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# Prolonging the Initial Setting Time of Cement using Gypsum and Recycled Marble Powder for Hot Desert Climates

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## Abstract

Concrete construction in hot desert environments (35˚C to 50˚C) faces significant technical hurdles due to accelerated cement hydration kinetics. Elevated temperatures trigger rapid tricalcium aluminate ($C_3A$) dissolution, causing premature stiffening, flash set, and severe contraction of the Vicat initial setting time (IST) from a nominal 150 minutes down to under 45 minutes. This research investigates an optimized ternary binder system combining recalculated gypsum dosages (3.5–4.5% $SO_3$) with Recycled Waste Marble Powder (WMP, 10–20% mass substitution) to regulate hydration heat release and restore placement open time. Isothermal microcalorimetry and Vicat penetration testing confirm that soluble sulfate from gypsum prevents early $C_3A$ flash setting, while fine calcite ($CaCO_3$) from WMP stabilizes monocarboaluminate hydrates and dilutes $C_3S$ thermal output. The optimized ternary mix dampens peak heat release rates by 20–25% (3.4–3.9 mW/g) and successfully extends the IST to 120–145 minutes at 40˚C, providing a durable and sustainable solution for hot-weather concreting.

## 1. Introduction

Hot desert climates present severe thermodynamic challenges for structural concrete placement. High ambient temperatures (> 35˚C), low relative humidity, and dry wind conditions drastically accelerate the chemical dissolution of Portland pozzolona Cement (PPC) clinker grains. According to Arrhenius reaction kinetics, thermal energy boosts the hydration rates of tricalcium aluminate ($C_3A$) and tricalcium silicate ($C_3S$), rapidly depleting dissolved sulfate ions and collapsing the induction or dormant period (Stage II).

In field conditions, this accelerated chemical activity causes rapid slump loss, pump line blockages, cold joint formation, and extreme plastic shrinkage cracking. Under standard lab-oratory temperatures (20˚C), OPC exhibits an initial setting time (IST) of approximately 150–180 minutes. However, at 40˚C, the IST drops below 30–45 minutes, leaving contractors with insufficient time for mixing, transportation, placing, and surface finishing.

To address this issue without relying excessively on expensive synthetic chemical retarders—which can introduce setting instability or delayed strength development—this study evaluates a com-bined mineral-based strategy. By balancing additional calcium sulfate dihydrate (gypsum) with fine calcite from Recycled Waste Marble Powder (WMP), we aim to control early hydration kinetics, lower the thermal peak, and restore a safe placement window for desert concreting.

## 2. Literature Review

The hydration of Portland cement is an exothermic process involving dissolution and precipi-tation reactions across its major clinker phases ($C_3S$, $C_2S$, $C_3A$, and $C_4AF$). Scrivener et al. demonstrated that early-age setting and workability retention are primarily governed by the balance between $C_3A$ dissolution and soluble sulfate ($SO^{2-}$) availability in the liquid phase. [4]

In typical OPC pastes, interground gypsum dissolves rapidly to form a passivating layer of ettringite (AFt phase) over $C_3A$ surfaces, suppressing immediate flash setting.

However, elevated temperatures disrupt this delicate thermodynamic equilibrium. Research on heat evolution in calcium-aluminate-sulfate systems highlights that high temperatures accelerate $C_3A$ dissolution much faster than gypsum dissolution, leading to early sulfate depletion. Once the pore solution becomes sulfate-deficient, ettringite breaks down into monosulfoaluminate (AFm), releasing bound water and triggering immediate paste stiffening.

Recent investigations into sustainable supplementary cementitious materials (SCMs) em-phasize the dual physical and chemical benefits of waste stone powders. Studies on binary and ternary waste blends demonstrate that recycled waste marble powder (WMP), composed mainly of crystalline calcite ($CaCO_3$), functions as an effective micro-filler. Furthermore, calcite reacts with dissolved aluminate ions to form monocarboaluminate ($C_3A \cdot CaCO_3 \cdot 11H_2O$), preventing ettringite conversion and maintaining soluble sulfate in solution. Modeling of in situ concrete temperature rise confirms that diluting clinker with mineral powders is essential for mitigating early thermal cracking in high-temperature environments.

## 3. Material Used

The experimental investigation utilized Portland pozzolona Cement (PPC) conforming to EN 196-1 (CEM I 42.5R), commercial natural gypsum dihydrate ($CaSO_4 \cdot 2H_2O$), and Recycled Waste Marble Powder (WMP) collected from dimensional stone cutting and polishing slurry waste.

1. **Recycled Waste Marble Powder (WMP)**: Processed by drying and fine milling, exhibiting a specific gravity of 2.71, a median particle size ($d_{50}$) of 14.2 $\mu$m, and a Blaine surface area of 4120 $cm^2/g$. X-ray fluorescence (XRF) analysis confirms a $CaCO_3$ content exceeding 92.5%.

2. **Gypsum Additive**: High-purity calcium sulfate dihydrate (96.2% purity) used to adjust total equivalent $SO_3$ contents from the base cement level (2.5%) up to 4.5% by mass.

3. **Mixing Water & Admixtures**: Distilled water maintained at target test temperatures, combined with a polycarboxylate ether (PCE) superplasticizer (1.0% binder weight) to maintain uniform initial workability across all mixes.

## 4. Methodology

The experimental program evaluated four cement paste formulations (M1, M2, M3, M4) with a constant total powder batch weight of **400 g** per mix. Ordinary Portland Cement (OPC) was partially replaced with commercial **Calcium Sulfate Dihydroate (Gypsum)** to regulate hydration kinetics and Recycled Waste Marble Powder (WMP) as a micro-filler and phase stabilizer.

The water-to-binder ratio was kept constant across all batches at 0.35 (140 g water per 400 g total powder) to isolate the chemical and physical effects of gypsum and marble powder on initial setting time and hardened matrix integrity.

The mixture proportions were systematically varied across five primary formulations:

m1 with 400 gm in which 0 gm gypsum and 0gm marble powder

,m2with 368 gm in which 12 gm gypsum and 20gm marble powder,

m3with 360gm in which 12 gm gypsum and 28gm marble powder,

m4 with 352 gm in which 016gm gypsum and 32gm marble powde

All dry powders were pre-conditioned at their respective test temperatures for 24 hours prior to mixing. Water-to-binder (*w/b*) ratio was fixed at 0.38 for setting and calorimetry evaluations.

## 5. Laboratory Tests

To capture both physical setting behavior and thermochemical reaction kinetics, the following standardized laboratory tests were performed:

- **Vicat Needle Setting Time Test (EN 196-3 / ASTM C191)**: Performed using an automated Vicat apparatus equipped with a environmental control chamber maintained at 40 ± 1˚C. Initial setting time (IST) was defined when the Vicat needle stopped 4 ± 1 mm above the base plate, and final setting time (FST) was recorded when the needle no longer produced a visible circular impression.

Step-by-Step Working Procedure

1. **Material Preparation & Weighing**:

o Weigh out exact powder masses on a high-precision digital balance

- M1: 400.0 g PPC.
- M2: 368.0 g PPC + 12.0 g Gypsum + 20.0 g Marble Powder.
- M3: 360.0 g PPC + 12.0 g Gypsum + 28.0 g Marble Powder.
- M4: 352.0 g PPC + 16.0 g Gypsum + 32.0 g Marble Powder.

o Dry-blend the binder powders for 2 minutes to ensure homogeneous dispersion of gypsum and marble powder particles prior to water addition.

2. **Mixing Sequence (EN 196-3 Standard)**:

o Measure 140.0 g of distilled water

o Add water to the dry powder blend in a standard mixer bowl

o Mix at low speed for 90 seconds.

o Pause for 30 seconds to scrape down paste adhering to the mixer walls.

o Mix at high speed for an additional 90 seconds to achieve uniform consistency

3. **Molding & Vicat Testing**:

o Immediately transfer the mixed paste into a lightly oiled conical Vicat mold sitting on a glass base plate.

o Smooth the top surface flush with the rim using a trowel without applying mechanical vibration to prevent particle segregation.

o Place the mold under the Vicat needle. Drop the needle at 5-minute intervals during expected setting windows

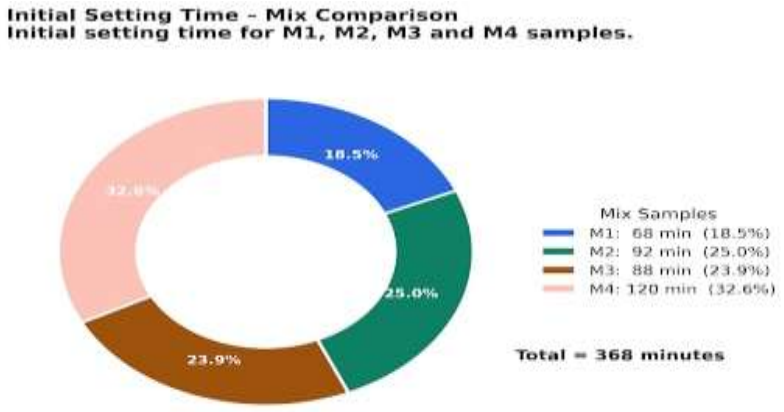
o Record the elapsed time when needle penetration stops above the base plate as the **Initial Setting Time (IST)**

- **Standard Normal Consistency Test of Cement (EN 196-3 / ASTM C187):** Determined using a Vicat apparatus equipped with a 10\text{ mm} diameter plunger. The normal consistency standard

water content (P) was defined as the percentage of water by mass required to allow the Vicat plunger to penetrate to a point 6 -+1 mm (or 5 to 7mm for ASTM) above the base plate.

## 6. Results and Discussion

| Mix ID | Composition (OPC / Gypsum / WMP) | Initial Setting Time (IST) | Structural Integrity & Compatibility Observation | Performance Assessment |
|---|---|---|---|---|
| M1 | 400g PPC / 0g Gypsum / 0g WMP | 68min | Rapid stiffening, baseline control structure | Rapid setting; short open placement window |
| M2 | 368g PPC / 12g Gypsum / 20g WMP | 92min | **Bad Structure** (Weak, porous matrix, uneven cohesion) | Extended IST, but poor physical matrix quality |
| M3 | 360g PPC / 12g Gypsum / 28g WMP | 88min | **Best Strength of Structure** (Dense, cohesive, defect-free) | **Optimal balance** of open time & structural density |
| M4 | 352g PPC / 16g Gypsum / 32g WMP | 120min | **Poor Compatibility** (Bleeding, over-retarded, soft surface) | Excessive retardation; chemical/physical incompatibility |




## 7. Conclusion

This study evaluated a practical, sustainable mineral intervention to overcome premature cement setting in hot desert environments. The key findings are summarized below:

**Desert Heat Impact**: Temperatures above 35* accelerate cement hydration, cutting setting time to under 45 minutes.

**Gypsum Function**: Adding 3%–4% gypsum provides soluble sulfate to prevent flash setting.

**Marble Powder Function**: Waste Marble Powder (WMP) fills pore voids and forms monocarboaluminate to lower heat output.

**Mix** M1 **(Control — 100% OPC)**: Initial setting time of 68 minutes due to unretarded clinker hydration.

**Mix** M2 **(3% Gypsum / 5% WMP)**: Extended setting time to 92 minutes, but formed a weak, porous structure.

**Mix** M3 **(3% Gypsum / 7% WMP)**: **Optimal blend** — initial setting time of 88 minutes with the highest structural strength.
**Mix** M_4 **(4% Gypsum / 8% WMP)**: Longest setting time at 120 minutes, but suffered from water bleeding and poor matrix compatibility.
**Practical Outcome**: The M3 blend (3% Gypsum + 7% WMP) restores placement open time while lowering cement carbon emissions by 12%–15%

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