Research Article

Effect of rich-MgO from low-calcium limestone on the calcination and properties of $C_4A_3\$-C_2S$ clinker

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ABSTRACT

As one type of low-calcium cement, $C_4A_3\$-C_2S$ clinker consumes less energy and can utilize rich-MgO low-calcium limestone. The effect of rich-MgO low-calcium limestone on its calcination and properties is studied by means of f-CaO, XRD Rietveld refinement, TG-DTG and Lithofacies analysis. The results show that 3%~5% MgO can promote the absorption of f-CaO and accelerate the formation of highly active monoclinic C_3S and C_4AF in clinker. When the MgO content is 7% and 8%, $C_4A_3\$-C_2S$ clinker calcined at 1380°C has excellent mechanical properties and its soundness is still qualified. This research shows that the rich-MgO low-calcium limestone can replace more than 50% of natural limestone to prepare $C_4A_3\$-C_2S$ clinker in cement industrial production.

1. Introduction

Although the profits of the global cement industry have declined in the past three years, China's cement production is still at a relatively high level, and the demand for cement is still significant. At present, the production of OPC clinker consumes a lot of fossil fuels and high-quality limestone. This leads to large amounts of CO_2 emissions, especially due to the decomposition of high-quality limestone (Wei et al., 2019). In recent years, the price of coal has risen sharply, and the government has strict environmental protection requirements for the cement industry. Currently, low-calcium cement prepared by using low-calcium limestone instead of high-quality limestone is the research direction.

One new type of low-calcium cement called $C_4A_3\$-C_2S$ clinker was studied, which has the potential to utilize rich-MgO low-calcium limestone. It is found that the mineral composition of $C_4A_3\$-C_2S$ clinker is C_3S 30%~50%, C_2S 25%~45%, $C_4A_3\$$ 5%~30%, C_4AF 5%~20% (Huang, 2014). $C_4A_3\$-C_2S$ clinker has a low lime saturation factor. Therefore, it can use low-quality limestone as a raw material, which means that $C_4A_3\$-C_2S$ clinker has lower CO_2 emissions. The high C_2S content makes the strength of $C_4A_3\$-C_2S$ clinker increase steadily with time and hydration heat is low. Previous research found that $C_4A_3\$-C_2S$ clinker has

impermeability and low alkalinity (Li et al., 2018; Quillin, 2001; Sahu and Majling, 1994; Xue et al., 2016). It shows that $C_4A_3\$-C_2S$ clinker has low $Ca(OH)_2$ content in the hydration products and good durability.

In addition, $C_4A_3\$-C_2S$ clinker can be formed at 1320~1380°C, which is lower than OPC, thus allowing the use of low calorific value fuels. The development of $C_4A_3\$-C_2S$ clinker conforms to the concept of green development and is conducive to production. However, the difficulty of calcining this new type of $C_4A_3\$-C_2S$ clinker lies in the coexistence of C_3S and $C_4A_3\$$. Currently, the calcination of $C_4A_3\$-C_2S$ clinker requires the doping of fluorine and sulfur. Moreover, it was found that the doping of fluorine and sulfur was conducive to the solid solution of MgO in $C_4A_3\$-C_2S$ clinker (Chen, 2013; Liu, 2017; Zhang, 2017; Zhao, 2018).

As a common component in limestone, a small content of MgO has a significant effect on the calcination and properties of OPC. It is well known that an appropriate amount of MgO will reduce the emergence temperature and viscosity of the liquid phase, which is beneficial to the conversion from raw materials to clinker. A certain amount of sulfur had a negative impact on the formation of C_3S in clinker, but MgO would weaken the negative impact of sulfur, which was beneficial to the coexistence of C_3S and $C_4A_3\$$(Li et al., 2000; Segata et al., 2019). This

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Table 1

Chemical analysis of raw materials/w%.

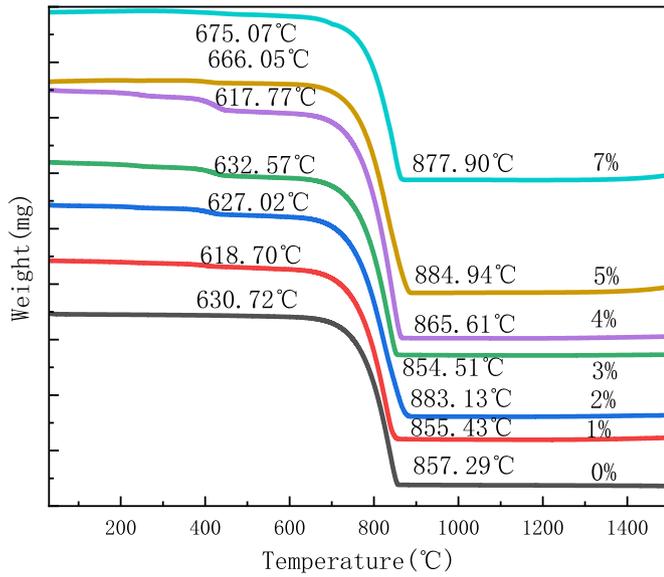| raw material	LOI	CaO	SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	SO$_3$	MgO
natural limestone	34.72	46.10	12.44	3.35	1.96	0.07	0.16
rich-MgO limestone	44.90	40.53	2.47	0.89	0.95	0.08	9.80
desulfurization gypsum	22.07	33.34	1.36	0.41	0.68	40.32	0.48
sandstone	4.84	1.80	56.92	19.30	11.22	0.36	1.07
wet fly ash	6.03	3.64	51.12	26.33	4.61	3.00	0.94
metal ash	3.95	1.92	28.50	4.18	61.00	0.52	1.08

Fig. 1. TG patterns of different MgO content raw materials.
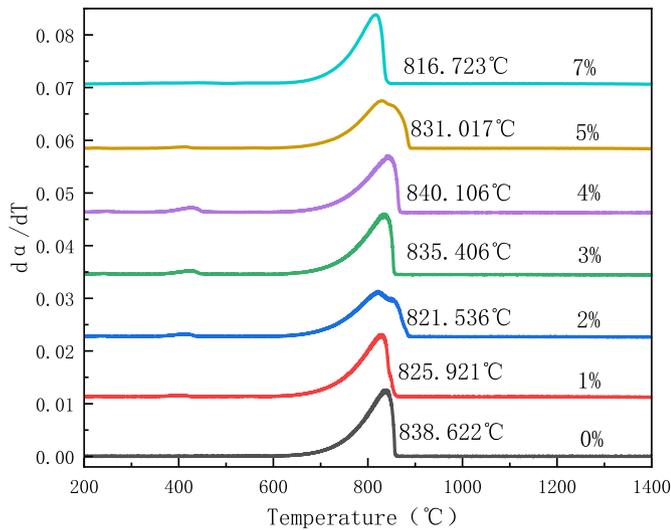

Fig. 2. DTG patterns of specimens with different MgO.
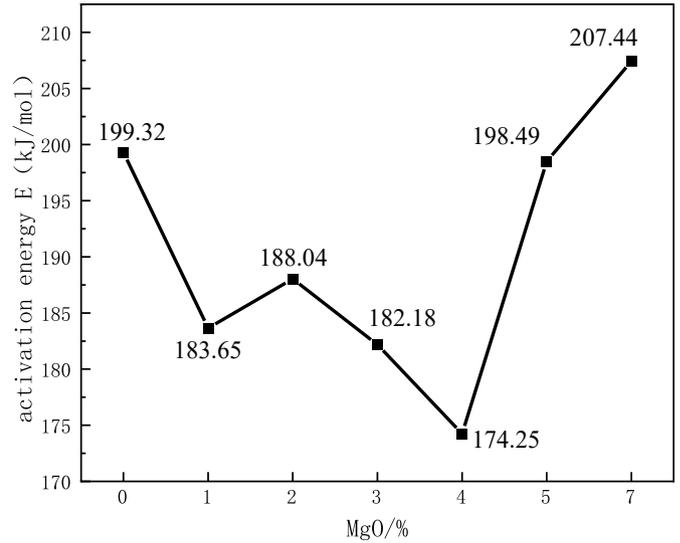

Fig. 3. The activation energy E of the specimens with different MgO.
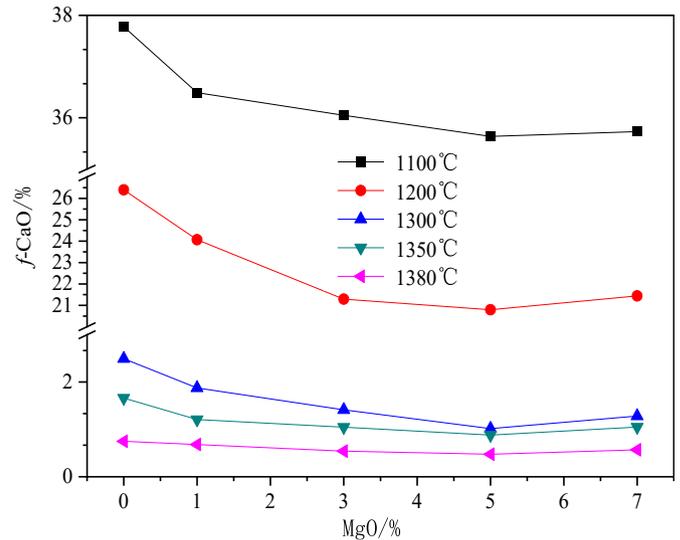

Fig. 4. f-CaO content of clinker with different MgO.

Table 2

TG data of the specimens with different MgO content.

MgO content/%	Weight loss percentage/%	Weight loss range/°C	reaction order n	activation energy E /KJ*mol^{-1}	apparent frequency factor A	correlation coefficient r
0	39.21	630.72~857.29	0.3	199.32	6.89	−0.9969
1	35.66	618.71~855.43	0.4	183.65	6.20	−0.9912
2	37.54	627.02~883.13	0.7	188.04	6.31	−0.9953
3	31.32	632.57~854.51	0.2	182.18	6.07	−0.9980
4	36.12	617.77~865.61	0.3	174.25	5.60	−0.9994
5	38.89	666.05~884.94	0.7	198.49	6.67	−0.9950
7	25.90	675.07~839.80	0.3	207.44	7.49	−0.9984

was due to that MgO had a good mineralization effect on $C_4A_3\$-C_2S$ clinker and promoted the formation of C_3S (jun, 1988; Klemm et al., 1979; Weiqiang et al., 2012). Moreover, the expansion performance of MgO could compensate the shrinkage of hardened paste and alleviate the quick setting of $C_4A_3\$-C_2S$ clinker (Wei, 2012; Zonghan et al., 1998). The addition of MgO was beneficial to improve the hydration activity of clinker (Lu et al., 2012) and decreased its activating energy of the clinker formation (Ali et al., 1994; Huang et al., 2015).

Increasing the fineness of raw materials or adding a certain amount of Cr_2O_3 was beneficial to the solid solution of MgO (Jing et al., 1997; Liu and Li, 1998; song, 2020). But excessive amounts of MgO affected the soundness of cement (Chunfang et al., 2011) and decreased the burnability (Tang, 2015; yu, 2013). Therefore, MgO content in OPC was limited to under 5% from GB 175–2007. It was found that $C_4A_3\$-C_2S$ clinker can accommodate more than 5% MgO. Therefore, it was expected that it would use more low-quality rich-MgO limestone in the $C_4A_3\$-C_2S$ clinker system (Lu et al., 2008; Wang, 2011). In summary, it is of great significance to study the effect of MgO on the $C_4A_3\$-C_2S$ clinker. This can better control MgO content in the $C_4A_3\$-C_2S$ linker and the large-scale utilization of rich-MgO limestone can be realized.

(a) 0%　　　　　　　　(b) 1. 0%
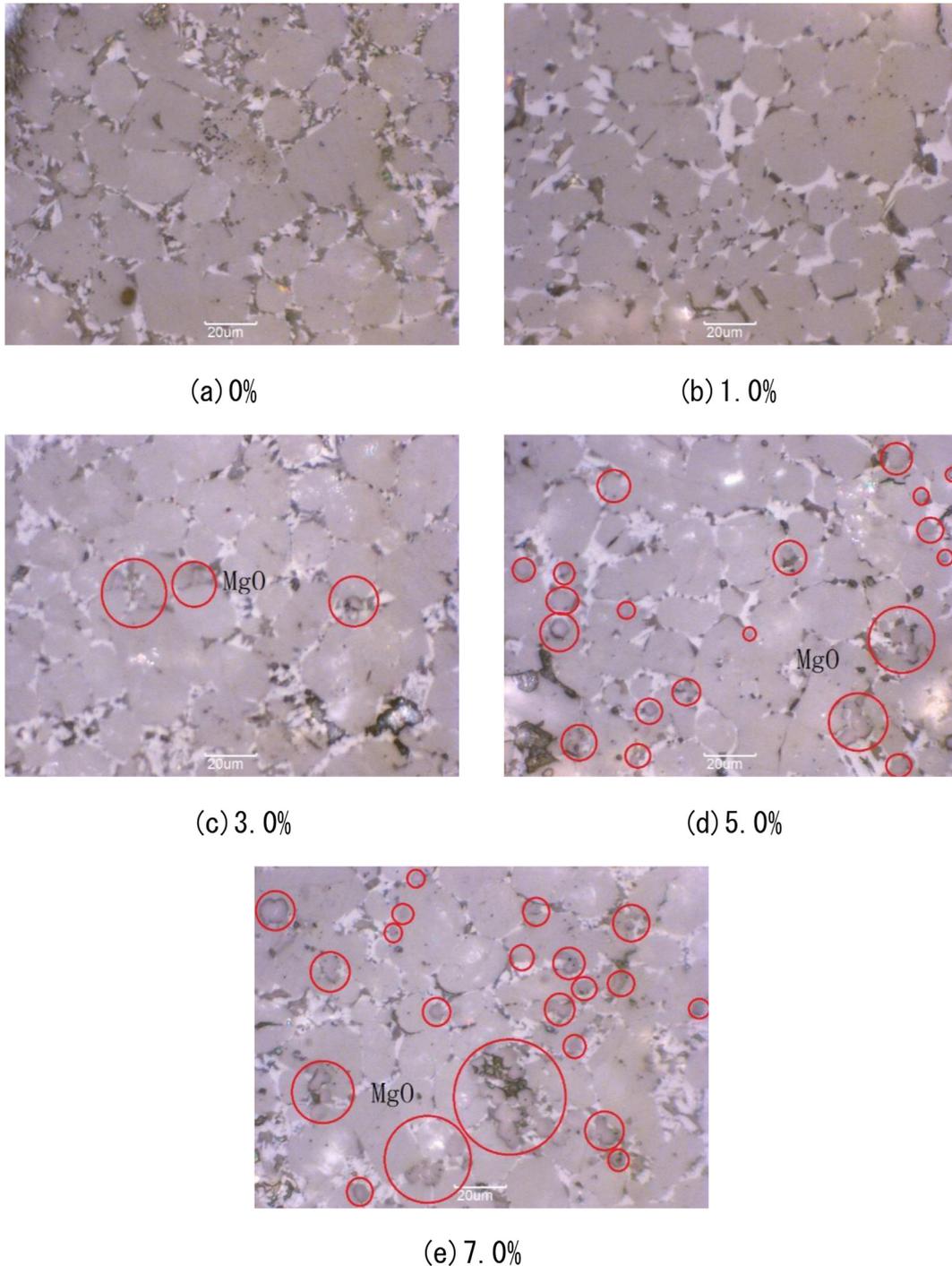
(c) 3. 0%　　　　　　　　(d) 5. 0%

(e) 7. 0%

Fig. 5. Lithofacies photograph (500 ×) of clinker samples calcined at 1380°C after water erosion (20°C, eroded for 9s).

2. Raw material and experiment method

2.1. Raw material

Raw materials nature limestone, rich-MgO limestone, sandstone, wet fly ash, desulfurization gypsum, and metal ash were all purchased from Zibo Shanshui CEMENT Co., Ltd. Analytical reagents CaF_2 was purchased from Sinopharm Chemical Reagent Co., Ltd., China. The chemical compositions of the raw materials used are listed in Table 1.

2.2. Experiment method

The raw materials according to the designed composition were prepared by mixing and grinding in a rotary ball mill for 2 h. Different samples were doped with 0%, 1.0%, 3.0%, 5.0%, 7.0%, 8.0%, 9.0% and 10.0% MgO. The samples were labeled as specimen M0, M1, M3, M5, M7, M8, M9, M10. The mixed raw materials were pressed into the discs of Φ 60 mm × 8 mm, respectively. Then the discs were dried and calcined in a resistance furnace with a heating-up rate of 5°C/min, with different calcining temperatures (1100, 1200, 1300, 1350 and 1380°C). Then the clinker samples were rapidly cooled in the air of 20°C.

2.3. Testing

The mass change of raw materials during calcination was measured by TGA/DSC January 1600 HT thermal analyzer. Mineralogical analyses of clinker were performed using powder X-ray diffraction (Bruker D8-ADVANCE, 2θ range: 5~60°, steps of 0.02°, 2 s/step), and Rietveld refinements were carried out through the TOPAS software. The phase structure and distribution of the clinker mineral were observed with the 4xz metallographic microscope, and the silicate minerals phase and the mesophase in clinker were identified and analyzed by Notic Image 3.2. The content of f-CaO in clinker was determined by ethanol-glycol method. The compressive strength of cement paste was tested by universal testing machine. The soundness of cement clinker specimens was measured by autoclave and ratio length meter. Experimental results of f-CaO testing, strength testing, and soundness testing were the average of the results from six samples.

3. Result and discussion

3.1. TG-DTG analysis

The TG curves of the specimens with different MgO were shown in Fig. 1. It could be seen that the decomposition temperature of $CaCO_3$ was between 620 and 1000°C, but the decomposition interval was different (see Fig. 3) (see Fig. 2).

According to the TG-DTG curves, the kinetic parameters were calculated using the differential-minus derivation method. The kinetic equation was established according to the Arrhenius equation:

$$\frac{d\alpha}{dT} = \frac{A}{\varphi} e^{-\frac{E}{RT}} (1-\alpha)^n$$

Seen from Table 2, the correlation coefficients (*r*) were all within the range of 0.99~1, indicating relatively good fitting results. However, the specimens with different MgO had different activation energy, 4% MgO for the lowest value and 7% MgO for the highest value. The introduction of a small amount of MgO led to the destruction of the surface structure of $CaCO_3$, rather than the formation of intermediate products or high-temperature molten liquid phase. A small amount of MgO could reduce the activation energy of the specimens and save energy to a certain extent.
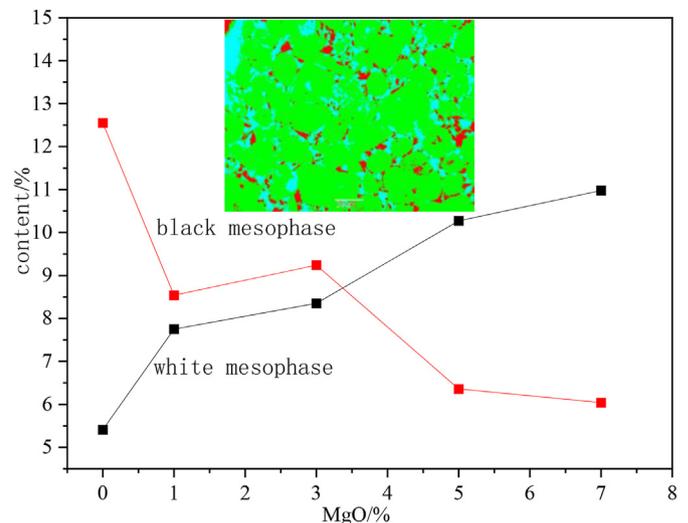

Fig. 6. Effect of MgO on the mesophase content of clinker calcined at 1380°C.

3.2. f-CaO analysis

Fig. 4 showed the determination results of the f-CaO content in the $C_4A_3\$-C_2S$ clinker with MgO. The effect of MgO addition on the f-CaO content was not obvious when the clinker was calcined at 1100°C, indicating that MgO had an insignificant effect on the mineralization of clinker. When the calcination temperature was in the range of 1200~1300°C, the content of f-CaO in the clinker significantly decreased with the increase of MgO, indicating that MgO began to play a role in mineralization and promoted the formation of the clinker. As the calcination temperature was between 1350 and 1380°C, the f-CaO content in the clinker decreased to less than 2.0%.

The f-CaO content in the clinker showed a significant decrease with the increase of MgO addition in the range of under 5.0%, indicating that MgO could promote the formation of the clinker. Differently, the f-CaO content in the clinker increased when MgO reached 7.0%, which may be indicated that an excessive amount of MgO entered the silicate minerals and replaced some of the calcium ion to form f-CaO. Therefore, the appropriate amount of MgO was beneficial to promote the formation of clinker at low temperatures and could save energy to a certain extent.

3.3. Lithofacies analysis

Fig. 5 showed the lithofacies photographs of the clinker samples calcined at 1380°C after water erosion. The color of the silicate phase was light brown, the color of C_3A as black mesophase was black, the color of C_4AF as white mesophase was white, the color of MgO appeared pale pink. It is observed that no MgO was found in the clinker with 1.0% MgO, indicating that MgO was solid-dissolved into the minerals. A small amount of MgO particles with a particle size of 3 μm appeared in the clinker with 3.0% MgO. And more MgO particles appeared in the clinker with 5.0% MgO, and the particle size increased to about 5~7 μm. When the content of MgO increased to 7.0%, the amount of MgO particles further increased and its size increased to over 10 μm, with the appearance of a large MgO mineral nest.

From petrographic analysis, large-sized MgO particles or mineral nests were found, which would affect the soundness of hardened paste. Therefore, it was necessary to test its autoclave expansion ratio to check if its soundness was qualified.
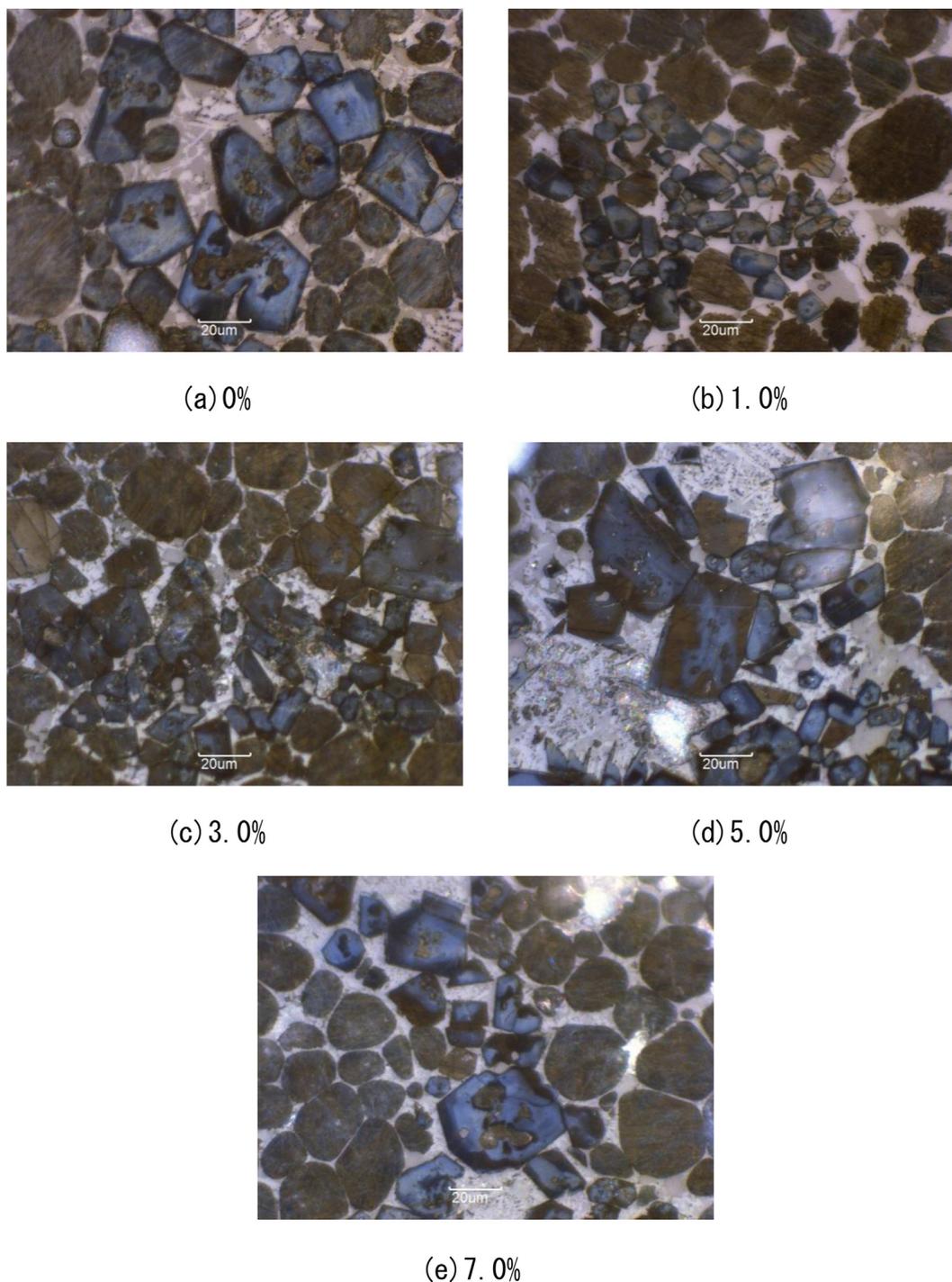
(a) 0%

(b) 1. 0%

(c) 3. 0%

(d) 5. 0%

(e) 7. 0%

Fig. 7. Lithofacies photograph (500 ×) of clinker samples calcined at 1380°C after NH_4Cl erosion (20°C, eroded for 8s).

The content of mesophase was expressed by the area of the mesophase in the clinker. The results were presented in Fig. 6. It could be seen that the content of black mesophase in the samples decreased and the content of white mesophase increased with the content of MgO increased. This may be due to the formation of some solid solution, such as $7CaO·MgO·2Al_2O_3$, $3CaO·MgO·2Al_2O_3$ or $MgO·Al_2O_3$. This increased the ratio of Fe_2O_3 to Al_2O_3 and indirectly increased the content of white mesophase.

The lithofacies photographs of the clinker calcined at 1380°C eroded by 1.0% NH_4Cl were shown in Fig. 7, the bright white area was pores filled with polishing powder of Al_2O_3. As shown in Fig. 7, C_3S in clinker samples without MgO was uniform in size, ranging 30~40 μm. When the MgO content was 1.0%, the size of the C_3S was less than 20 μm. This was mainly because MgO could promote the formation of small-sized C_3S. The size of C_3S fluctuated greatly when the MgO content was in the range of 3.0%~5.0%. The addition of MgO increased the content of white mesophase (C_4AF) in the samples, the viscosity of the white mesophase was lower than that of the black mesophase. Therefore the CaO could react with C_2S to form large-sized C_3S. These lithofacies photographs indicated that some content of MgO could adjust the liquid phase of $C_4A_3\$-C_2S$ clinker, promote the formation of C_3S and facilitate the clinker calcination.
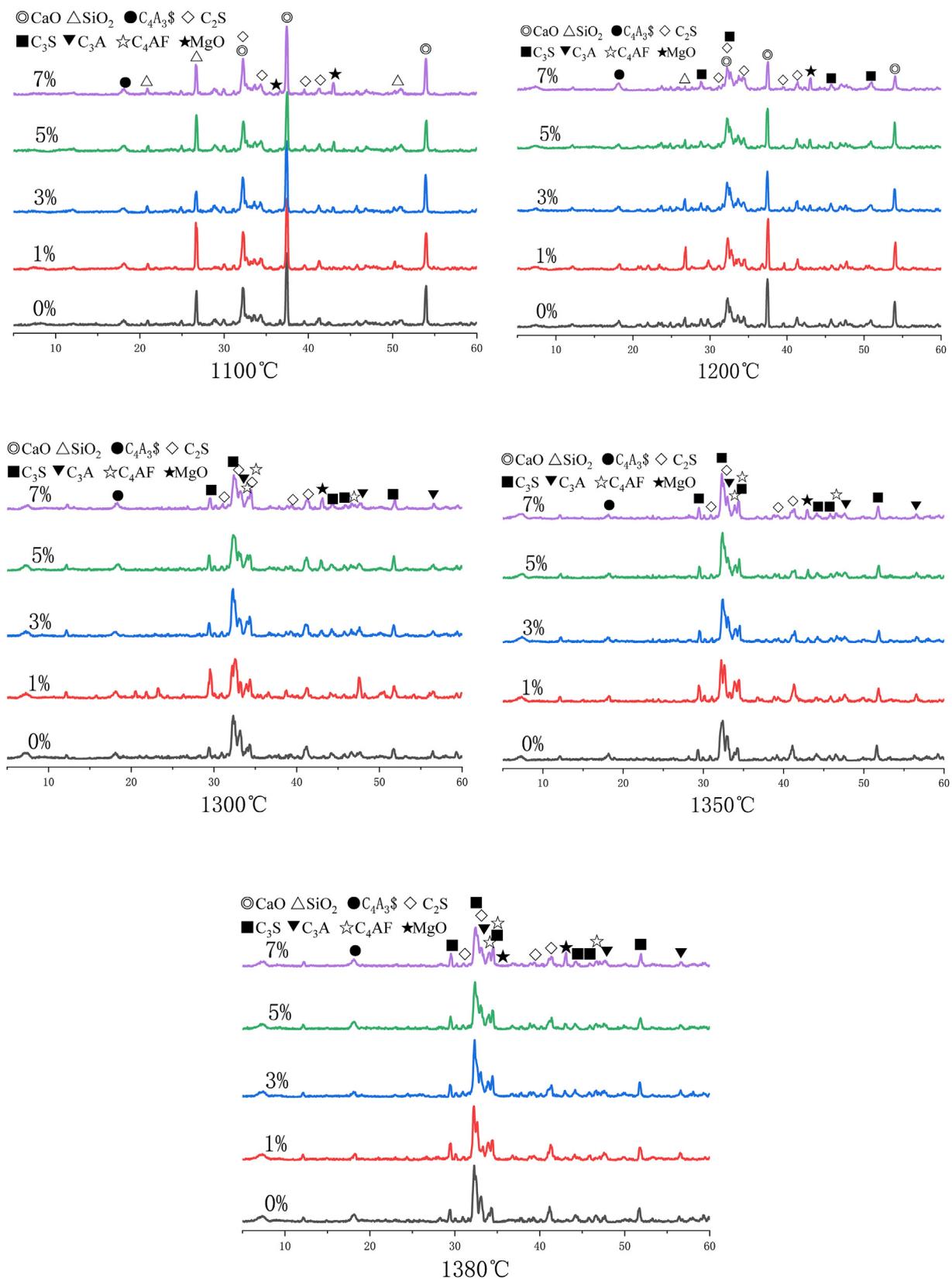
Fig. 8. XRD patterns of specimen with different MgO calcined at various calcination temperatures.

3.4. XRD analysis

Fig. 8 showed the XRD pattern of the MgO-containing clinker. It could be seen that a small amount of C_2S was formed in clinker calcined at 1100°C. The intensity of characteristic diffraction peaks of CaO decreased with the increase of MgO in the clinker calcined at 1200°C, indicating that MgO promoted the absorption of f-CaO and could save energy of clinker calcination with low temperature. When the calcination temperature was in the range of 1300~1380°C, the characteristic diffraction peaks of CaO and SiO_2 in the sample almost disappeared, and C_2S, C_3S, mesophase and $C_4A_3\$$ all had obvious characteristic diffraction peaks. Whereas, it was difficult to distinguish the influence of MgO on the mesophase formation due to its low content.

Fig. 9 showed the XRD pattern partial window of clinker calcined at 1380°C. As shown in Fig. 9, the characteristic diffraction peaks of C_3S and C_2S shifted to the right, indicating that their interplanar crystal spacing decreased. In the clinker, some MgO would form the solid solution, and excess MgO would exist in the form of f-MgO (Bohá et al., 2022; Iwao, Tao et al., 2018; Xu et al., 1995; Yun-Xuan, 2019; Zhao et al., 2021). F. Locher and Iwao Maki found that the Mg^{2+} in Alite only replaced Ca^{2+}, leading to changes in the crystalline form of C_3S. In other words, the smaller Mg^{2+} (72 p.m.) replaced the larger Ca^{2+} (100 p.m.) in the silicate mineral (Weiwei et al., 2007).

3.5. Rietveld refinement analysis

XRD data was performed by the Rietveld whole-pattern fitting method and the content of silicate minerals with different crystal forms in the clinker were shown in Table 3, in which the Rwp was less than 15, indicating that the Rietveld refinement results were good (Mccusker et al., 1999; Yun-Xuan and Hong-Mei, 2018). As shown in Table 3, the
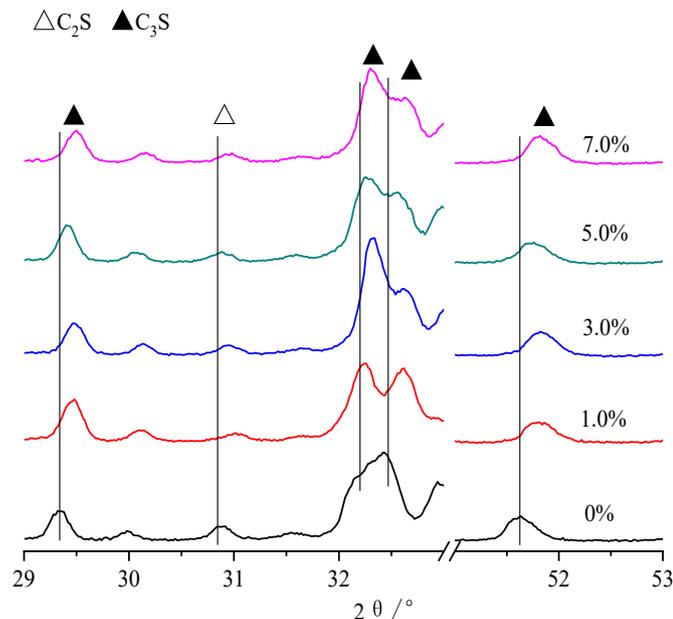
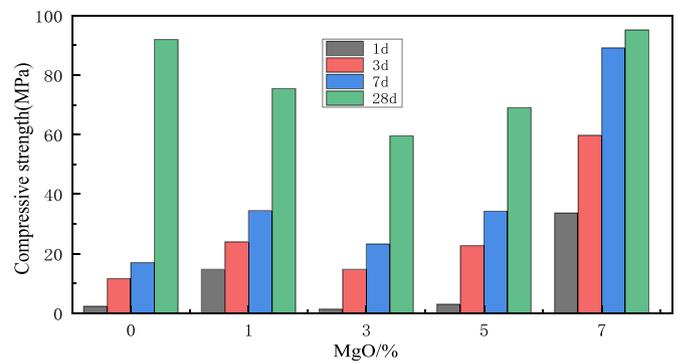**Fig. 9.** XRD pattern partial window of clinker samples calcined at 1380°C.

Fig. 10. Compressive strength of specimen with different MgO content (0~7%).
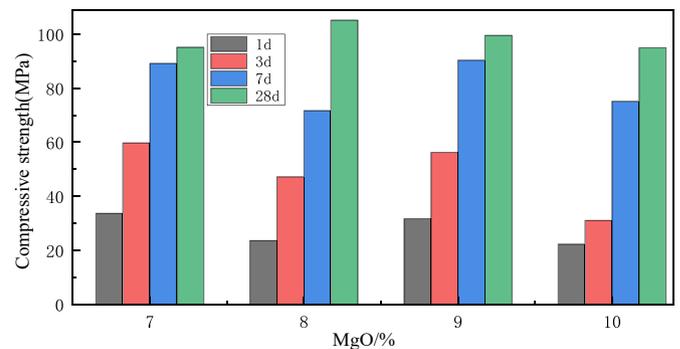
Fig. 11. Compression strength of specimen with different MgO content (7~10%).

Table 4
Autoclave expansion ratio/%.

Sample	Initial reading/ mm	After boiling/ mm	After autoclave/ mm	Boiling expansion ratio/%	Autoclave expansion ratio/%
M7	4.676	4.704	4.778	0.011	0.041
M8	4.021	4.073	4.174	0.021	0.061
M9	3.458	3.531	outrange	0.029	outrange
M10	2.702	2.831	outrange	0.052	outrange

total content of C_2S and C_3S in the clinker calcined at 1380°C remained stable with the increase of MgO content, but the crystal morphology of the silicate minerals changed significantly. In detail, the crystal form of C_3S in reference was mainly $M-C_3S$, with a small amount of T_3-C_3S. The crystal form of the C_2S in reference was mainly β-C_2S, with a small amount of α′low-C_2S.

With the content of MgO increased, the results were: (*i*) the content of M-C_3S in the clinker samples increased, (*ii*)the content of T_1-C_3S and T_3-C_3S decreased, meanwhile, (*iii*)the content of β-C_2S increased, and (*iv*)the content of α′low-C_2S decreased. The above results indicated that MgO could stabilize M-C_3S and β-C_2S in clinker. A small amount of MgO improved the activity of $C_4A_3\$-C_2S$ clinker, which was beneficial to its early strength.

Table 3
Content of silicate minerals of clinker calcined at 1380°C/%.

MgO/%	M-C_3S	T_1-C_3S	T_3-C_3S	$\sum C_3S$	α′$_{low}$ -C_2S	β-C_2S	$\sum C_2S$	R_{wp}
0	26.96	4.12	6.21	37.29	17.37	20.19	37.56	10.978
1.0	28.85	3.63	3.77	36.25	14.26	22.99	37.25	9.612
3.0	30.68	2.19	2.58	35.45	12.82	25.21	38.03	10.150
5.0	33.06	0.91	2.19	36.16	5.34	32.28	37.62	7.756
7.0	34.41	0	1.86	36.27	4.80	33.65	38.45	12.616

Fig. 12. Photos of specimen M7, M8 and M9 after autoclave curing.

3.6. Compression strength

Fig. 10 and Fig. 11 showed the compressive strength of specimens with different MgO content (0%, 1.0%, 3.0%, 5.0%, 7.0%, 8.0%, 9.0% and 10.0%). As shown in these two figures, the early compressive strength of M1 increased obviously, but its 28 d compressive strength decreased. Both M3 and M5 showed a certain increase in early compressive strength compared with the control group, but their 28 d strength was lower than that of M1. Compared with the sample with low MgO content, the M7 sample performed well in early strength, and its 28 d compressive strength was also higher. Compared with M7, the 28 d compressive strength of M8, M9 and M10 increased slightly, but their early compressive strength also decreased slightly. High MgO content often means poor soundness. Therefore, it is necessary to check the soundness.

3.7. Soundness analysis

According to the requirement of GB T1346-2011, the MgO content should not exceed 6% in clinker, and the soundness of groups M7 to M10 should be tested.

Table 4 showed the autoclave expansion ratio of group M7 to M10, and the photos after autoclave curing were presented in Fig. 12. As shown in Table 4 and Fig. 12, the soundness of M7 and M8 still met the standard, confirming the ability of $C_4A_3\$-C_2S$ clinker to dissolve more MgO. This laid the theoretical foundation for the use of low-grade high-MgO limestone in $C_4A_3\$-C_2S$ clinker production. As for M9 and M10, their soundness was not qualified. In particular, sample M10 was pulverized, confirming its extremely poor soundness. The result showed that the soundness of $C_4A_3\$-C_2S$ clinker with MgO content less than 8% is qualified.

Under the condition of good working performance and soundness, $C_4A_3\$-C_2S$ clinker can indeed accommodate more MgO, so rich-MgO low-calcium limestone can be used to produce $C_4A_3\$-C_2S$ clinker.

4. Conclusion

Appropriate content of MgO could decrease the activation energy of clinker, 1%~5% MgO could promote the absorption of f-CaO, which was conducive to the formation of silicate minerals. Appropriate MgO distorted the mineral lattice, changed the crystalline shape of silicate minerals, stabilized M-C_3S and β-C_2S and improved the hydration activity of clinker. The compressive strength of clinker with 7%~8% MgO content had the best performance under the condition of qualified soundness. The addition of MgO promoted the calcination of $C_4A_3\$-C_2S$ clinker and reduced the calcination temperature. That was beneficial to reduce CO_2 emissions and save energy. In conclusion, the appropriate amount of MgO improved the early strength of $C_4A_3\$-C_2S$ clinker and decreased its calcination temperature, which provided the basis for the application of rich-MgO low-calcium limestone.

The research of $C_4A_3\$-C_2S$ clinker has a broad green development prospect. In the future industrial production of $C_4A_3\$-C_2S$ clinker, the use of fossil fuels is saved, and more than 50% of rich-MgO low-calcium limestone can be used to replace natural limestone, which reduces the production cost.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

CRediT authorship contribution statement

Yiping Qiu: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Chengming Li:** Methodology, Data curation. **Yiqun Zhang:** Methodology, Data curation. **Yuan Feng:** Methodology, Data curation. **Sergei Leonovich:** Methodology, Data curation. **Piqi Zhao:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Shoude Wang:** Writing – review & editing, Supervision, Conceptualization.

Acknowledgements

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