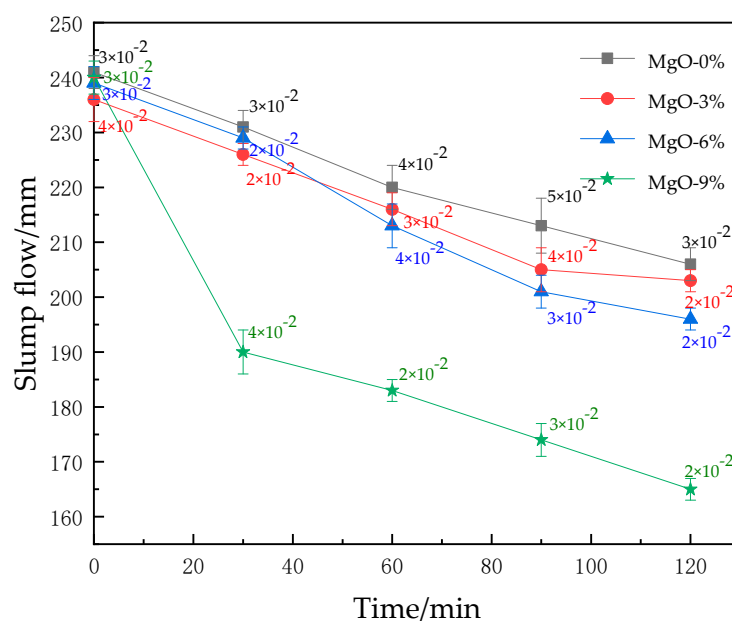


Figure 3. Slump flow of the fresh cement paste.

Figure 4 shows the relationship between the shear stress and shear rate of three groups of fresh paste mixed with LBMO. As presented in Figure 4, the shear stress of the fresh paste mixed with 3% LBMO is higher than that of the reference group without LBMO, while the shear stress of the fresh paste with 6% LBMO is lower than the reference group. As the shear rate increases, the shear stress of the fresh paste gradually increases as well. As presented in Figure 5, when the shear rate is higher than 50 L/s, the shear viscosity of the fresh paste mixed with 3% LBMO is higher than the reference group and higher than that of the fresh paste mixed with 6% magnesium oxide. At the early stage of shear, the paste's plastic viscosity is more prominent due to more free water wrapped in the cement. However, with an increase in rotating speed, the cement's flocculation structure opens, and the free water is released, so its viscosity decreases gradually. Macroscopically, the fresh

paste shows characteristics of shear dilution. It may lead to segregation and bleeding and other undesirable phenomena in the pouring process.

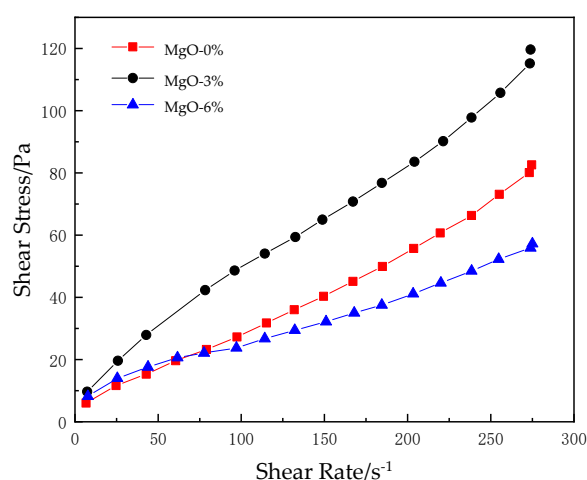


Figure 4. Shear stress as a function of shear rate.

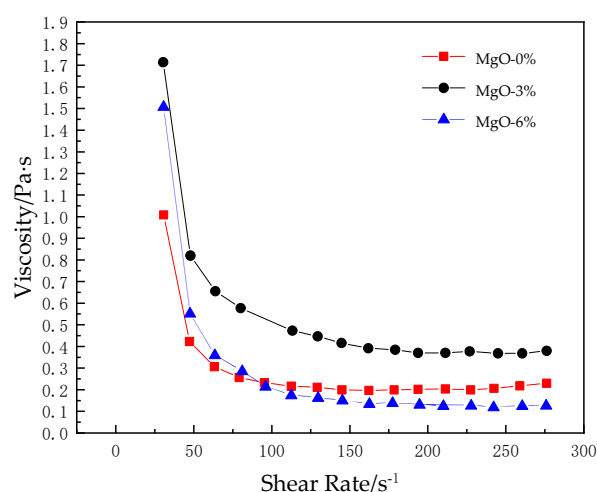


Figure 5. Shear viscosity as a function of shear rate.

According to the Bingham model, the yield stress and plastic viscosity can be obtained by fitting the data measured by a rheometer. Figure 6 shows the relationship between yield stress and the content of magnesium oxide. It can be seen that the paste mixed with a magnesium oxide content of 3% shows the largest yield stress of 0.386 Pa, but the yield stress of the paste mixed with 6% magnesium oxide is lower than the reference group. Figure 7 shows the relationship between the plastic viscosity and magnesium oxide content. It can be found that magnesium oxide's addition can significantly increase the plastic viscosity of the paste, and the paste mixed with 3% MgO shows a higher plastic viscosity than that of samples with 6% MgO. As described in prior research, the shear strength of the slurry can be improved by adding a proper amount of MgO [28]. Therefore, the addition of a certain amount of LBMO improves the plastic viscosity of the fresh paste.

Figure 8 shows the thixotropic ring areas with different magnesia expansion agent contents. The plastic viscosity and shear stress were fitted by Rheo3000 to obtain the area of the thixotropic ring. The thixotropic ring area reflects the size of the paste's thixotropy, and the thixotropy reflects the flocculent structure in the paste. As presented in Figure 8, the thixotropic ring area of the paste mixed with 3% magnesium is larger than the reference group, while the paste mixed with 6% magnesium oxide is smaller than that of the reference group. At a low content, LBMO can increase the contact point of the network

space structure and make the flocculation structure in the cement paste dense and not easy to disintegrate, so the thixotropic area of the paste becomes larger. As described in prior research, when the MgO content is low, the flocculation structure in the slurry is compact [29]. However, the addition of magnesium oxide can destroy the reticular space of the structure's contact points and make the flocculation structure in the cement paste looser and easier to disassemble, so the thixotropy of the paste becomes smaller.

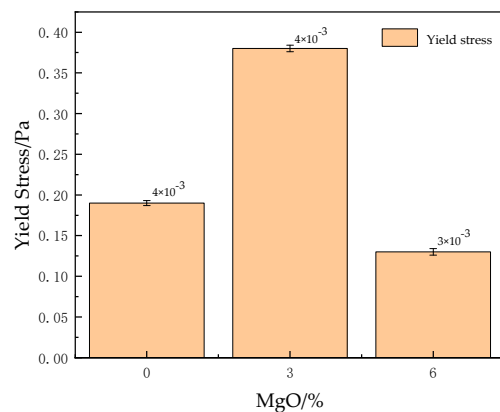


Figure 6. Yield stress of the fresh paste.

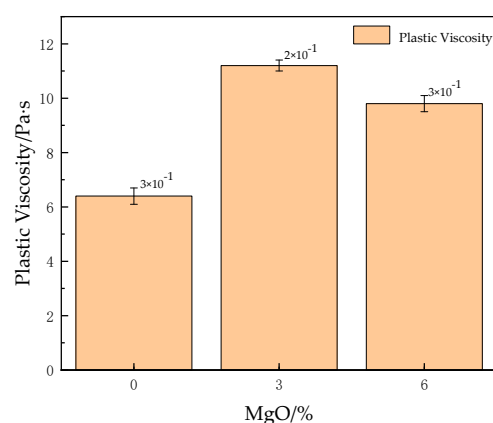


Figure 7. Plastic viscosity of the fresh paste.

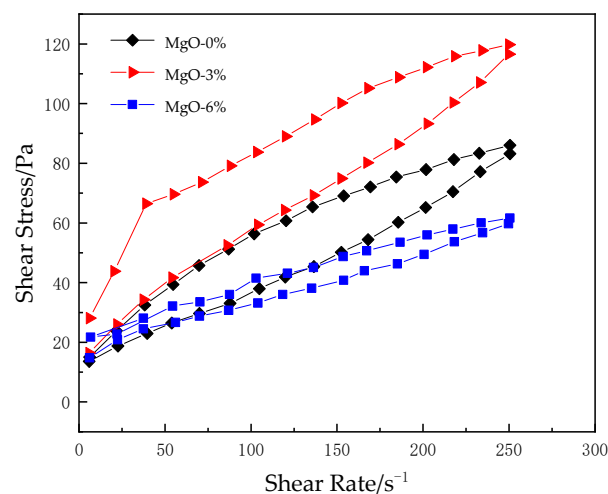


Figure 8. Thixotropic ring areas with different magnesia expansion agent contents.

3.2. The Compressive Strength and Expansion Value Rate of Concrete

In this study, the slump flows of the corresponding fresh cement paste of all groups of the concrete are similar. Table 4 shows the slump flow, the expansion value and content of water-reducing agent of each group. As shown in Table 4, the addition of magnesia expansion agents reduces the workability of the fresh paste. For C30 concrete, with the increase in magnesium oxide content, to ensure that the slump flow and the expansion value of concrete reach the established requirements (the slump flow is above 220 mm, and the expansion value rate is higher than 0.015%), the amount of water-reducing agent gradually increases, and the increment of the water-reducing agent between adjacent groups also increases. Moreover, for C50 concrete, the amount of water-reducing agent changes with the amount of magnesium oxide. It is similar to the C30 group, and the amount of water-reducing agent changing between adjacent groups is more prominent. Each 3% increase in the amount of magnesium oxide is able to increase the amount of water-reducing agents. Especially in the C50-9 group, the amount of water-reducing agent exceeds 3% of the gelling material's total amount. It is no longer possible to obtain sufficient slump flow and expansion value with a reasonable amount of water-reducing agent. The water-reducing agent can easily act on the surface of cement particles other than the magnesium oxide expanding agent and has less contact with magnesium oxide. Therefore, the water-reducing agent demonstrates little effect on the dispersion and fluidity of the fresh cement matrix with MgO [30]. Consequently, the slump flow is decreased, and the plastic viscosity is increased by the addition of the water-reducing agent. In order to obtain a fresh cement matrix with MgO and large fluidity, high content of a water-reducing agent should be added. However, a large amount of water-reducing agent can easily cause segregation and bleeding. Therefore, magnesium oxide in this study should not exceed 9%.

Table 4. The workability and water-reducing agent dosage of C30 and C50 concrete.

Group	Slump Flow (mm)	Expansion Value (mm)	Water-Reducing Agent (%)
MgO-C30-0	245	−10	0
MgO-C30-3	240	0	0.5%
MgO-C30-6 30-6	240	0	1.2%
MgO-C30-9	220	10	2.5%
MgO-C50-0	240	−50	0%
MgO-C50-3	240	−30	0.7%
MgO-C50-6 30-6	235	−40	1.5%
MgO-C50-9	230	20	>3%

Figures 9 and 10 show C30 and C50 concrete's compressive strength at different curing ages. It can be seen that the addition of magnesium oxide at a certain dosage can increase the compressive strength of concrete. Concrete with 3% magnesium oxide shows higher strength than the reference group, but as the magnesium oxide content increases to 9%, concrete with LBMO shows even lower compressive strength than the reference group. After 60 days' curing, the magnesium oxide expansion agent demonstrated an improving effect on the compressive strength of C30 and C50. Moreover, the C30 concrete shows a higher increasing ratio of compressive strength.

It can be seen from the Figure 10 that an appropriate amount of magnesium oxide instead of cement can increase the strength of concrete at various ages. The possible reason may be that the microexpansion caused by the hydration of magnesium oxide can make the concrete denser. Magnesium oxide can absorb an appropriate amount of water, which can reduce the water–binder ratio of the cement paste, thereby increasing the strength of the concrete. As described in prior research, MgO will absorb the water in the slurry, and its dosage should not exceed 6% [31]. However, when the magnesium oxide content is too high, the excessive expansion can cause the hardened concrete structure to burst and

crack. The excessive water absorption will also increase the amount of unreacted particles, leading eventually to the decreased compressive strength of concrete [32,33].

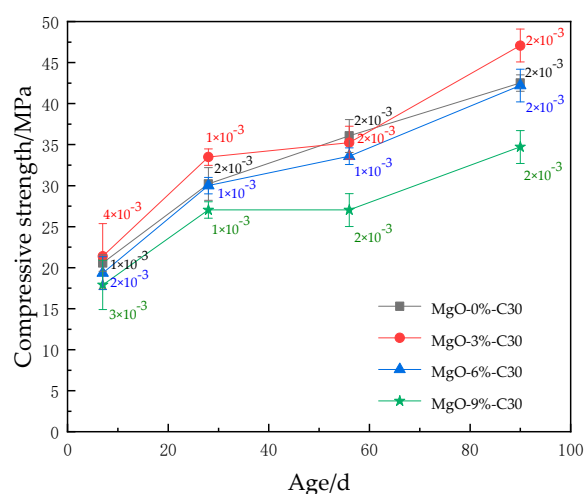


Figure 9. Compressive strength of C30 concrete.

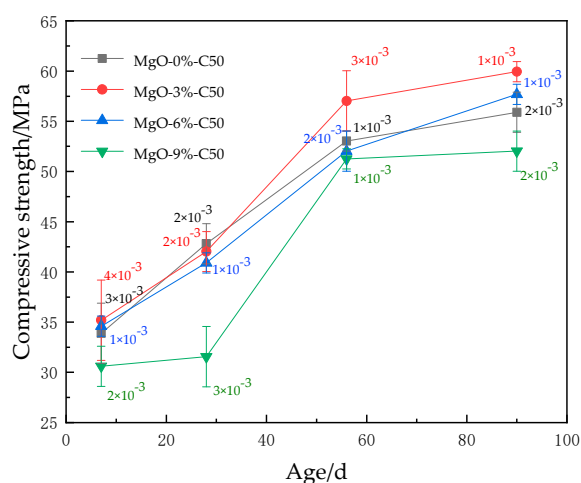


Figure 10. Compressive strength C50 concrete.

Figures 11 and 12 show the relationship between the free expansion value rate. Additionally, the curing time is obtained by measuring the expansion value of concrete at different ages. It is worth noting that magnesium oxide expansion agents have an excellent free expansion rate. With the increase of magnesium oxide content, the free expansion rate of concrete is higher, while the expansion of concrete without magnesium oxide is in a contraction state. Both C30 and C50 concrete show the same trend. For C50 concrete, the free expansion of MgO tends to be stable after 28 days, while the free expansion of C30 concrete tends to be stable after 120 days. Therefore, the expansion of lightly burned magnesia is slightly faster in the early curing stage and tends to be slow in the later curing stage. Compared with the calcium-oxide-based expansion agent, the MgO expansion agent's expansion rate is lower, and the expansion time is longer [34]. Choi et al. [7] found that magnesium oxide's hydration was prolonged before 28 days, and hydration accelerated after 28 days. Compared with concrete without magnesia, the 28 days strength of magnesia concrete is lower, but after 90 days, the magnesia expansion agent concrete exhibits higher strength. This is attributed to the fact that the magnesia hydrates more fully, thus decreasing the porosity of concrete leading eventually to the denser structure of concrete.

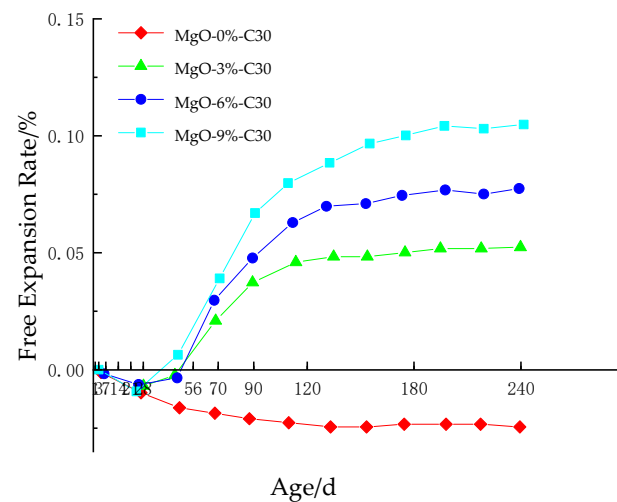


Figure 11. Free expansion value rate of C30 concrete under different magnesia contents.

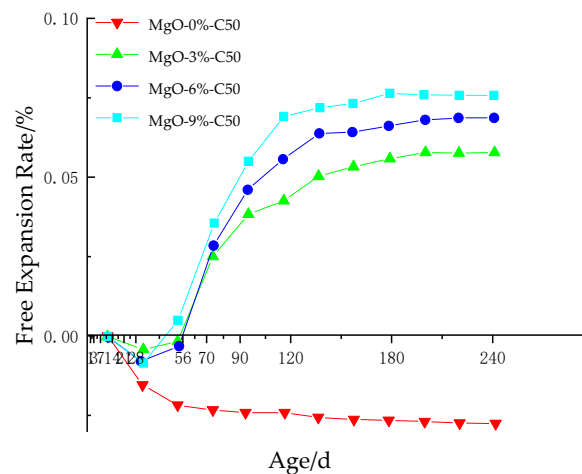


Figure 12. Free expansion rate of C50 concrete with different magnesia contents.

3.3. Carbonation Depth of Concrete

Figure 13 shows the carbonation depth of the concrete. It can be observed from Figure 13 that the carbonation depth of concrete decreases with the addition of lightly burned magnesium oxide. This is attributed to the fact that when the lightly burned magnesium oxide starts to hydrate, the alkalinity of concrete increases, thus leading to improving the carbonation resistance of concrete [33]. Moreover, as shown in Figure 13, the increased curing age and the increased strength grade of concrete lead to a reduction in the carbonation depth due to the denser structure of the cement matrix by the increased strength grade and curing age [35]. Figure 14 show the relationship between the carbonation depth (D) and compressive strength. As illustrated in Figure 14, the carbonation depth increases in quadratic function with the increasing compressive strength. Comparing Figure 14a,b, it can be found that the fitting degree of the concrete with a strength grade of 30 MPa is higher than that with a strength grade of 50 MPa.

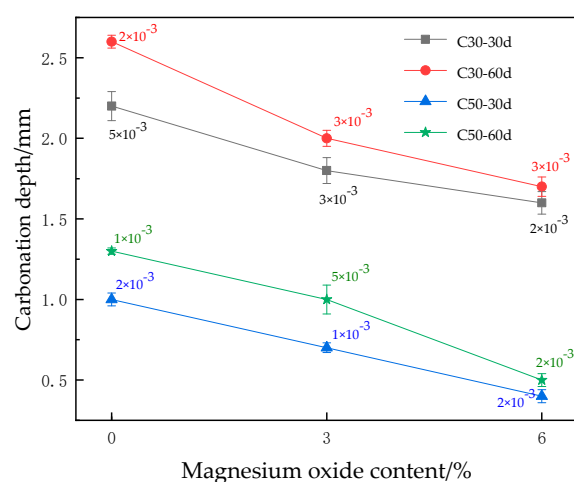


Figure 13. Carbonation depth of concrete.

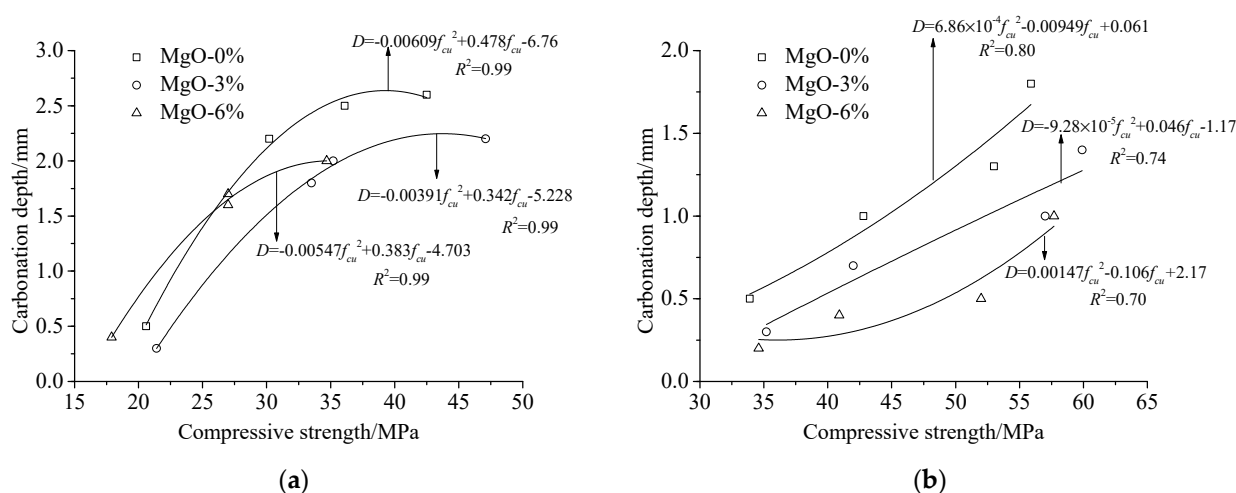


Figure 14. The relationship between carbonation depth (D) and compressive strength (f_{cu}): (a) C30; (b) C50.

Figure 15a,b shows the scanning electron microscope images of C30 concrete after 120 days' curing. As shown in Figure 15a,b, magnesium hydroxide generated by magnesium oxide hydration is distributed in the hardened structure. With the increase in MgO content, more and more magnesium hydroxide is generated. Meanwhile, there is also some unreacted MgO, which can lead to future cracking. It can be seen from Figure 15a,b that part of the magnesium oxide expansion agent is hydrated, and the generated magnesium hydroxide is dispersed in the concrete-hardening structure, resulting in a decrease in the porosity of the concrete structure, improving the compressive strength.

Figure 15c shows the SEM of C30 concrete with 6% MgO content at the age of 120 d. Liu et al. [36] found that with the increase in MgO content, the cement matrixes' total porosity increases because the free expansion value causes cracks in the cement matrix, thereby reducing the strength. The incorporation of a magnesium oxide expansion agent also affects the interface between the cement matrix and aggregate. At the content from 4% to 6%, the mortar and coarse aggregate are firmly bonded, and the interface is dense. As the content of magnesium oxide increases to 8% and 14%, cracks appear at the interface between the cement mortar and coarse aggregate [37]. Finally, the cement mortar appears loose as a whole due to the MgO expansion agent's expansion effect [38].

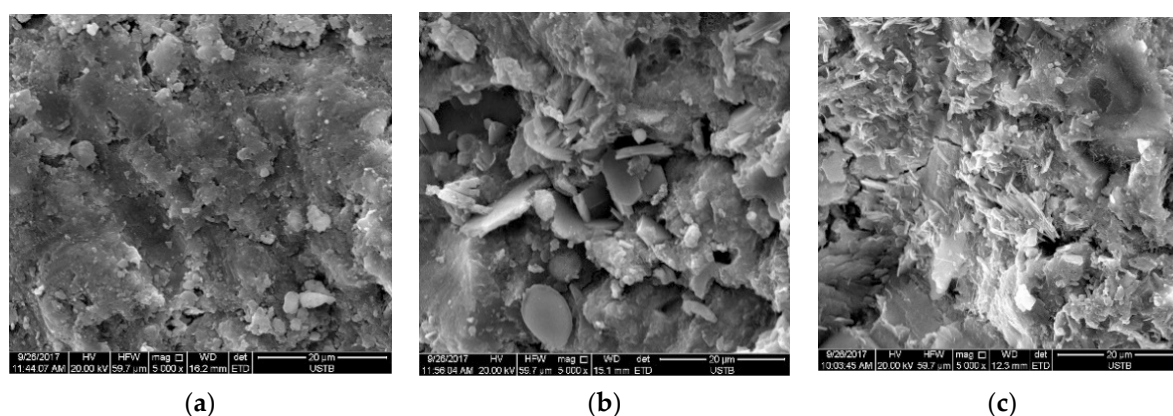


Figure 15. SEM of C30 concrete with different magnesium oxide contents at the age of 120 d. (a) C30-0%-120d; (b) C30-3%-120d; (c) C30-6%-120d.

4. Conclusions

Based on this study, the following conclusions can be obtained:

1. The lightly burned magnesium oxide expansion agent demonstrates a decreased effect on the fluidity and an improved effect on the plastic viscosity of fresh cement paste.
2. When the dosage of the lightly burned magnesium oxide expansion agent is higher than 3%, the compressive strength of concrete is decreased due to the induced deformation of lightly burned magnesium oxide. When the content of the lightly burned magnesium oxide expansion agent is 6%, cracks occur in concrete.
3. The expansion of concrete is increased effectively by lightly burned magnesium oxide. The influence of lightly burned magnesium oxide on the compressive strength and expansion is similar when the strength grades of concrete are C30 and C50.
4. The increased dosage of LBMO, curing age and strength grade lead to improving the carbonization resistance of concrete. As obtained from this study, the relationship between carbonization depth and compressive strength correlates well with the quadratic function.

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