



Modifications in Setting, Strength and Moisture Resistance of Magnesium Oxysulfate by Mixing Polyvinyl Alcohol as an Additive

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ABSTRACT: Magnesium oxysulfate cement (MOS) is a novel kind of binder with numerous advantageous qualities, including good binding capabilities, fire resistance, environmentally friendly etc. There are many benefits of using non-hydraulic MOS instead of traditional Portland cement. MOS saves energy and preserves the environment, which makes it an attractive research area. Its low early strength and poor moisture resistance make large-scale commercial application impractical, despite of its many benefits. In this experiment, Polyvinyl alcohol (PVA) was used as an additive to overcome its drawbacks. The results showed that PVA increased initial as well as final setting time of MOS. PVA also improved compressive strength and water resistance of MOS. Experimental results are supported by FTIR analysis.

KEYWORDS: Compressive strength, Magnesium oxysulfate cement, Polyvinyl alcohol, Water resistance.

INTRODUCTION

Magnesium oxysulfate cement (MOS) was discovered in 1867 by French chemist S. T. Sorel [1]. MOS is an inorganic cementitious material made from the chemical reaction of light-burned magnesium oxide and magnesium sulfate heptahydrate in the presence of water [2]. This exothermic process generates heat, weakening the cement and reducing its moisture resistance, rendering the completed product unsound [3]. To solve this issue, dolomite used as inert filler in the matrix uses. Using a three-body collision mechanism, inert filler absorbs heat instead of taking part in the cementing reaction [4]. MOS is lightweight, resistant to fire, and has good decorative value [5-8]. As a result, it is widely employed in refractory components, external wall insulation boards, and other fields [9].

MOS has garnered interest due to its quick setting, high early strength, and environmental advantages, such as reduced CO₂ emissions during manufacturing [10, 11]. However, MOS's moisture sensitivity and susceptibility to sulfate leaching can compromise long-term performance [12,13]. Improving its endurance while maintaining mechanical qualities is crucial for widespread adoption. Improving the performance of construction materials is crucial to fulfilling the growing demand for long-lasting, high-performing infrastructure. MOS has attracted interest in recent years as an environmentally friendly alternative to typical Portland cement because of its decreased carbon footprint, rapid setting, and good bonding qualities [14,15]. However, MOS still has very high-water sensitivity and poor long-term strength development, which limit its industrial application [16-18]. MOS provides several desired mechanical and technical features, but its widespread application is limited by low water resistance, which causes a considerable reduction in strength when the hardened material is immersed or exposed to moisture over time [19,20]. Zhen Li et al. studied that citric and phosphoric acids enhanced the strength and microstructure of MOS, with citric acid showing superior performance [21]. Qiyan Li et al. demonstrated that incorporating FA or GGBFS with CO₂ curing enhances MOS cement's durability and water resistance while enabling carbon sequestration [22]. Qiao et al. studied that incorporating PBTC and HPMA significantly enhances the mechanical strength and water resistance of MOS. Their chelation with Mg²⁺ modifies the hydration process, promoting the formation of a stable 5·1·7 crystalline phase [23]. Wang et al. investigated that adding up to 15 wt.% of low-cost quartz (Q) significantly enhances both early and later mechanical strength of MOS by improving hydration, phase formation, and pore structure [24]. However, its beneficial effects diminish when the content exceeds 20 wt.%. Polyvinyl alcohol (PVA) is a water-soluble, synthetic polymer known for its excellent film-forming, emulsifying, and adhesive properties. In the context of magnesium oxysulfate, PVA is used to enhance various mechanical and durability characteristics.



EXPERIMENTAL

Materials

The raw materials used in the study included lightly calcined magnesium oxide (MgO), magnesium sulfate heptahydrate, and dolomite powder as an inert filler.

- Magnesium oxide [25]: Commercial grade Magnesia (lightly calcined) was used in the synthesis of MOS. The analysis of Magnesite powder is SiO₂=4.51%, CaO=2.80%, MgO=89.70%, Fe₂O₃=0.12%, Al₂O₃=0.98%, LOI=3.42%.
- Magnesium sulfate [26]: Technical grade magnesium sulfate was used in the formation of MOS. The chemical composition of Epsom salt is MgSO₄ = 96.80%, Fe₂O₃=0.02%, Al₂O₃=0.07%, CaO=1.40%, Acid insoluble = 0.11%, moisture = 0.98%.
- Dolomite [27]: To make MOS, locally available dolomite powder was utilized as an inert filler. The chemical composition of Dolomite is SiO₂ =8.45%, CaO=36.68%, MgO=11.80%, Fe₂O₃=0.73%, Al₂O₃=0.13%, LOI=41.80%, CaCO₃=65.50%, MgCO₃ =24.78% Brightness=89.20%, Whiteness=92.60%.
- PVA: It is collected from Rajasthan scientific works, Jaipur India.

A. Sample Preparation

Magnesium oxide and dolomite were mixed in a 1:1 ratio to produce a dry mix. 0%, 1%, 3%, 5%, and 10% of PVA was mixed with dry mix in different samples. A proper amount of magnesium sulfate solution (25⁰Be) was added to the dry mix to prepare a workable consistency paste of MOS [28]. To find out the effect of PVA on the properties of MOS, the following experiments were carried out. All the experiments were repeated three times, and then the average was reported. The samples of the experiments were prepared according to Table 1.

Table- 1: Samples of the experiment

Sample No.	Composition of the dry-mix	
	MgO : Dolomite	% of PVA
M1	1:1	0
P1	1:1	1
P2	1:1	3
P3	1:1	5
P4	1:1	10

METHODS

A. Setting Time Investigations

The influence of PVA on the setting properties of magnesium oxysulfate cement was investigated by admixing PVA in various quantities into the dry mix. Initial and final setting times were calculated using the Vicat needle instrument [29]. Figure 1 shows the results.

B. Water Resistance Investigations

A steam test was used to examine the impact of PVA on soundness of the product. To conduct this test, all setting time blocks with various quantities of PVA were cured under identical conditions for 60 days. After the curing period, the samples were placed in a closed steam chamber and exposed to boiling water for at least 30 hours. The relative resistance to moisture absorption was monitored over time. Since moisture ingress negatively affects soundness, an inverse relationship was observed between the two [30]. The outcomes of this study are summarized in Table 2.

C. Compressive Strength Investigations

To evaluate the effect of PVA on the compressive strength of MOS, standard cubes measuring 70.6 mm on each side (equivalent to a surface area of 50 cm²) were prepared using pastes with varying PVA content. After curing for 30 days under consistent conditions, the samples were tested for compressive strength using a compressive strength testing machine. The results are presented in Fig. 2.

D. Linear Changes

Standard blocks (200 mm × 25 mm × 25 mm) were cast using wet mixes with varying amounts of PVA additive to assess its effect on the linear changes in MOS. The initial lengths of the trial beams were measured after 24 hours. After 28 days of curing under the identical conditions, their final lengths were recorded. The difference between the two measurements indicated the linear dimensional change (either expansion or shrinkage). A smaller difference implies better dimensional stability and soundness of the material. The findings are presented in Table 3.

E. Spectral Analysis

Spectral analysis like as FT-IR was performed on M1 and P3 (best modified MOS sample).

RESULTS AND DISCUSSION

A. **Setting time analysis:** Incorporating PVA into MOS increases both the initial and final setting times (Fig. 1). As the amount of the additive increases, setting time (initial & final) also continuously increases. The M1 sample contains half amount of MgO and half amount of dolomite powder. Hence, the heat amount of the exothermic reaction is absorbed by the inert filler dolomite powder via a three-body collision mechanism. With respect to the M1 sample, as the amount of the PVA increases, the time of setting (initial & final) continuously increases. This retardation is due to the adsorption of PVA on MgO particles, forming a semi-permeable film that delays hydration. By forming hydrogen bonds with water, PVA may reduce the amount of free water available for early crystal growth. The setting delay was moderate up to 5% PVA, but at 10%, it became significantly pronounced.

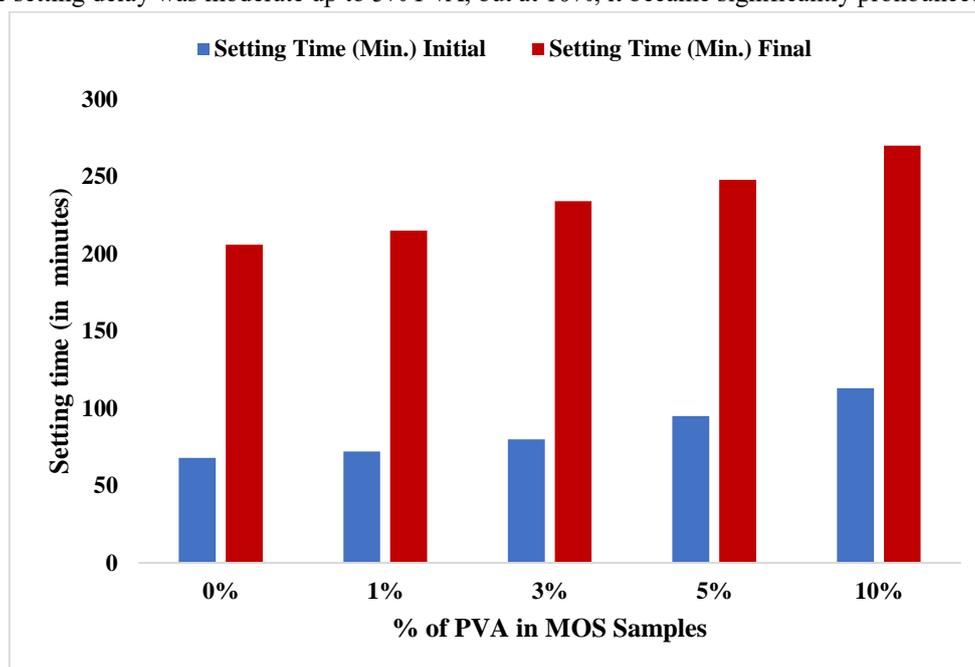


Fig. 1: Initial and final setting times of different types of MOS samples.

B. **Water resistance analysis:** The incorporation of PVA significantly enhances the water resistance of MOS. Polyvinyl alcohol (PVA), due to its film-forming and water-binding properties, modify the internal microstructure of MOSC to enhance water resistance, particularly by reducing capillary porosity, forming a water-repellent polymer film, and improving phase stability in moist conditions. The addition of PVA significantly enhances the water resistance of MOS, with the best performance at 5% addition.



Table 2: Effect of PVA on water resistance property of MOS

Sample	% of PVA in dry-mix Composition	Cement blocks were kept in boiling water for					
		0-5 hrs.	5-10 hrs.	10-15 hrs.	15-20 hrs.	20-25 hrs.	25-30 hrs.
M1	0%	-	-	-	-	-	-
P1	1%	-	-	-	-	-	-
P2	3%	-	-	-	-	-	-
P3	5%	-	-	-	-	-	-
P4	10%	-	-	-	-	-	-

C = crack - = No effect

C. **Compressive strength analysis:** Fig. 2 shows the effect of PVA on the compressive strength of MOS cement. Compressive strength improved by the incorporation of PVA additive. The highest compressive strength was observed for the P3 sample, which is 60% higher than the M1 sample. The strength of the P4 sample continuously decreases. The compressive strength increased from 32.5 MPa (0%) to 52 MPa (5%), indicating PVA’s role in improving the packing density and internal cohesion of the cement matrix. PVA likely promotes the formation of a denser microstructure by filling capillary pores, enhancing bonding between MgO, dolomite, and hydrated phases (especially the 5-phase: $5\text{Mg}(\text{OH})_2 \cdot \text{MgSO}_4 \cdot 8\text{H}_2\text{O}$), and improving flexibility and crack-bridging within the matrix.

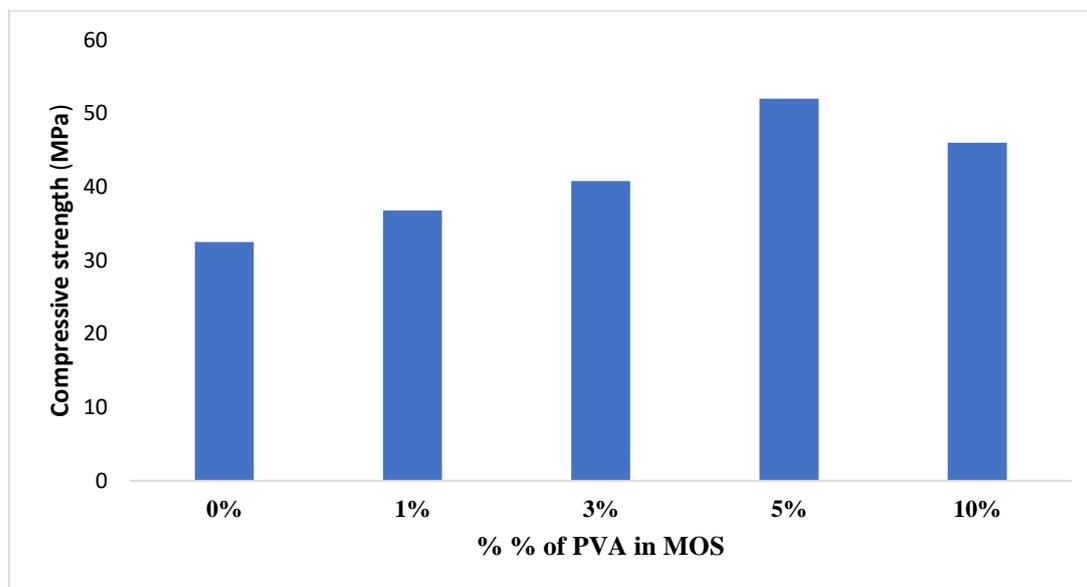


Fig. 2: Compressive strength of different type of MOS samples.

D. **Linear changes:** The linear dimensional stability of MOS was influenced by the addition of PVA, particularly in terms of expansion and shrinkage behavior. In unmodified MOS, slight linear shrinkage occurs during the initial setting and drying stages due to loss of free water and crystallization of hydration products such as the 5-phase ($5\text{Mg}(\text{OH})_2 \cdot \text{MgSO}_4 \cdot 8\text{H}_2\text{O}$). It was observed that mixes containing 1–5% PVA exhibited reduced shrinkage, with the 3–5% PVA mixes showing the lowest dimensional changes, indicating PVA’s role in restraining drying shrinkage and minimizing microcrack development due to its film-forming and water-retentive properties. However, at 10% PVA, slight dimensional expansion or instability was noted, potentially due to excess polymer causing delayed setting and residual internal moisture migration. Overall, the results support the use of moderate PVA dosages for enhancing dimensional stability of MOS cement.

Table 3 Effect of PVA on Linear changes of Magnesium oxysulfate cement

Sample	% of PVA in dry-mix Composition	Length of Beam		Change in Length (in mm)
		Initial	Final	
M1	0%	200.0	199.985	0.015
P1	1%	200.0	199.988	0.012
P2	3%	200.0	199.990	0.010
P3	5%	200.0	199.991	0.009
P4	10%	200.0	199.984	0.016

E. **FT-IR Analysis:** The spectra result of M1 and P3 are shown in Fig. 3. The FT-IR spectra of the MOS sample showed broad absorption bands in the range of 3700–3200 cm⁻¹, attributed to O–H stretching vibrations, and a peak around 1640 cm⁻¹, corresponding to H–O–H bending. These features indicate the presence of physically adsorbed water and hydroxyl groups from Mg(OH)₂ and other hydration products. When PVA was added, the O–H stretching band became broader and shifted slightly to lower wavenumbers, suggesting the formation of hydrogen bonds between PVA’s –OH groups and the hydration phases in the MOS cement. A noticeable reduction in the intensity of the 1640 cm⁻¹ band in PVA-modified samples indicated that some of the free water was likely bound by the polymer. In the region of 1120–980 cm⁻¹, assigned to the symmetric and asymmetric stretching of sulfate groups (SO₄²⁻), the peaks became sharper and more defined at PVA contents of 5%, reflecting improved crystallinity and greater phase stability of the 5-phase (5Mg(OH)₂·MgSO₄·8H₂O). A new, weak absorption band near 2900 cm⁻¹ was also observed in the PVA-containing mixes, corresponding to C–H stretching vibrations from the polymer backbone, confirming its successful integration into the matrix. Overall, the spectral changes indicate that PVA interacts physically with the MOS cement matrix, mainly through hydrogen bonding, which helps improve internal cohesion and water retention.

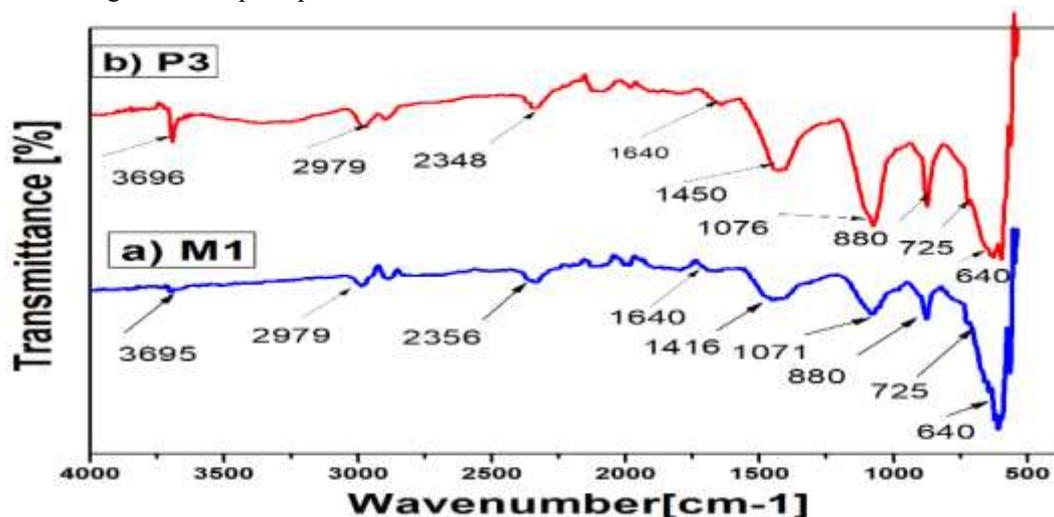


Fig. 3: Combine FT-IR spectra of M1 and P3

CONCLUSION

The addition of 5% PVA to MOS cement significantly improves compressive strength, water resistance, and stability. FT-IR analysis confirmed physical interactions between PVA and hydration products through hydrogen bonding. The water absorption decreased linearly up to 5% PVA, indicating enhanced water resistance. Overall, 5% PVA is an effective and ecologically friendly additive for increasing the durability, strength, and dimensional stability of MOS.

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COMPETING INTERESTS

The authors have no competing interests to disclose.

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