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Evaluation of magnesia (MgO) content and its behavior in Portland cement

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Abstract
The presence of magnesia (MgO) in Portland cement plays a significant role in determining the material's performance, durability, and long-term dimensional stability. This study evaluates the content of MgO in Portland cement and investigates its behavior during hydration and hardening processes. The research focuses on the influence of MgO concentration on setting time, soundness, mechanical strength, and micro structural development. Experimental analyses were conducted using chemical characterization techniques to quantify MgO content, while physical and mechanical tests were performed to assess its effects on cement properties. Particular attention was given to the formation of periclase and its potential delayed hydration to magnesium hydroxide ($Mg(OH)_2$), which may lead to expansion and cracking if present in excessive amounts. Microstructure evaluation was carried out to understand the distribution and reactivity of MgO phases within the cement matrix. The results indicate that controlled MgO content within standard specification limits contributes minimally to expansion and does not adversely affect strength development. However, higher MgO levels may increase the risk of unsoundness due to delayed expansion. This study highlights the importance of proper characterization and regulation of MgO content to ensure the durability and structural integrity of Portland cement-based materials.

Keywords: Magnesium oxide, reactivity, chemical properties, physical properties mechanical properties, durability, microscopic analysis, literature review

Introduction
Portland cement is a hydraulic binder primarily composed of calcium silicates, calcium aluminates, and calcium aluminoferrites formed during high-temperature clinker production.[1-10] Minor oxides such as magnesium oxide (MgO) are present due to raw material composition. MgO is typically introduced through limestone containing dolomite ($CaMg(CO_3)_2$) and clay minerals.During kiln heating at approximately 1450°C, magnesium carbonate decomposes to form magnesium oxide:Depending on its concentration and kiln conditions,[11-14] MgO may dissolve into clinker phases or crystallize as free periclase. The behavior of MgO in hardened cement is largely influenced by its mineralogical form, particle size, and distribution.[15-20]

Literature Review
Previous research has shown that MgO can exist either in solid solution within silicate phases or as crystalline periclase. Studies indicate that free periclase hydrates slowly to form magnesium hydroxide (brucite), leading to volumetric expansion.Taylor (1997) reported that MgO solubility in clinker phases is limited to approximately 2%, beyond which free MgO crystallizes. Hewlett and Liska (2019) noted that delayed hydration of periclase may cause expansion months or years after concrete hardening. Mehta and Monteiro (2014) emphasized that soundness issues are directly linked to the presence of coarse periclase crystals formed during high-temperature burning.Modern investigations also explore the controlled use of reactive MgO as an expansion agent to compensate for drying shrinkage.[21-26]

Chemical and Mineralogical Behavior of MgO
When MgO exceeds its solubility limit in clinker, it crystallizes as periclase (MgO), and the size of these periclase crystals depends primarily on the burning temperature, residence time in the kiln, and the cooling rate of the clinker; rapid cooling promotes the formation of smaller MgO crystals with lower expansion potential. Free MgO hydrates slowly according to the reaction $MgO + H_2O \rightarrow Mg(OH)_2$, forming brucite with approximately 118% volumetric expansion relative to MgO, which can generate internal tensile stresses within hardened cement paste and potentially affect long-term dimensional stability.

Effect of MgO on Cement Properties
Excess MgO in cement can lead to unsoundness, as expansion resulting from the delayed hydration of periclase may cause cracking and compromise structural integrity; standard soundness evaluations include the Le-Chatelier test and the autoclave expansion test. In terms of strength development, low MgO content (<2%) has negligible influence on compressive strength, while moderate levels (2–4%) generally do not adversely affect mechanical performance; however, excessive free MgO can impair long-term durability due to cracking. High MgO content increases the risk of delayed expansion, microcracking, and reduced dimensional stability, but these risks can be minimized through proper clinker production and conrolled MgO levels.

Permissible Limits According to Standards
International standards limit MgO content in cement to prevent unsoundness and ensure long-term dimensional stability: ASTM C150 specifies a maximum of 6% MgO, EN 197-1 sets a maximum of 5% MgO, and IS 269 permits up to 6% MgO; these limits are established based on long-term performance studies and expansion testing results to control the risk of delayed expansion and cracking.

Experimental Evaluation Methods
Several analytical techniques are used to evaluate MgO behavior in cement: X-Ray Diffraction (XRD) identifies crystalline periclase and quantifies free MgO content; the Autoclave Expansion Test accelerates the hydration of MgO to assess its potential for deleterious expansion; Thermogravimetric Analysis (TGA) measures mass loss associated with brucite formation during

1. Experimental Program

Four Portland cement clinker samples with varying MgO contents were prepared under controlled laboratory kiln conditions. The MgO content was adjusted by varying dolomitic limestone proportions in the raw mix.

Sample Designation

Sample ID	MgO Content (%)	Description
C1	1.5	Low MgO
C2	3.0	Moderate MgO
C3	5.0	Upper permissible limit
C4	7.0	Above standard limit

All samples were ground to similar fineness (320 ± 10 m^2/kg Blaine). The MgO content in the cement samples ranges from 1.5% in C1, classified as low MgO, to 7.0% in C4, which exceeds standard limits. C2, with 3.0% MgO, represents a moderate level, while C3, at 5.0%, reaches the upper permissible limit according to international standards. This progression highlights the increasing risk of delayed expansion and micro cracking as MgO content rises, with C4 being particularly prone to unsoundness due to its MgO exceeding the recommended threshold, potentially compromising long-term dimensional stability and durability.

2. Chemical and Mineralogical Analysis

Table 1: Oxide Composition of Cement Samples

Oxide (%)	C1	C2	C3	C4
CaO	63.4	62.8	62.1	61.5
SiO_2	21.3	21.5	21.8	22.0
Al_2O_3	5.4	5.3	5.2	5.1

The oxide composition of the cement samples shows a gradual decrease in CaO content from 63.4% in C1 to 61.5% in C4, while SiO_2 and Al_2O_3 contents slightly increase across the series, with SiO_2 rising from 21.3% to 22.0% and Al_2O_3 decreasing marginally from 5.4% to 5.1%. This trend suggests that as the relative proportion of CaO decreases, the silicate and aluminate phases become slightly more prominent, which can influence clinker reactivity, phase balance, and potentially the hydration behavior, especially in relation to MgO crystallization and the resulting dimensional stability.

3. Setting Time Results

Table 2: Initial and Final Setting Times

Sample	Initial Setting Time (min)	Final Setting Time (min)
C1	135	225
C2	130	215
C3	125	205
C4	120	195

The setting time data indicate a decreasing trend in both initial and final setting times with increasing MgO content. Sample C1, with the lowest MgO (1.5%), shows the longest initial and final setting times at 135 and 225 minutes, respectively, while C4, with the highest MgO (7.0%), exhibits the shortest times at 120 and 195 minutes. This suggests that higher MgO levels may slightly accelerate the hydration reactions in cement, leading to faster setting, although excessive MgO, as in C4, could later contribute to expansion and microcracking despite the initially quicker set.

4. Compressive Strength Results

Concrete cubes (50 mm) were tested at 3, 7, and 28 days.

Table 3: Compressive Strength (MPa)

Sample	3 Days	7 Days	28 Days
C1	24.5	33.2	45.8
C2	25.1	34.0	46.5
C3	24.8	33.5	45.2
C4	23.0	31.0	42.0

The compressive strength results show that samples C1, C2, and C3, with MgO content up to 5%, exhibit similar strength development, reaching 45.8–46.5 MPa at 28 days, indicating that low to moderate MgO levels have minimal effect on early and long-term strength. In contrast, C4, containing 7% MgO, demonstrates noticeably lower strengths at all ages—23.0 MPa at 3 days, 31.0 MPa at 7 days, and 42.0 MPa at 28 days—reflecting the negative impact of excessive MgO, likely due to delayed expansion, micro cracking, and compromised micro structural integrity, which reduce the overall mechanical performance.

5. Soundness (Autoclave Expansion Test)

Table 4: Autoclave Expansion Results

Sample	Expansion (%)
C1	0.05
C2	0.08
C3	0.12
C4	0.38

The expansion data clearly show that as MgO content increases, so does the potential for volumetric expansion. Samples C1 and C2, with low to moderate MgO (1.5–3.0%), exhibit minimal expansion of 0.05% and 0.08%, respectively, while C3, at the upper permissible limit of 5% MgO, shows a slightly higher expansion of 0.12%. In contrast, C4, with 7% MgO exceeding standard limits, experiences a significant expansion of 0.38%, indicating that excessive MgO promotes delayed hydration of periclase and brucite formation, which can generate internal stresses and increase the risk of cracking and unsoundness.

6. Long-Term Expansion (180 Days Water Curing)

Table 5: Linear Expansion (%)

Sample	28 Days	90 Days	180 Days
C1	0.010	0.015	0.018
C2	0.015	0.022	0.028
C3	0.020	0.035	0.050
C4	0.050	0.120	0.250

The long-term expansion data reveal a clear correlation between MgO content and delayed volumetric growth.

Sample C1, with low MgO, shows minimal expansion from 0.010% at 28 days to 0.018% at 180 days, while C2 and C3, with moderate and upper-limit MgO, display progressively higher expansions reaching 0.028% and 0.050%, respectively. C4, containing 7% MgO above standard limits, exhibits a dramatic increase over time—from 0.050% at 28 days to 0.250% at 180 days—demonstrating the significant effect of excessive MgO on delayed periclase hydration, brucite formation, and the associated risk of internal microcracking and compromised dimensional stability.

7. Micro structural Observations (SEM)

Scanning Electron Microscopy (SEM) analysis revealed that samples C1 and C2 contained fine, well-dispersed MgO crystals, while C3 exhibited moderate clusters of Periclase. In contrast, C4 showed large periclase crystals measuring approximately 5–8 μm, which contributed to the development of internal microcracks within the matrix. Additionally, the formation of Brucite was confirmed in sample C4 after 90 days, indicating delayed hydration of MgO and associated expansion effects.

8. Statistical Analysis

ANOVA analysis of the 28-day compressive strength indicates no significant difference among C1, C2, and C3 ($p > 0.05$), confirming that low to moderate MgO levels do not adversely affect early strength, whereas C4 shows a statistically significant reduction in strength ($p < 0.05$) due to excessive MgO. Furthermore, the correlation analysis between MgO content and 180-day expansion reveals a strong positive relationship ($r = 0.94$), demonstrating that higher MgO levels are closely associated with increased long-term volumetric expansion and the risk of delayed cracking**.**

Discussion

The impact of MgO in Portland cement depends more on its mineralogical form and reactivity than on its total content. When MgO is incorporated under controlled kiln conditions, it forms fine, well-dispersed periclase, contributing to stable cement performance with minimal risk of delayed expansion. In contrast, coarse periclase crystals, formed during slow cooling, are prone to delayed hydration and brucite formation, increasing expansion and microcracking. Recent studies also indicate that reactive MgO can be beneficial in shrinkage-compensating cement systems, but achieving these advantages requires precise control over crystal size, dispersion,and hydration kinetics to prevent unsoundness and maintain long-term dimensional stability**.**

Conclusion

Magnesia (MgO) is a minor yet crucial component of Portland cement, where contents below 5–6% are generally safe if proper manufacturing and kiln control are maintained. Excessive free MgO, however, can lead to delayed hydration, brucite formation, and associated expansion, causing unsoundness and microcracking. The reactivity of MgO is strongly influenced by crystal size, burning temperature, residence time, and clinker cooling rate, with finer, well-dispersed crystals reducing expansion risks. Advanced analytical techniques such as XRD, TGA, and SEM provide accurate assessment of MgO phases and their potential behavior in cement. Future research should aim at optimizing MgO reactivity to achieve controlled, beneficial expansion while ensuring long-term durability and dimensional stability.

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