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*Thème*

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**Valorization of marble waste in mortar  
partial replacement of ciment**

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Soutenu le, **08-07-2026**

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## ***Dedication***

*I dedicate the fruits of this humble effort...*

*To the Source of Tenderness and Light of My Life (My Mother): To the very first heart that cradled me, and the one whose memory is engraved with light upon my soul. To her who cleared the thorns from my path to plant love and hope, turning her own strength into a sanctuary for my sorrows and a nest for my dreams. You taught me resilience, remaining my guiding star and the ultimate balm for life's hardships.*

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Industrial marble processing activities generate massive volumes of discarded fragments, causing severe environmental challenges when left unmanaged. Upcycling these residues into construction materials offers an innovative solution that reduces waste and lowers the carbon footprint of building components. This dissertation investigates the incorporation of thermally activated marble powder (TAMP) into cement mortars and evaluates its effects on mechanical and fresh-state performance. The marble waste was manually collected, cleaned, ground, and thermally activated at 900°C for one hour.

The experimental program was carried out using a standard Portland-limestone cement (CEM II/A-L 42.5 N, NA 442) mixed with local sand. Mortar mixtures were prepared with varying TAMP contents (0%, 5%, 10%, and 15% by weight of cement). Fresh properties were analyzed through consistency and Vicat setting time tests. Compressive strength was assessed at 3, 7, and 28 days using prismatic specimens, while flexural tensile strength and durability metrics (water absorption and mass loss) were measured at later ages.

The results showed that the inclusion of 5% TAMP maintained excellent mechanical performance, retaining 98% of the 28-day control compressive strength. While higher substitution dosages (10% and 15%) experienced an early-age strength drop at 3 days due to binder dilution, their 28-day compressive values recovered past a structurally viable 31 MPa. Furthermore, the 10% and 15% mixes achieved a 3.9% and 3.5% increase in flexural strength, respectively, due to the micro-filler effect that densified the matrix and reduced capillary water absorption to 6.27%. In contrast, the 15% mix showed a higher mass loss under environmental exposure, highlighting performance limits at elevated dosages.

This study demonstrates the technical feasibility of recycling marble fragments in mortar, offering a dual benefit: environmental protection through waste valorization and optimized material performance.

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**Keywords:** Marble waste valorization, Thermal activation, Green binder, Eco-mortar, Mechanical performance, Circular economy, Startup (MarmorVivo).

تتسبب أنشطة تصنيع الرخام في توليد كميات ضخمة من الشظايا المهملة، مما يطرح تحديات بيئية جسيمة ويقدم إعادة تدوير هذه البقايا في مواد البناء حلاً مبتكراً يقلل من النفايات . عند التخلص منها بطرق عشوائية يتناول هذا البحث إمكانية إدماج مسحوق الرخام المنشط حرارياً . ويخفض البصمة الكربونية لمركبات البناء وقد تم . في الملاط الإسمنتي، ودراسة تأثيره على الخصائص الميكانيكية وفي الحالة الطازجة (TAMP) درجة مئوية لمدة ساعة واحدة 900 جمع مخلفات الرخام يدوياً، تنظيفها، طحنها، ثم تنشيطها حرارياً عند

(CEM II/A-L 42.5 N, NA 442) تم تنفيذ البرنامج التجريبي باستخدام إسمنت بورتلاندي مركب (0 %، 5 %، 10 % ) جرى إعداد خلطات الملاط بأربع نسب مختلفة من المسحوق . مخلوطاً بالرمل المحلي تم تقييم خصائص الملاط الطازج عبر اختبارات الاتساق وزمن الشك بواسطة . (من وزن الأسمنت 15 % و يوماً باستخدام عينات منشورية، بينما تم تقييم مقاومة 28 و 7 و 3 واختبرت مقاومة الضغط عند . جهاز فيكات الشد بالانحناء ومؤشرات الديمومة (امتصاص الماء وفقدان الكتلة) . عند أعمار لاحقة

98 من المسحوق حافظت على أداء ميكانيكي ممتاز، حيث احتفظت بنسبة 5 % أظهرت النتائج أن إضافة سجلت (15 % و 10 %) ورغم أن نسب الاستبدال العالية . يوماً 28 من مقاومة الضغط المرجعية عند % يوماً تعافت لتتجاوز 28 أيام نتيجة لتأثير التخفيف، إلا أن قيمها عند 3 انخفاضاً مبكراً في المقاومة عند عمر زيادة في مقاومة 15 % و 10 % علاوة على ذلك، حققت خلطات . ميغاباسكال المقبولة إنشائياً 31 عتبة على التوالي، نظراً لتأثير الحشو الميكروي الذي كثف المصفوفة وخفض 3.5 % و 3.9 % الانحناء بنسبة فقداً أكبر للكتلة تحت الظروف 15 % في المقابل، أظهرت خلطة . 6.27 % امتصاص الماء الشعري إلى الجوية، مما يوضح حدود الأداء عند النسب العالية

حماية : تُظهر هذه الدراسة إمكانية إعادة تدوير شظايا الرخام في الملاط بشكل فعال، مع تحقيق فائدة مزدوجة وتشجع النتائج على اعتماد هذا المسحوق في مشاريع . البيئة عبر تثمين النفايات وتحسين خصائص المواد البناء المستدام، خاصةً في العناصر مسبقة الصنع والتطبيقات غير الإنشائية، مع التوصية بإجراء دراسات إضافية حول الديمومة طويلة المدى.

**الكلمات المفتاحية:** تثمين نفايات الرخام، التنشيط الحراري، رابط أخضر، ملاط بيئي، الأداء الميكانيكي، (MarmorVivo) الاقتصاد الدائري، مؤسسة ناشئة.

Les activités de transformation du marbre génèrent d'importants volumes de fragments rejetés, causant de graves problèmes environnementaux lorsqu'ils ne sont pas gérés. Le recyclage de ces résidus dans les matériaux de construction offre une solution innovante qui réduit les déchets et abaisse l'empreinte carbone des composants du bâtiment. Ce mémoire examine l'incorporation de poudre de marbre thermiquement activée (TAMP) dans les mortiers de ciment et évalue ses effets sur les performances mécaniques et à l'état frais. Les déchets de marbre ont été collectés manuellement, nettoyés, broyés, puis activés thermiquement à 900°C pendant une heure.

Le programme expérimental a été réalisé en utilisant un ciment Portland au calcaire standard (CEM II/A-L 42.5 N, NA 442) mélangé à du sable local. Des mélanges de mortier ont été préparés avec différents taux de TAMP (0%, 5%, 10% et 15% par rapport au poids du ciment). Les propriétés à l'état frais ont été analysées par des essais de consistance et de temps de prise Vicat. La résistance à la compression a été mesurée à 3, 7 et 28 jours sur des éprouvettes prismatiques, tandis que la résistance en traction par flexion et les indicateurs de durabilité (absorption d'eau et perte de masse) ont été évalués à des âges ultérieurs.

Les résultats ont montré que l'ajout de 5% de TAMP maintient d'excellentes performances mécaniques, préservant 98% de la résistance à la compression du témoin à 28 jours (38,98 MPa). Bien que les dosages de substitution plus élevés (10% et 15%) aient subi une baisse de résistance à un âge précoce (3 jours) en raison de l'effet de dilution, leurs valeurs de compression à 28 jours ont récupéré pour dépasser un seuil structurellement fiable de 31 MPa. De plus, les mélanges à 10% et 15% ont obtenu une augmentation de la résistance en flexion de 3,9% et 3,5% respectivement, grâce à l'effet micro-filler qui a densifié la matrice et réduit l'absorption d'eau capillaire à 6,27%. En revanche, le mélange à 15% a présenté une perte de masse plus élevée sous exposition environnementale, soulignant les limites de performance aux dosages élevés.

Cette étude démontre la faisabilité technique du recyclage des fragments de marbre dans le mortier, apportant un double avantage : protection de l'environnement par la valorisation des déchets et optimisation des propriétés des matériaux.

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**Mots-clés:** Valorisation des déchets de marbre, Activation thermique, Liant vert, Éco-mortier, Performance mécanique, Économie circulaire, Startup (MarmorVivo).

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# Introduction

The modern civil engineering and construction industry is caught in a difficult paradox. On one hand, it must keep pace with rapid global urbanization; on the other, it must urgently curb the severe ecological damage caused by traditional building practices. Data from current literature indicates that the construction sector alone consumes nearly half of the world's fresh water and raw natural resources. Furthermore, it contributes roughly 40% of global greenhouse gas emissions. The ultimate core of this environmental crisis lies in manufacturing Ordinary Portland Cement (OPC), which remains the third-largest human-made source of carbon dioxide (CO<sub>2</sub>) emissions on Earth. Producing just one ton of OPC releases about 0.8 to 0.9 tons of CO<sub>2</sub> directly into the atmosphere, a consequence of both the fossil fuels burned to heat kilns up to 1400°C and the chemical calcination of limestone.

At the same time, regional ecosystems are straining under the weight of unmanaged solid industrial waste. Stone processing plants and quarries generate massive volumes of discarded marble fragments and fine powder slurry, with material waste rates frequently hitting 70% of the original rock massif. When left unchecked, dumping these marble residues causes severe local environmental and public health hazards. The fine particles choke riverbeds, alter groundwater chemistry by spiking alkalinity, turn fertile soils into sterile, impermeable layers, and trigger respiratory illnesses like silicosis when they become airborne.

Faced with these overlapping crises, the construction sector is adopting the principles of a "Circular Economy" and "Waste-to-Resource" frameworks. This engineering paradigm shifts away from old linear habits by routing industrial waste back into the manufacturing chain of building components, serving either as aggregate replacements or active binders. This approach significantly shrinks the carbon footprint of structural materials while safeguarding virgin natural reserves.

This research project is built directly within this sustainable framework. It explores the structural valorization of Carrara marble fragments which were manually cleaned, crushed, ground, and thermally activated at 900°C for one hour to serve as a partial mass-and-volume cement replacement in mortar at ratios of 5%, 10%, and 15%. Using a rigorous laboratory testing program, this study monitors how Thermally Activated Marble Powder (TAMP) alters the fresh properties of mortar (consistency and Vicat setting times), its mechanical strength as it hardens

## **Introduction**

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(compressive strength at 3, 7, and 28 days, plus flexural tensile strength), and its long-term durability metrics (water absorption by immersion and evaporation mass loss under ambient exposure).

# **Chapter I**

# **Recycling in Civil**

# **Engineering**

### I.1. Introduction

Traditional civil engineering has historically left a massive scar on the environment by sticking to a rigid "take-make-waste" approach. Right now, the global construction sector drives roughly 40% of all greenhouse gas emissions and pumps out staggering amounts of waste, pushing planetary ecosystems to a dangerous breaking point. To push back against this damage, modern construction is forcing a much-needed shift toward a circular economy, a model where cutting down on waste and protecting natural reserves take center stage.

A huge part of this green transition relies on the smart valorization of industrial byproducts. Essentially, this means taking the leftover waste and sludge from manufacturing plants and finding a way to give them a second life. Instead of letting these processing residues choke local habitats or pile up in landfills, engineers are turning them into useful construction feedstocks. For example, they can swap out a chunk of ordinary cement or natural sand inside standard mortar and concrete mixes. Ultimately, flipping these industrial wastes into high-value engineering assets gives the construction sector a real chance to slash its carbon footprint and actually protect the planet.

### I.2. Environmental Impact of Construction Activities

The environmental impact of construction is escalating, causing severe disruption to natural ecosystems. As infrastructure expands, traditional construction practices generate critical environmental liabilities (Oyebode, 2019).

#### I.2.1. Consumption of Natural Resources

The construction industry remains one of the largest consumers of natural resources, placing a massive strain on the Earth's finite supplies (WD Re-Thinking Ltd, 2010). Global construction demands result in the extraction of over 92 billion tons of materials annually, a rate growing by roughly 3.2% each year (Labaran et al., 2021). The ecological burden is characterized by:

- **Aggregates and Sand:** These dominate global resource usage, accounting for roughly 60% of all bulk materials used by humanity, Mining these materials permanently alters topography and degrades riverbeds (WD Re-Thinking Ltd, 2010).
- **Water Resources:** The sector consumes approximately 50% of available global freshwater, creating a direct threat to water security (WD Re-Thinking Ltd, 2010).
- **Biodiversity Loss:** Approximately 60% of global timber products and up to 90% of hardwoods are consumed by the built environment, leading to severe habitat fragmentation (Expedition Engineering, 2023).
- **Energy Intensity:** The building sector accounts for 45%–50% of global energy consumption, while the heavy processing of primary materials like cement and steel generates roughly 26% of global carbon emissions (Labaran et al., 2021).

### 1.2.2. CO<sub>2</sub> Emissions from Cement Production

The cement industry is the third-largest source of carbon dioxide (CO<sub>2</sub>) emissions, trailing only fossil fuels and land-use changes . Emissions arise from a combination of high-energy kiln operations and the chemical conversion of limestone (Andrew, 2019).

- **Process Emissions (Calcination):** Inside the kiln, limestone (CaCO<sub>3</sub>) is heated to 1400°C to produce lime (CaO). This chemical reaction is an inherent source of CO<sub>2</sub>, accounting for approximately 65% of the total emissions in the cement sector (Lehne & Preston, 2018).
- **Fuel Combustion:** To reach the temperatures required for calcination, kilns burn carbon-intensive fuels (coal, oil, gas). This accounts for the remaining 35% of industry emissions (Lehne & Preston, 2018).
- **Indirect Emissions:** Additional emissions are tied to the electricity consumed by grinding mills and logistics chains (WBCSD, 2011). Because process emissions are chemically tied to clinker production, they cannot be eliminated solely by fuel switching, necessitating research into alternative binders and carbon capture (Lehne & Preston, 2018).

### I.2.3. Environmental Degradation and Pollution

Beyond global emissions, active construction sites act as concentrated points of local environmental degradation. Rapid urbanization frequently outpaces existing mitigation and regulatory frameworks, leading to systemic ecological disorder.

- **Atmospheric and Noise Pollution:** Heavy machinery and material transportation release particulate matter (PM) and gases (NO, SO<sub>2</sub>), alongside significant noise pollution that impacts local biodiversity and human health (Hoque et al., 2024).
- **Soil and Water Contamination:** Improper waste management and runoff from excavation sites transport pollutants, including heavy metals and chemical residues, into local water bodies, destroying aquatic habitats (Hoque et al., 2024).
- **Long-term Ecological Disorder:** The permanent alteration of land-use patterns and the disruption of natural topography stress local ecosystems, leading to biodiversity loss that persists long after the construction phase concludes (Ali Mohsin et al., 2025).

## I.3. The Concept of Recycling and valorization in Civil Engineering

### I.3.1. Definition

- ✧ **Recycling:** In the context of sustainable material engineering, recycling is defined as the process of collecting, processing, and transforming discarded post-industrial or post-demolition waste materials into new functional resources or building components. Unlike basic reuse, which involves reinstating a component in its original form without structural alteration, recycling alters the physical, mechanical, or chemical state of the waste material (Kumbhar et al., 2013). In civil engineering, this typically manifests as mechanical processing (e.g., crushing concrete into recycled aggregates), thermal processing (e.g., thermal activation of mineral powders), or chemical alteration to produce alternative hydraulic binders, mortars, or structural elements.

✧ **Valorization:** In modern environmental engineering and material science frameworks, the concept of valorization represents a fundamental paradigm shift in treating industrial by-products. Waste valorization can be defined as any industrial processing activity or alternative technological method aimed at transforming waste streams or residual by-products into raw materials or products with a clear added economic and functional value (Rada, 2016). This engineering strategy specifically targets the recovery of chemical or physical properties inherent to the waste material, integrating them back into manufacturing cycles—such as substituting commercial binders or raw aggregates in construction material design (Djouimaa et al., 2021).

### I.3.2. Environmental and Economical Benefits of Recycling in Civil Engineering

#### ✧ **Environmental Benefits**

- **Reduction of Greenhouse Gas Emissions:** Integrating recycled mineral powders or industrial waste materials as supplementary cementitious materials (SCMs) directly reduces the demand for ordinary Portland cement (OPC). Because clinker production requires high-temperature calcination ( $\text{CaCO}_3 \longrightarrow \text{CaO} + \text{CO}_2$ ), every ton of OPC avoided eliminates approximately 0.8 to 0.9 tons of atmospheric carbon dioxide (Andrew, 2019).

- **Conservation of Natural Ecosystems and Biodiversity:** Utilizing recycled alternatives diminishes the demand for primary aggregate extraction. This limits habitat fragmentation, severe land contamination, and ecosystem disruption caused by traditional quarrying and riverbed dredging (Expedition Engineering, 2023).

- **Preservation of Landfill Capacity:** Large-scale recycling diverts high-volume, dense mineral wastes away from municipal and industrial waste repositories, extending the functional lifespan of existing landfills and reducing local environmental contamination (Czarnecki & Rudner, 2023).

- **Lowered Embodied Energy:** Processing recycled components typically demands significantly less thermal and mechanical energy compared to the raw extraction, multi-stage processing, and long-distance transport of virgin geological resources.

### ✧ Economical Benefits

- **Lower Raw Material Procurement Costs:** Discarded industrial or demolition by-products are frequently available at a fraction of the cost of premium virgin aggregates and traditional chemical binders, reducing overall material expenditures for masonry and structural mixes.

- **Reduction in Transportation and Logistics Expenditures:** By establishing localized waste processing and recycling loops on or near urban centers and demolition sites, contractors drastically cut the fuel and transport costs associated with shipping virgin materials from remote quarries.

- **Avoidance of Landfill Levies and Regulatory Penalties:** Many regions impose substantial financial penalties or strict dumping fees on construction waste. On-site segregation and transformation into recycled secondary components completely bypasses these operational costs.

- **Creation of Secondary Markets:** Developing structured recycling pathways drives new market demands for processed waste derivatives, establishing localized processing centers and providing regional economic value from materials previously classified as liabilities.

### I.3.3. Waste-to-Resource Approach

The "Waste-to-Resource" approach functions as a core operational column of the global circular economy strategy, moving away from traditional linear economic models (Extraction → Manufacturing → Disposal) that cause severe resource depletion and high carbon impacts (Hazemba and Clube, 2024). Adopting this perspective links two massive industrial sectors together: the stone processing industry (which generates substantial volumes of fine marble slurries and powder fractions) and the civil engineering construction

industry (which is globally responsible for intense energy consumption and greenhouse gas emissions) (Bellara et al., 2024). Transforming fine marble fractions from a localized environmental waste hazard into an engineered resource feedstock allows the construction sector to significantly preserve natural virgin aggregates, optimize binder matrices, and drastically reduce the carbon footprint and energy demands associated with traditional cement processing (Hazemba and Clube, 2024).

### I.3.4. Circular Economy

The circular economy (CE) represents a restorative and regenerative economic system that deviates from the conventional linear model of production and consumption (Afshari and Górecki, 2019). It functions as a systemic approach to economic development designed to benefit businesses, society, and the environment. Unlike the traditional linear model, which relies on the depletion of finite resources, the circular economy prioritizes the continuous circulation of materials, ensuring that resources retain their highest utility and value for as long as possible within the economy (Deshmukh et al., 2026).

#### ✧ Principles

The circular economy is governed by several core principles aimed at decoupling economic growth from the consumption of finite resources. Key principles include:

- **Design for Durability and Disassembly:** Products and structures are designed to be long-lasting and easily dismantled, allowing components to be recovered and reused at the end of their service life (Arup, 2016).
- **Resource Efficiency:** Maximizing the use of raw materials by minimizing waste generation during production and usage phases (Afshari and Górecki, 2019).
- **Closing the Loop:** Implementing systematic processes to recover, recycle, and repurpose materials, ensuring that waste streams are effectively reintegrated as secondary resources (Deshmukh et al., 2026).

- **Regeneration of Natural Capital:** Shifting focus toward renewable energy sources and sustainable material extraction to minimize environmental impact (Afshari and Górecki, 2019).

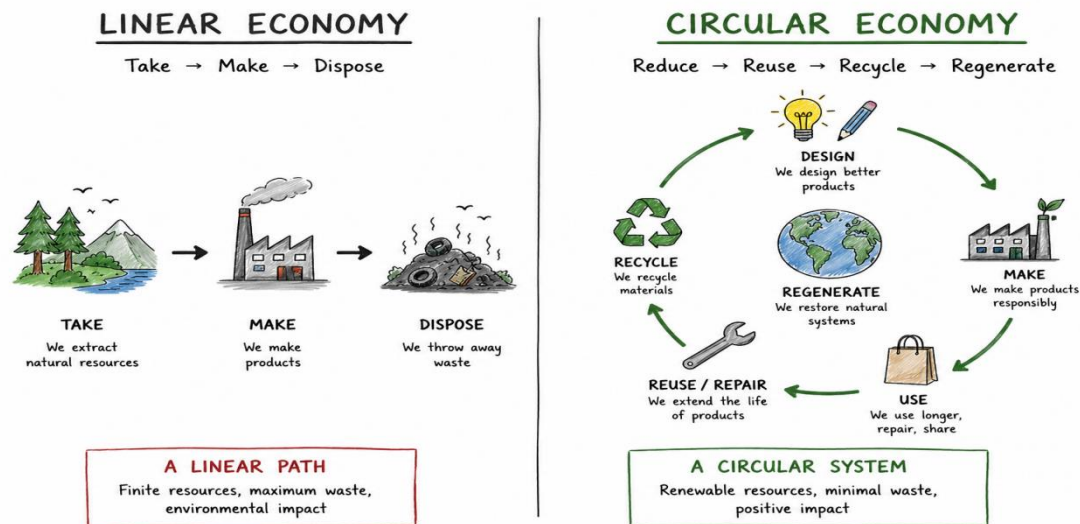


Figure I.1. Comparison between linear economy and circular economy

## I.4. Recycled Materials in Civil Engineering

### I.4.1. Industrial By-Products

The integration of industrial by-products into construction materials has become an essential strategy for mitigating the heavy environmental footprint of traditional infrastructure development. The civil engineering sector consumes a massive volume of raw natural resources while simultaneously contributing significantly to global solid waste production . To address this, common industrial by-products such as fly ash, silica fume, granulated blast furnace slag, and cement kiln dust are heavily researched and utilized as secondary cementitious materials or soil stabilizers (Bolden et al., 2013). Incorporating these mineral by-products into cementitious matrices not only minimizes the volume of industrial waste diverted to municipal landfills but also actively saves energy, reduces chemical air and water pollutants, and lowers overall greenhouse gas emissions during binder production (Bolden et al., 2013).

### **I.4.2. Recycled Aggregates**

Recycled concrete aggregates (RCA) obtained from construction and demolition waste (CDW) represent one of the largest potential material streams for sustainable construction. Research indicates that using recycled coarse aggregates extracted from old structural elements or demolished concrete components can successfully serve as a partial or full substitute for scarce natural aggregates in new concrete mixtures ( Neupane et al., 2023). However, the engineering performance and final compressive strength of the fresh composite matrix are highly dependent on the baseline quality and strength source of the source recycled aggregate. Because recycled aggregates possess a higher porosity due to old attached mortar, adjusting fresh mix designs either by adding calculated increments of mixing water or utilizing superplasticizer admixtures to maintain the target workability and slump is required to ensure structural performance (Neupane et al., 2023).

### **I.4.3. Glass and Ceramic Waste**

The utilization of crushed waste glass and fine ceramic residues offers a highly viable pathway for manufacturing green, high-performance concrete composites. Millions of tons of waste glass (CWG) and ceramic scraps are generated annually from consumer and industrial sources, presenting major management and environmental disposal challenges (Sabbrojjaman et al., 2024). When processed into a fine aggregate replacement, recycled fine glass and ceramics can successfully improve the mechanical and durability-related properties of composite mixtures. Furthermore, fine waste glass particles have been shown to drastically reduce the plasticity index, lower optimum moisture content, and optimize the California Bearing Ratio (CBR) and dry density parameters when utilized as an engineered mineral additive in civil engineering soil stabilization works (Sabbrojjaman et al., 2024).

### **I.4.4. Stone and Marble Waste**

Industrial processing activities within the ornamental stone cutting and marble sawing sectors generate immense volumes of raw stone fragments and highly problematic fine marble waste powder (Pathan et al., 2019). The accumulation of unmanaged marble dust and

slurry runoff creates severe local environmental problems, contributing to dust pollution and environmental degradation. Within civil engineering applications, utilizing waste marble powder as a partial replacement for fine aggregate or as a mineral addition to replace cement offers a dual benefit. It provides a highly cost-effective alternative to expensive commercial binders while protecting the local ecosystem from hazardous industrial dust accumulation (Pathan et al., 2019). Experimental studies confirm that integrating engineered fractions of marble powder can successfully maintain or optimize the physical and mechanical properties of concrete and mortar composites, forming a strong basis for its large-scale adoption in sustainable material design (Pathan et al., 2019).

### I.5. Conclusion

The data and research reviewed across this opening chapter provide a solid groundwork for using recycled industrial waste in everyday construction practice. Evaluating global building trends highlights a major contradiction: while civil engineering is the backbone of modern cities, it is also a primary driver behind shrinking natural resources, broken ecosystems, and massive carbon footprints. To fix these heavy ecological impacts, material science has shifted its focus away from the old, wasteful "take-make-waste" routine and toward green binders and sustainable alternatives.

At the center of this change is the smart reuse and engineering of industrial and demolition leftovers. Finding a new purpose for high-volume waste products—like slag, fly ash, old concrete aggregates, glass, and ceramics—does more than just keep trash out of local dumps. It preserves virgin stone reserves, extends the lifespan of landfills, and cuts down the total energy baked into these materials.

Looking closely at stone processing, the mountains of marble fragments and muddy sawing runoff create serious local disposal and pollution headaches. However, these fine stone by-products can do a lot more than just fill low-grade, non-structural gaps. True engineering valorization changes the physical and inner structure of these fine powders

through targeted processing or heat. This transforms an environmental waste hazard into an active ingredient that actively improves how fresh and hardened mortars perform.

In the end, these specific technical fixes are not just isolated laboratory trials. Instead, they offer a highly practical way to support the broader goals of a circular economy, trading destructive resource consumption for a regenerative setup that focuses on high efficiency and closed material loops. This entire baseline sets up a clear, scientific starting point for the lab testing, mix ratios, and physical data that we will dive into across the upcoming chapters.

# **Chapter II**

## **Marble valorization**

### II.1 Introduction

The continuous consumption of ordinary Portland cement and natural aggregates by the global construction sector places intense pressure on raw mineral resources. Concurrently, the stone processing sector generates massive quantities of unutilized carbonated mineral byproducts that are routinely discarded into terrestrial and aquatic ecosystems, altering groundwater chemistry and generating ambient particulate matter.

To bridge this ecological gap, contemporary research focuses on a circular economy framework: redirecting processed industrial marble waste back into cementitious manufacturing tracks as a partial substitute for active binders or fine aggregates. Far from being an inert space-filler, waste marble modifies the performance of cementitious materials through distinct physical, chemical, and mineralogical vectors. This chapter provides a concentrated evaluation of the life cycle, extraction processing, and consolidated physicochemical mechanisms that govern marble-modified hydration kinetics.

### II.2 Marble Extraction and Processing Life Cycle

The extraction of marble, or quarrying, marks the initial stage of the material's life cycle. Modern quarrying operations isolate giant rectangular stone massifs from the natural mountain face utilizing diamond wire saws, chain-cutter machines, or advanced drilling frameworks to eliminate micro-fissures (Gomes and Gomes, 2023). Once isolated, these immense stone segments are sawn and squared into commercial-grade rectangular raw blocks suitable for transport.

At secondary processing mills, these raw blocks are converted into commercial architectural slabs via high-speed multi-wire saw machines (Gomes and Gomes, 2023). To reinforce porous stones displaying structural micro-fissures, slabs are consolidated with epoxy or methyl methacrylate resins (Gomes and Gomes, 2023). Slabs then pass through automated lines where calibrating and polishing heads smooth out surface irregularities before computerized automatic cutting machines handle final sizing templates (Gomes and Gomes, 2023).



Figure II.1. Marble's life cycle

### II.3. Generation of Marble Waste

The transformation of raw geological formations into commercial architectural products is characterized by a highly linear, low-efficiency material lifecycle (Thakur, Pappu and Thakur, 2018). As industrial processing plants scale up production to meet global infrastructure demands, the volume of unutilized mineral by-products has increased exponentially (Kechagia et al., 2020). Rather than being treated as potential secondary resources, these carbonated mineral streams are routinely discarded into open ecosystems, introducing severe structural and environmental challenges (Fawad et al., 2021; Khan et al., 2025).

#### II.3.1. Quantity of Waste Produced

The quantified volume of waste generated by the marble industry is disproportionately high compared to the usable final product. The precise mass-balance metrics show the following losses:

- **Quarry Extraction Losses:** Data from the Mohamand marble zone and global extraction baselines show that practically half (50%) of the gross natural stone mass is lost immediately at the initial quarry site in the form of irregular stone scrap and fractured massifs (Fawad et al., 2021).
- **Gangsaw Slicing Losses:** Once the surviving blocks reach the processing mills, they are sawn into commercial slabs. Due to the thickness (kerf width) of diamond blades and multi-wire arrays, an additional 30% to 40% of the block mass is ground directly into ultra-fine slurry (State Bank of Pakistan, 2014).
- **Total Cumulative Loss:** Summing the extraction, trimming, shaping, and polishing stages, researchers establish that up to 70% of the original, in-situ marble resource is completely wasted before a finished tile is sold (Barzegar, 2026). This creates hundreds of millions of metric tons of mineral waste annually on a global scale (Thakur, Pappu and Thakur, 2018).

### II.3.2. Environmental Impact of Marble Waste Disposal

Unregulated dumping of untreated marble waste exerts severe environmental pressures across multiple ecosystems, as detailed in regional impact studies:

- **Destruction of Hydrological Networks and Aquifers:** Case studies tracking industrial discharges into seasonal streams (such as the Subhan Kharh stream which flows into the Kabul River) show that during rainy seasons, carbonated slurry flows directly into natural waterways (Fawad et al., 2021). The ultra-fine particles cause heavy sedimentation, clogging porous gravel riverbeds, choking aquatic life, and blocking natural subterranean aquifer recharge pathways, which triggers localized artificial flooding (Fawad et al., 2021).
- **Groundwater Alkalinization and Contamination:** When slurry interacts with regional water tables, it dramatically alters the chemical property parameters of the water. Industrial field measurements show that groundwater near marble clusters exhibits highly elevated Electrical Conductivity (EC), excessive Total Dissolved Solids (TDS), and high pH levels

that exceed safe limits, rendering the water completely unfit for domestic consumption or agricultural irrigation (Khan et al., 2025).

- **Soil Desiccation and Agricultural Sterilization:** When liquid marble sludge is dumped onto fertile land, it dries into a highly compacted, non-porous crust layer (Fawad et al., 2021). This hard mineral layer seals the soil surface, destroying its porosity, preventing rainwater infiltration, and shifting soil chemistry to a highly alkaline state (Fawad et al., 2021). This process leaves the land completely sterile and unable to support vegetation (Barzegar, 2026).

- **Air Pollution and Chronic Public Health Risks:** Upon complete desiccation of open-air slurry dumps, wind action easily lifts the dry powder into the atmosphere (Khan et al., 2025). This creates a persistent haze of suspended particulate matter (PM) that settles on nearby plants, blocking photosynthesis (Khan et al., 2025). Furthermore, it poses severe public health threats to surrounding communities, causing a high incidence of chronic respiratory conditions, including asthma, bronchitis, and silicosis (Barzegar, 2026).

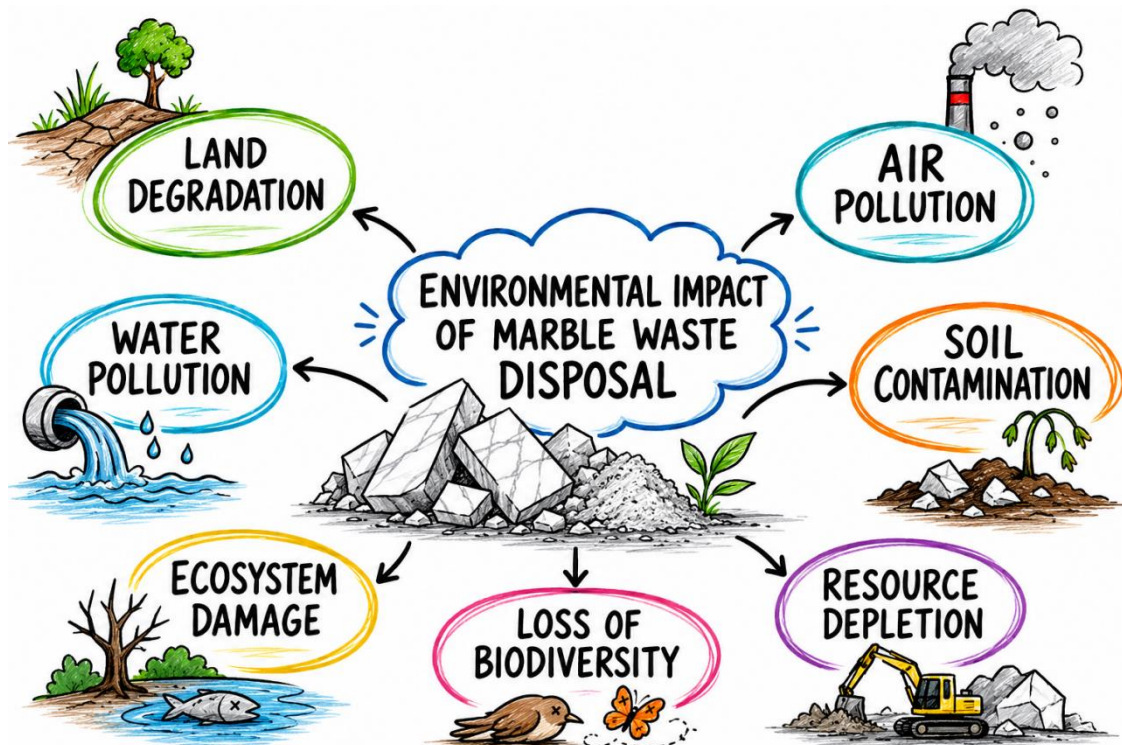


Figure II.2. Environmental impact of marble waste disposal

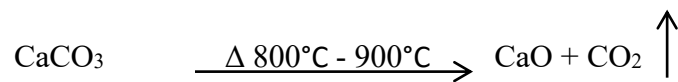
### II.4. Treatment of Marble Waste

To transform hazardous or unutilized marble by-products into valuable engineering resources, the raw waste streams must undergo systematic treatment operations (Bakshi et al., 2020). Untreated marble waste, particularly fine industrial sludge, cannot be directly integrated into high-performance civil engineering or environmental applications due to its high water content, variable particle sizing, chemical impurities from blade wear, and structural instability (Arslan et al., 2005; Pereira et al., 2026). Consequently, advanced processing frameworks including physical extraction, chemical purification, mechanical modification, and high-temperature mineralogical transformation are applied to standardize the material (Ali et al., 2025).

#### II.4.1 Thermal Activation and Calcination

High-temperature thermal activation is used to alter the underlying molecular chemistry and crystal structure of the stone, transforming stable calcium carbonate into highly reactive oxides (Ali et al., 2025).

- **The Calcination Process:** Dried marble powder ( $\text{CaCO}_3$ ) is subjected to thermal calcination inside high-temperature rotary furnaces or muffle kilns (Ali et al., 2025; Pereira et al., 2026). When temperatures cross the critical thermal threshold between  $800^\circ\text{C}$  and  $900^\circ\text{C}$ , a complete endothermic decomposition occurs, driving off carbon dioxide gas  $\text{CO}_2$  and converting the material into highly reactive quicklime ( $\text{CaO}$ ) (Science Class X, 2021; Ali et al., 2025):



- **Synthesis of Expansive Mitigating Agents:** Thermal profile variations determine the performance characteristics of the treated waste (Pereira et al., 2026). For instance, heating marble waste at  $900^\circ\text{C}$  for one hour alters its mineralogical phase to synthesize an effective expansive structural agent (Pereira et al., 2026). When incorporated into Portland cement

composites, this calcined material undergoes delayed hydration to form calcium hydroxide  $\text{Ca(OH)}_2$ , providing controlled mineral expansion that offsets natural autogenous shrinkage and drying cracks (Pereira et al., 2026).

**- Synthesis of Climate Mitigation and Carbon Removal (CDR) Materials:** When calcined marble waste is thermally co-processed with magnesium-silicate minerals (such as lizardite-rich feedstocks) at temperatures reaching  $750^\circ\text{C}$ – $900^\circ\text{C}$ , it undergoes structural restructuring (Ali et al., 2025). This thermal combination breaks down unreactive mineral matrices into a highly reactive mixture of forsterite  $\text{Mg}_2\text{SiO}_4$  and lime  $\text{CaO}$  (Ali et al., 2025). When exposed to industrial emissions or atmospheric moisture, this activated material converts rapidly into stable synthetic calcite, providing a sustainable pathway for permanent carbon capture and storage (CDR) (Ali et al., 2025).

### II.5 Consolidated Physicochemical Properties and Cementitious Mechanisms

When integrated into cement mortars and concrete mixtures, waste marble influences performance through several concurrent mechanisms: the filler effect, dilution effect, nucleation effect, and direct interaction with hydration chemistry (Ghorbani et al., 2020). The raw physical, chemical, and mineralogical properties of the waste map directly onto these construction mechanisms:

#### II.5.1 Physical Fineness and the Filler Effect

**- Property Baseline:** Laser diffraction analysis indicates that industrial marble waste powder exhibits an ultra-fine particle size distribution (PSD), with a significant volume of grains falling below  $50\text{--}60\mu\text{m}$  (Attia et al., 2022). In sand-replacement mortar formulations, the particle diameter concentrates between  $0.075\text{ mm}$  and  $0.125\text{ mm}$  (Guendouz et al., 2024).

**- Cementitious Mechanism:** While uncalcined marble dust lacks independent hydraulic reactivity, its extreme physical fineness allows it to pack into the microscopic interstitial gaps between sand aggregates and cement grains.

- **Structural Impact:** This physical micro-filler action fills internal voids and densifies the porous Interfacial Transition Zone (ITZ) around aggregates. This reduces total capillary porosity by up to 27%, directly mitigating weight-bearing vulnerabilities and enhancing bulk density and mechanical compactness ( Ghorbani et al., 2020).

### II.5.2 Mass Gravity and Volumetric Balancing

- **Property Baseline:** The specific gravity of marble waste powder ranges between 2.65 and 2.72 (Guendouz et al., 2024), while its loose dry bulk density varies from 1.15 to 1.45 g/cm<sup>3</sup> based on mechanical compaction limits (Thakur, Pappu and Thakur, 2018).

- **Cementitious Mechanism & Impact:** Because this physical gravity range matches standard quartz sand and conventional limestone aggregates (Kechagia et al., 2020), marble waste can replace fine aggregates volumetrically without modifying the design dead loads, volume-to-mass batch equations, or spatial yield of the target concrete mixtures.

### II.5.3 Grain Morphology, Water Demand, and Workability

- **Property Baseline:** Mechanically crushed marble waste features an angular, irregular grain morphology with sharp edges and rough surface textures (Samad et al., 2021). This geometry yields an elevated Blaine specific surface area typically ranging from 3000 to 4500 cm<sup>2</sup>/g (Kechagia et al., 2020).

- **Cementitious Mechanism & Impact:** This immense surface area demands higher early water volumes to fully coat the particle lattices, which can stiffen fresh mixtures and reduce slump flow values ( Guendouz et al., 2024). However, under optimized dosing thresholds or with the inclusion of small superplasticizer fractions, the smooth crystalline facets of the calcite grains function as internal lubricants, enhancing thixotropic stability, fresh consistency, and flow times in self-compacting mortars (Li et al., 2019).

### II.5.4 Chemical Profile and the Dilution Effect

- **Property Baseline:** X-ray Fluorescence (XRF) spectroscopy confirms that the dry powder is chemically dominated by Calcium Oxide  $\text{CaO}$ , which constitutes 50.0% to 55.5% of its absolute dry mass (Handayani et al., 2023). This corresponds to a raw Calcium Carbonate  $\text{CaCO}_3$  mineral purity exceeding 90–95% (Pereira et al., 2026). Secondary structural oxides are limited, containing 0.5%–4.0% Silica  $\text{SiO}_2$ , under 1.0% Aluminum Oxide  $\text{Al}_2\text{O}_3$ , and 0.4%–2.5% Magnesium Oxide  $\text{MgO}$  (Handayani et al., 2023). The carbonate-bound configuration yields a high Loss on Ignition (LOI) between 40.0% and 44.0% due to  $\text{CO}_2$  gas release above  $800^\circ\text{C}$  (Science Class X, 2021).

- **Cementitious Mechanism:** The dilution effect occurs when a chemically active clinker binder phase is replaced by this inert mineral powder. Because raw marble consists primarily of stable calcium carbonate, it cannot replicate the hydraulic clinker reactions responsible for creating long-term strength-giving calcium silicate hydrate (C-S-H) gels.

- **Structural Impact:** Consequently, replacing cement directly with marble powder progressively reduces the concentration of active binder, leading to a linear downward trend in long-term compressive and flexural strength values at high volumes. To avoid structural deficits, direct cement substitution should be restricted to conservative limits under 10% to 25% (Ghorbani et al., 2020).

### II.6 Conclusion

The utilization of industrial marble waste within concrete and mortar composites is a balanced trade-off between its structural physical advantages and chemical clinker limitations. Because raw uncalcined marble is chemically non-pozzolanic, the dilution effect sets a definite ceiling on direct cement substitution; exceeding conservative boundaries (10% to 25%) dilutes the active binder volume, leading to a drop in long-term strengths. However, these mechanical drop-offs can be effectively counteracted by leveraging the material's physical attributes.

The filler effect optimizes particle packing to reduce matrix porosity, while the nucleation effect provides heterogeneous templates that accelerate early clinker hydration kinetics. By carefully restricting direct binder substitution while optimizing aggregate replacement within a 20% to 30% range, the civil engineering sector can safely upcycle hundreds of millions of tons of industrial stone waste. This structural path provides a scalable approach to producing high-compactness, environmentally sustainable infrastructure without sacrificing early mechanical safety margins.

# **Chapter III**

## **Materials and Methods**

### III.1. Introduction

This chapter comprehensively outlines the materials, precise mix proportions, and standardized laboratory procedures utilized throughout the investigation of the behavior of marble powder in a mortar matrix. Specifically, this study focuses on the structural valorization of Carrara marble waste as a partial, mass-for-mass substitution for cement.

Initially, the chemical, mineralogical, and physical properties of the baseline components, consisting of cement, sand, and processed Thermally Activated Marble Powder (TAMP), are thoroughly characterized in compliance with current national and international normative frameworks. Following this, the precise chemical protocols, sequential mechanical mixing sequences, casting steps, and water-curing regimes required to produce consistent 4×4×16 cm prismatic specimens are detailed. Finally, the testing criteria for both fresh-state hardened performance traits (compressive strength, flexural tensile strength, water absorption by total immersion, and long-term evaporation mass loss) are defined. This multi-parameter approach establishes a rigorous comparative framework between the control group and the TAMP-modified variants.

### III.2. Materials

#### III.2.1. Cement

The cement used in this study is a Portland-limestone cement, **CEM II/A-L 42.5 N**, provided by the **GICA** group (GICA BÉTON) and conforming to the Algerian standard **NA 442-2013**. This cement is characterized by high early strength and is suitable for reinforced concrete and civil engineering structures.

The technical characteristics, as provided by the manufacturer's data sheet, are summarized in **Table III.1**. Notably, the clinker mineralogy, determined by Bogue's equations, shows a high tricalcium silicate (**C<sub>3</sub>S**) content of **60 ± 3%**, which contributes to the strength development observed in the 28-day compression tests (**≥ 42.5 MPa**). The physical properties include a Blaine fineness ranging between **3200 and 4200 cm<sup>2</sup>/g** and an initial setting time of at least **+ 60 minutes**.

**Table III.1: Technical characteristics of CEM II/A-L 42.5 N cement**

| Category      | Characteristic                  | Value / Range                | Standard    |
|---------------|---------------------------------|------------------------------|-------------|
| Chemical      | Sulfate content ( $SO_3$ )      | <2.5%                        | NA 442-2013 |
|               | Chloride content ( $Cl^-$ )     | <0.1%                        | NA 442-2013 |
|               | Magnesium oxide ( $MgO$ )       | <1%                          | NA 442-2013 |
|               | Loss on Ignition (PAF)          | 0.5–8.5%                     | NA 442-2013 |
| Mineralogical | Tricalcium silicate ( $C_3S$ )  | 60±3%                        | Bogue       |
|               | Tricalcium aluminate ( $C_3A$ ) | 3–7.5%                       | Bogue       |
| Physical      | Blaine fineness (SSB)           | 3200–4200 cm <sup>2</sup> /g | -           |
|               | Initial setting time            | ≥60 min                      | -           |
|               | Final setting time              | 150–350 min                  | -           |
| Mechanical    | Compressive strength (2 days)   | ≥10 MPa                      | EN 196-1    |
|               | Compressive strength (28 days)  | ≥42.5 MPa                    | EN 196-1    |



Figure III.1 Commercial GICA Béton CEM II/A-L 42.5N cement used in the study.

### III.2.2. Fine Aggregate

The fine aggregate used in this research is a local siliceous sand. To prepare the material for testing, the sand was subjected to a drying process in a laboratory oven at **105°C for 24 hours** to eliminate all moisture and achieve a constant mass. The physical properties of the sand were determined through a series of standardized laboratory tests, including sieve analysis and sand equivalent tests. The results of these characterizations are summarized in **Table III.2**. The sand equivalent value (ES) of **92.52%** indicates a high degree of cleanliness, making it suitable for high-performance mortar. Furthermore, the fineness modulus (FM) was calculated as **2.07**, classifying the material as a fine sand.

Table III.2: Physical properties of the natural sand.

| Property         | Formula                                                           | Value                 |
|------------------|-------------------------------------------------------------------|-----------------------|
| Fineness Modulus | $FM = \frac{\text{Sum of cumulative \% retained on sieves}}{100}$ | 2.07                  |
| Sand Equivalent  | $Es = \frac{H1}{H2} * 100$                                        | 92.52                 |
| Absolute Density | $\rho = \frac{m}{V}$                                              | 2666Kg/m <sup>3</sup> |



Figure III.2 Laboratory sieve shaker used for the granulometric analysis of the sand.

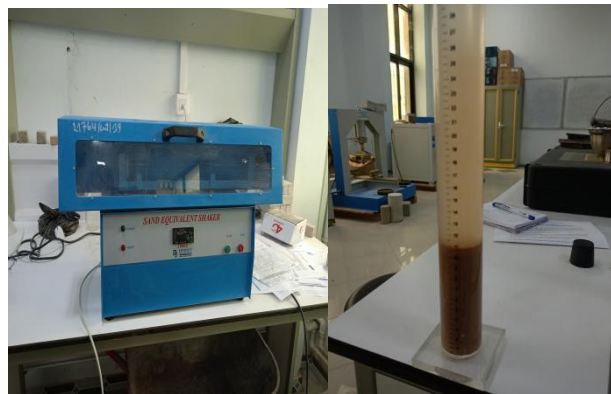


Figure III.3 Sand equivalent test (ES) used to determine the cleanliness and proportion of fine clay particles.

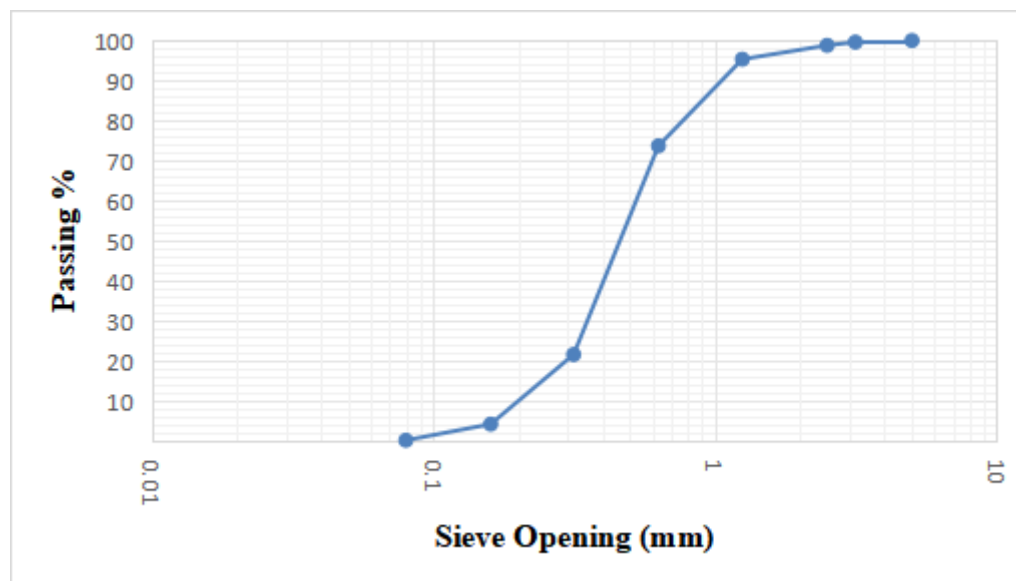


Figure III.4 Particle size distribution curve (grading curve) of the natural sand.

### III.2.3. Marble

The marble used in this study was sourced from Carrara marble waste, specifically discarded fragments collected after industrial cutting processes. To transform the raw marble pieces into a suitable cementitious replacement, a multi-stage preparation process was executed:

#### A. Preparation Protocol

To ensure the material met the requirements for mortar substitution, a four-stage processing sequence was implemented

**1- Purification:** The marble fragments were washed under pressurized running water to remove surface dust and organic contaminants. The fragments were then dried to a constant mass to ensure no residual moisture would affect the grinding efficiency.



**Figure III.5 Raw Carrara marble waste fragments during the cleaning process.**

**2- Mechanical Grinding:** The dry fragments were reduced to powder using a **Los Angeles abrasion machine** (conforming to NF EN 1097-2) utilized as a high-energy ball mill. The marble was processed with a internal steel charge until sufficient fineness was achieved. Then the powder was sifted in a 0.078mm sieve to achieve the final result.



**Figure III.6 Mechanical grinding of marble fragments using the Los Angeles machine.**

**3- Thermal Activation:** The resulting powder was subjected to a thermal treatment in a laboratory muffle furnace (illustrated in **Figure III.7**). The material was heated to **900°C** and maintained at this temperature for **1 hour**. This process aims to enhance the chemical reactivity of the calcite through partial decarbonization.

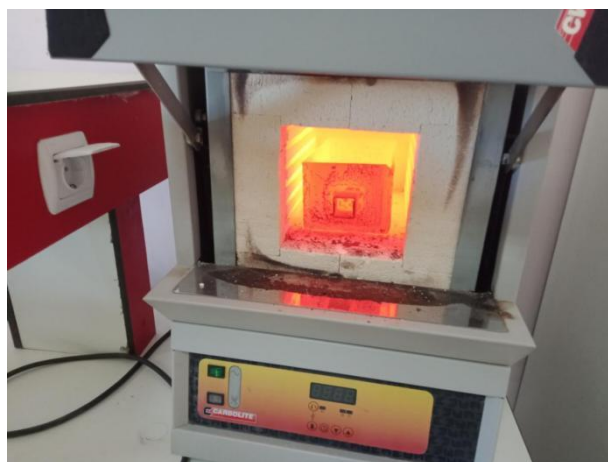


**Figure III.7 Thermal activation of marble powder in a Carbolite muffle furnace at 900°C.**

**4- Controlled Cooling and Storage:** Following the activation period, the material was cooled gradually within the furnace to avoid thermal shock. To prevent pre-hydration or carbonation from atmospheric humidity, the TAMP was stored in a sealed paper bag.

### B. Physical Characterization

The physical properties of the TAMP were determined through standardized laboratory testing. The absolute density was measured using the pycnometer method, while the fineness was assessed via the Blaine air-permeability test (**Figure III.8**).

**Table III.3: Physical Properties of the Prepared Marble Powder**

| Property                  | Value | Unit               |
|---------------------------|-------|--------------------|
| Absolute Density          | 2703  | kg/m <sup>3</sup>  |
| Specific Surface (Blaine) | 1075  | cm <sup>2</sup> /g |



**Figure III.8 Measurement of TAMP fineness using an automatic Blaine air-permeability apparatus.**

### III.3. Mix Design and Formulation of Mortars

The formulation of the mortar mixtures in this study is based on the standardized composition as defined by **NF EN 196-1**. The design maintains a constant Binder/Sand ratio of **1/3** and a Water/Binder (W/B) ratio of **0.5**.

### **III.3.1. Mix Proportions**

The baseline proportions for a single batch are detailed in **Table III.4**. These quantities ensure the production of three standardized 4×4× 16cm prisms per batch.

**Table III.4: Mix design proportions for mortar.**

| <b>Component</b>                        | <b>Proportion</b> | <b>Quantity<br/>(g)</b> | <b>Specification / Details</b>        |
|-----------------------------------------|-------------------|-------------------------|---------------------------------------|
| <b>Binder (Cement<br/>+TAMP)</b>        | 1                 | <b>450.000</b>          | CEM II/A-L 42.5 N<br>(GICA)           |
| <b>Sand</b>                             | 3                 | <b>1350.000</b>         | Standardized fine<br>aggregate        |
| <b>Water</b>                            | 0.5               | <b>225.000</b>          | Potable / Distilled                   |
| <b>Admixture(super<br/>plasticizer)</b> | 0.0075            | <b>3.375</b>            | Polycarboxylate-based<br>(0.75% bwob) |
| <b>Water-Binder<br/>Ratio (w/b)</b>     | 0.5               | -                       | -                                     |

### **III.3.2. Mortar Identification and Substitution Rates**

To test the effect of Thermally Activated Marble Powder (**TAMP**), four distinct mortar groups were formulated. The substitution of cement by TAMP was performed on a mass-for-mass basis.

- ♦ **M0 (Control):** Reference mortar with 100% cement and 0% TAMP.
- ♦ **M5:** Mortar containing 5% TAMP and 95% cement as the binder.
- ♦ **M10:** Mortar containing 10% TAMP and 90% cement as the binder.
- ♦ **M15:** Mortar containing 15% TAMP and 85% cement as the binder.

#### III.3.3. Mixing Procedure and Protocol

To ensure maximum homogeneity and eliminate the risk of structural segregation across all mixtures (M0, M5, M10, and M15), a standardized mechanical mixing procedure was strictly followed in compliance with the **NF EN 196-1** standard. The operation was carried out using a standard planetary laboratory mixer at a controlled ambient temperature of  $20 \pm 2^\circ\text{C}$ .

The exact sequential steps of the mixing protocol were executed as follows:

- a) **Initial Binder Wetting:** The total quantities of distilled water and the designated binder component (comprising the pre-blended CEM II/A-L 42.5 N cement and the corresponding percentage of Thermally Activated Marble Powder) were added to the mixing bowl. The mixer was started immediately at a low speed for a continuous duration of **30 seconds**.
- b) **Aggregate Introduction:** While maintaining the mixer at a low speed, the entire mass of the sand 1350 g was added steadily and uniformly over the next **30 seconds**.
- c) **High-Speed Acceleration:** The mixer was then switched to a high-speed setting and run for an additional **30 seconds**.
- d) **Rest and Scraping Period:** The machine was stopped for a total rest period of **90 seconds**. During the first 30 seconds of this pause, a scraper was used to detach any unmixed mortar paste or accumulated fine material adhering to the walls and bottom of the bowl, pushing it back into the center of the mixture. The mortar was then allowed to stand undisturbed for the remaining 60 seconds.
- e) **Final Homogenization:** The mixing process was concluded by restarting the machine at high speed for a final, intensive cycle of **60 seconds**.



**Figure III.9 Standard planetary mortar mixer (UTEST)**

**Note: Important Chemical Protocol Regarding the Admixture**

The polycarboxylate-based superplasticizer (0.75% by weight of binder) was incorporated exclusively during this mortar mixing stage. It was pre-dissolved directly into the mixing water prior to contact with the dry binder components to optimize its dispersing efficiency, ensure a stable fresh state consistency, and control workability across the varying replacement rates. Crucially, this chemical admixture was strictly excluded during the raw binder paste tests conducted via the Vicat apparatus (standard consistency and initial/final setting time determinations) to ensure that the fundamental hydraulic kinetics of the TAMP-cement blends were evaluated under pure, unadulterated baseline conditions.



**Figure III.10 SikaPlast® 40 Pro third-generation superplasticizer**

### III.3.4. Preparation and Casting of Specimens

Following the mixing phase, the mortar specimens were prepared according to the following procedure:

- ♦ **Molding:** Triple-gang steel molds were used to cast the mortar into prisms with standard dimensions of **4×4× 16cm**.
- ♦ **Filling and Compaction:** To ensure the removal of entrapped air and to achieve maximum density, the molds were filled in two equal layers. Each layer was compacted using a standardized jolting apparatus.
- ♦ **Leveling:** Once compaction was complete, a metal straightedge was used to strike off the excess mortar. This ensured that the top surface of each specimen was perfectly flush with the edges of the mold.

### III.3.5. Curing and Storage

Controlled curing is essential for the hydration of both the cement and the marble powder. The following conditions were maintained:

- ♦ **Initial Period:** The filled molds were stored in the laboratory at a temperature of **20 ± 2°C** for a duration of **24 hours**.
- ♦ **Demolding and Identification:** After the first 24 hours, the specimens were carefully demolded. Each prism was labeled with its specific substitution rate (0%, 5%, 10%, or 15%) and the date of casting to ensure accuracy during testing.
- ♦ **Water Curing:** The labeled prisms were submerged in a water tank. The specimens remained fully submerged until the time of their scheduled mechanical tests.
- ♦ **Testing Ages:** Specimens were removed from the curing tank only at the designated ages of **3, 7, and 28 days** for strength evaluation.

### III.4. Characterization and Testing Procedures

The experimental program was designed to evaluate the influence of TAMP on both the fresh and hardened properties of the mortar. All tests were conducted in accordance with international and national standards to ensure the reliability of the results.

#### III.4.1. Fresh State Properties

Before casting the specimens into molds, the following properties were assessed to understand the workability and reactivity of the mixtures:

**Consistency and Setting Time:** The binder paste's initial and final setting times were determined using a standardized **Vicat apparatus** (as shown in **Figure III.10**) as defined by **NF EN 196-3**. This test ensured that the substitution of cement with marble powder did not negatively impact the hydration timeline or the manageability of the mortar.



**Figure III.11 Vicat apparatus used for determining the standard consistency and setting time of the binder pastes.**

### III.4.2. Mechanical Properties

The evaluation of the mortar in its hardened state focused on its mechanical performance, specifically its resistance to compressive and tensile forces. These tests were conducted in strict accordance with the **NF EN 196-1** standard. To ensure the statistical accuracy of the data, each test was repeated multiple times across different specimens.

#### A. Compressive Strength Test

The compressive strength was measured using a standard hydraulic press (as shown in **Figure III.12**). Specimens were tested at the specific ages of **3, 7, and 28 days** to monitor the strength gain over time. The two halves of the prisms resulting from the flexural test were placed between the platens of the press and loaded until failure.



**Figure III.12 Hydraulic press displaying failure load and stress-time curve during compression testing**

#### B. Flexural Tensile Strength Test

The tensile capacity of the mortar was evaluated through a three-point flexural test. The results for this specific characterization were obtained at the age of **28 days**, representing the standardized maturity of the mortar. The test was performed using a 3-

**point flexion apparatus** following the normative procedures to ensure consistent results across all mixtures (M0, M5, M10, and M15).



**Figure III.13. Three-point flexural tensile strength testing configuration on a 4×4×16 cm mortar prism specimen**

### III.4.3. Physical Properties

#### A- Water Absorption Test

To evaluate the capillary porosity and permeability characteristics of the modified mortar matrices, a water absorption by total immersion test was conducted over the 28-day curing cycle.

The experimental procedure was carried out as follows:

- **Initial Mass Determination ( $M_0$ ):** Immediately following the 24-hour initial hardening period, the specimens were carefully demolded and weighed in their fresh state to establish their initial mass ( $M_0$ ).
- **Saturation Phase:** The prisms were then fully submerged in a water storage container for a duration of **28 days**. This extended immersion allowed the mortar to undergo complete hydration while water filled the accessible open pore network.
- **Saturated Mass Determination ( $M_s$ ):** At the conclusion of the 28-day curing period, the specimens were removed from the water container, gently wiped with a damp cloth to remove excess surface water, and immediately weighed to obtain the saturated surface-dry mass ( $M_s$ ).

- **Oven Drying Phase:** Following the saturation measurement, the specimens were placed in a ventilated drying oven maintained at a constant temperature of **105°C** for **24 hours**.
- **Dry Mass Determination ( $M_d$ ):** After 24 hours of drying, the specimens were removed from the oven, cooled to room temperature, and weighed one final time to determine the absolute dry mass ( $M_d$ ).

The water absorption percentage ( $A_w$ ) is calculated using the following standardized relationship:

$$A_w = \frac{M_s - M_d}{M_d} * 100 \quad (\text{III.1})$$

Where:

$M_s$  is the saturated surface-dry mass of the specimen after 28 days of immersion (g).

$M_d$  is the final dry mass of the specimen after oven drying (g).

#### **B- Mass Loss Test**

To evaluate the kinetics of water evaporation and gain insight into the drying shrinkage behavior of the modified mortars, a long-term mass loss stability test was conducted in a controlled laboratory environment.

The experimental sequence was performed as follows:

- ◆ **Initial Reference Mass ( $M_0$ ):** Immediately following the demolding process after 24 hours of initial casting, each prismatic specimen was weighed to determine its initial baseline mass ( $M_0$ ).
- ◆ **Storage Environment:** Instead of being placed in the curing tank, these specific specimens were stored in an open, dry, and ventilated location within the laboratory, maintaining standard ambient temperature and relative humidity conditions.

- ◆ **Periodic Monitoring:** To track the rate of moisture loss over time, the specimens were systematically re-weighed at regular **15-day intervals**.
- ◆ **Data Analysis:** The reduction in weight recorded at each interval represents the evaporation of free, uncombined water from the capillary network of the mortar.

The percentage of mass loss ( $LM_t$ ) at any given testing age ( $t$ ) is calculated using the following relationship:

$$LM_t = \frac{M_0 - M_t}{M_0} * 100 \quad (III.2)$$

Where:

$M_0$  is the initial mass of the specimen immediately after demolding (g).

$M_t$  is the mass of the specimen recorded at the specific monitoring interval (e.g., day 15, day 30, etc.) (g).

#### III.5. Conclusion

This chapter detailed the specific materials and experimental methods used in this study. Presenting the fundamental properties of the cement, aggregates, water, and thermally activated marble powder (TAMP), alongside the exact laboratory steps required to process and prepare them. The mix proportions were systematically built around incremental replacement levels ( $M_0$ ,  $M_5$ ,  $M_{10}$ , and  $M_{15}$ ) to maintain a strict comparative baseline against the control mixture. Additionally, the precise procedures for mixing the mortar, casting the specimens, curing, and running the physical and mechanical tests were explained to ensure all results are reliable and repeatable.

By establishing this clear experimental framework, the chapter provides the necessary background for the next phase of the study. The upcoming chapter will focus on presenting and discussing the test results, focusing on how the partial replacement of cement with thermally activated marble powder affects the physical and mechanical performance of the final mortar mixtures.

# **Chapter IV**

## **Results and Discussions**

### IV.1. Introduction

The primary objective of this chapter is to present, analyze, and discuss the comprehensive experimental results obtained from the laboratory testing of mortars modified with Thermally Activated Marble Powder (TAMP). To evaluate the technical and ecological viability of utilizing processed industrial marble waste as a partial substitution for Ordinary Portland Cement (OPC), a systematic characterization was conducted covering both the fresh state properties and the long-term hardened characteristics of the material. By establishing a direct comparison against a pure cement control mixture, this research seeks to identify the specific performance thresholds, structural changes, and durability boundaries that govern this alternative binder system.

The empirical investigation is structured sequentially to capture the material's life cycle behavior, beginning with its fluid phase and culminating in long-term exposure testing. The first diagnostic phase addresses the fresh state behavior of the binder paste, evaluating the influence of varying TAMP percentages on normal consistency water requirements and hydration kinetics via initial and final Vicat setting times. This is immediately followed by the mechanical performance evaluation, which quantifies the evolutionary compressive strength development at early and standard curing ages of 3, 7, and 28 days. To provide a holistic mechanical profile, this phase also includes the evaluation of 28-day flexural tensile strength alongside a detailed characterization of the physical failure and cracking modes of the broken specimens.

The final phase of the chapter transitions into long-term durability profiling, which is crucial for determining the material's real-world applicability in civil engineering infrastructure. This involves assessing the microstructural permeability and capillary pore network maturity of the specimens through 28-day water absorption by immersion tests. Furthermore, the chemical resilience of the matured matrix is challenged through a 60-day mass loss evaluation under aggressive environmental exposure. By cross-referencing these physical, mechanical, and chemical datasets, this chapter explores the underlying microstructural mechanisms, such as the cement dilution effect, heterogeneous nucleation,

and the physical micro-filler effect, that dictate the performance of sustainable TAMP-modified mortars.

#### **IV.2. Consistency and Setting Times of Fresh Binder Pastes**

The fresh properties of the binder paste were evaluated by measuring the normal consistency and setting times using the Vicat apparatus. The results show a steady trend: as the percentages of thermally activated marble powder (TAMP) replacement increases, the required water-to-binder (W/B) ratio slightly decreases and stabilizes, while both the initial and final setting times experience a marginal acceleration compared to the control (0%).

**Table IV.1 Influence of TAMP substitution on normal consistency (W/B ratio) and Vicat setting times.**

| <b>TAMP Percentage</b> | <b>W/B ratio</b> | <b>Initial setting time (min)</b> | <b>final setting time (min)</b> |
|------------------------|------------------|-----------------------------------|---------------------------------|
| <b>0%</b>              | 0.28             | 158                               | 187                             |
| <b>5%</b>              | 0.27             | 157                               | 187                             |
| <b>10%</b>             | 0.27             | 156                               | 186                             |
| <b>15%</b>             | 0.27             | 156                               | 185                             |

The physical characteristics of the binder in its fresh state are fundamental to determining the workability, placement window, and early structure formation of the modified mortar. The experimental results detailing the standard consistency water-to-binder **W/B** ratio alongside the initial and final Vicat setting times are compiled in **Table IV.1**.

A distinct trend is observed regarding the mixing water requirements. The control pastes **0%** TAMP required a W/B ratio of **0.28** to achieve standard normal consistency. Upon the incorporation of TAMP at **5%**, **10%**, and **15%** substitution levels, the required **W/B** ratio experienced a marginal decrease, stabilizing uniformly at **0.27**. This reduction in water demand is directly supported by the physical characteristics of the processed material, which exhibits a Blaine specific surface area of **1075cm<sup>2</sup>/g**. Because the fineness is lower than that

of conventional Ordinary Portland Cement, the cumulative surface area of the binder system is reduced, minimizing surface water adsorption. Consequently, the coarser, smooth-cleaved TAMP particles act as physical lubricants within the fresh matrix, enhancing paste fluidity without demanding additional water.

Concurrently, the setting kinetics of the paste exhibited a minor acceleration as TAMP content increased. The initial setting time decreased from **158** minutes for the control mix to **156** minutes for the **15%** replacement mix. Similarly, the final setting time shortened from **187** minutes to **185** minutes.

While the dilution of Portland cement typically delays setting times due to the reduction of highly reactive aluminate  $C_3A$  and silicate  $C_3S$  phases, this effect was completely counteracted by the accelerated nucleation mechanism induced by the thermally activated marble powder. Despite having a lower overall bulk Blaine fineness  $1075\text{cm}^2/\text{g}$  compared to conventional cement, the individual surfaces of the processed TAMP grains still offer high surface energy and serve as highly effective heterogeneous nucleation sites. This accelerates the precipitation and early growth of calcium silicate hydrate C-S-H gel networks, leading to a slightly faster transition from a plastic state to a rigid structural matrix.

### IV.3. Compression test

The compressive strength of the different mortar mixtures was evaluated at **3, 7, and 28 days** in accordance with **EN 196-1**. As expected, the results show a clear trend: as the percentages of thermally activated marble powder (TAMP) replacement increases, the compressive strength of the mortar generally decreases compared to the control (0%).

#### IV.3.1. Effect on compressive strength

##### IV.3.1.1. Test at 3-Days

The experimental results for the 3-day compressive strength of the mortar mixtures are compiled in **Table IV.2** and visually illustrated in **Figure IV.1**. As clearly demonstrated by the decreasing height of the bars in the chart, an inverse relationship exists between the TAMP replacement percentage and the early-age compressive strength.

The control mixture (**0% TAMP**) achieved an average compressive strength of **15.46MPa** at **3 days**. With the introduction of **5% TAMP**, the strength experienced a minor reduction of approximately **4%**, yielding an average value of **14.91MPa**. This indicates that low replacement levels do not drastically compromise the early structural integrity of the mortar.

However, as the replacement level increases to **10%** and **15%**, the decline in compressive strength becomes more pronounced. The **10%** replacement mix exhibited a strength of **13.81MPa** (an **11%** decrease compared to the control), while the **15%** replacement mix showed the lowest performance at **11.13MPa**, representing a significant **28%** reduction.

This systematic decrease at **3 days** can be primarily attributed to the dilution effect, where the reduction of cement content limits the initial formation of calcium silicate hydrate (C-S-H) gels responsible for early strength development. Furthermore, while TAMP undergoes thermal activation, its reaction kinetics at very early ages (**3 days**) are slower compared to the rapid hydration of pure Portland cement.

**Table IV.2 Compressive strength at 3 days.**

| <b>Specimen</b> | <b>P (kN)</b> | <b><math>f'_c</math> (MPa)</b> | <b><math>f'_c/f'_{c0}</math></b> |
|-----------------|---------------|--------------------------------|----------------------------------|
| C-3-0-1         | 24.78         | 15.51                          | -                                |
| C-3-0-2         | 24.37         | 15.25                          | -                                |
| C-3-0-3         | 24.94         | 15.61                          | -                                |
| <b>Avg</b>      | <b>24.69</b>  | <b>15.46</b>                   | <b>1</b>                         |
| C-3-5-1         | 26.28         | 16.45                          | -                                |
| C-3-5-2         | 21.36         | 13.37                          | -                                |
| C-3-5-3         | -             | -                              | -                                |
| <b>Avg</b>      | <b>23.82</b>  | <b>14.91</b>                   | <b>0.96</b>                      |
| C-3-10-1        | 23.79         | 14.89                          | -                                |
| C-3-10-2        | 21.54         | 13.48                          | -                                |
| C-3-10-3        | 20.84         | 13.05                          | -                                |

|          |       |       |      |
|----------|-------|-------|------|
| Avg      | 22.06 | 13.81 | 0.89 |
| C-3-15-1 | 18.65 | 11.67 | -    |
| C-3-15-2 | 19.22 | 12.03 | -    |
| C-3-15-3 | 15.50 | 9.70  | -    |
| Avg      | 17.79 | 11.13 | 0.72 |

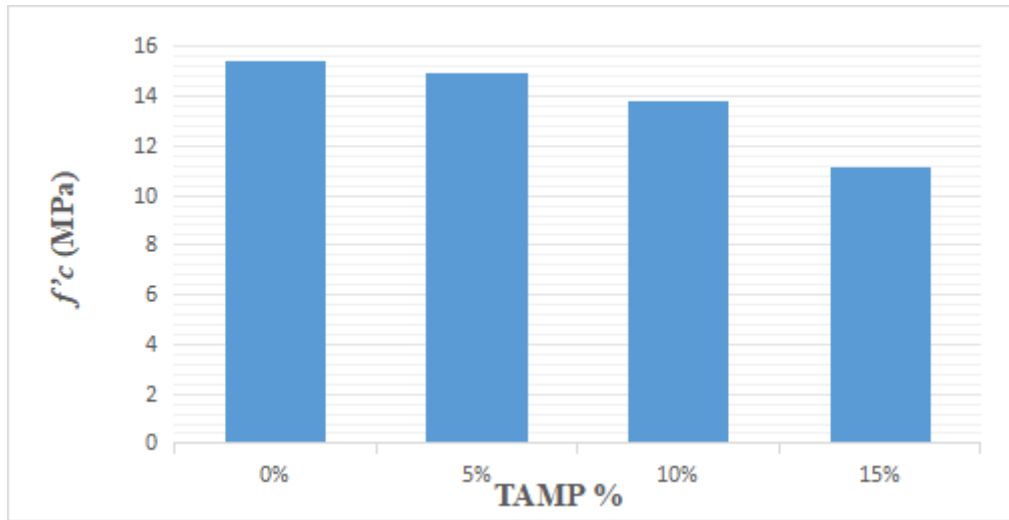


Figure IV.1 Compressive strength at 3 days.

#### IV.3.1.2. Test at 7-Days

The experimental results for the **7-day** compressive strength are presented in **Table IV.3** and visually summarized in **Figure IV.2**. At this curing age, a clear progression in strength development is observed across all mixtures, accompanied by a notable recovery in relative performance compared to the early **3-day** baseline.

The control mixture (**0% TAMP**) achieved an average compressive strength of **27.83MPa**. Remarkably, the mixture with **5% TAMP** replacement exhibited an identical average compressive strength of **27.84MPa** (a relative strength ratio of approximately **1.00**). This demonstrates that at a **5%** substitution level, the initial early-age strength deficit observed at **3 days** is completely mitigated by **day 7**.

For higher substitution rates, the **10%** and **15%** TAMP mixtures reached compressive strengths of **23.77MPa** and **23.17MPa**, respectively. While these values still represent a drop compared to the control, the performance deficit has substantially shrunk. Specifically, the

15% replacement mix improved from a 28% strength loss at 3 days to just a 19% loss at 7 days.

This positive evolution in strength can be attributed to the mid-term activation of the TAMP particles. Beyond acting as a physical filler that accelerates cement hydration via the nucleation effect, the thermally activated marble powder begins to actively participate in the formation of secondary hydration products. This dual mechanism densifies the mortar matrix and partially offsets the cement dilution effect that dominated the 3-day results.

Table IV.3 Compression strength at 7 days.

| Specimen   | P (kN)       | $f'_c$ (MPa) | $f'/f'_{c0}$ |
|------------|--------------|--------------|--------------|
| C-7-0-1    | 45.3         | 28.33        | -            |
| C-7-0-2    | 45.78        | 28.33        | -            |
| C-7-0-3    | 42.33        | 26.49        | -            |
| <b>Avg</b> | <b>44.47</b> | <b>27.83</b> | <b>1</b>     |
| C-7-5-1    | 44.05        | 27.57        | -            |
| C-7-5-2    | 46.36        | 29.01        | -            |
| C-7-5-3    | 43.04        | 26.94        | -            |
| <b>Avg</b> | <b>44.48</b> | <b>27.84</b> | 0.98         |
| C-7-10-1   | 36.85        | 23.06        | -            |
| C-7-10-2   | 38.96        | 24.38        | -            |
| C-7-10-3   | 38.12        | 23.86        | -            |
| <b>Avg</b> | <b>37.98</b> | <b>23.77</b> | 0.84         |
| C-7-15-1   | 37.28        | 23.33        | -            |
| C-7-15-2   | 35.54        | 22.24        | -            |
| C-7-15-3   | 38.23        | 23.93        | -            |

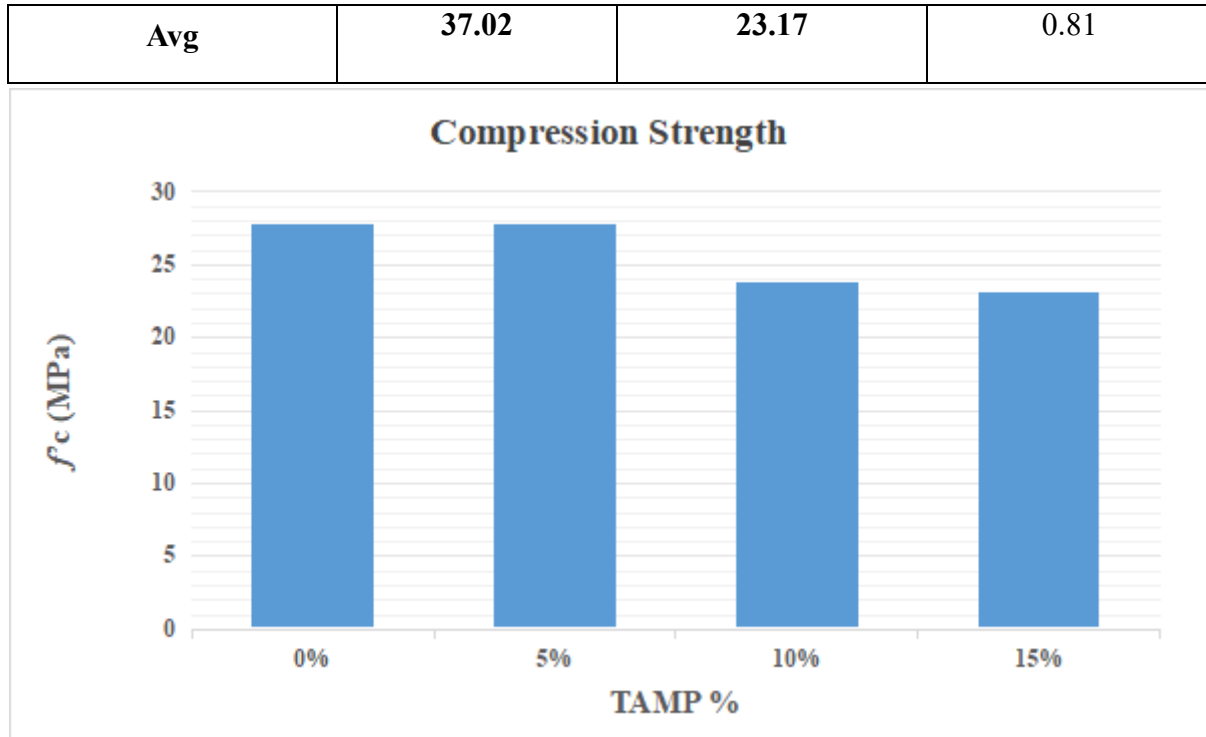


Figure IV.2 Compressive strength at 7 days.

#### IV.3.1.3. Test at 28-Days

The **28-day** mechanical performance evaluation represents the definitive benchmark for assessing the ultimate structural capacity of the modified mortar mixtures. The experimental results and corresponding graphical distributions are detailed in **Table IV.4** and **Figure IV.3**, respectively.

At **28 days** of curing, the control mixture achieved a reference compressive strength of **39.80MPa**. The **5%** TAMP replacement mixture demonstrated highly favorable long-term performance, reaching an average strength of **38.98MPa**. This corresponds to a relative strength ratio of **0.98**, mirroring its **7-day** behavior and confirming that low-level cement replacement with thermally activated marble powder preserves the targeted design strength of the material.

Conversely, the mortars containing **10%** and **15%** TAMP replacement stabilized at lower ultimate values of **32.28MPa** and **31.32MPa**, representing permanent strength drops of **19%** and **21%** relative to the control. The stabilization of these ratios between **day 7** and **day 28**

indicates that the accelerated hydration kinetics provided by the TAMP particles reach their practical limit as the matrix matures.

The long-term deficit at higher replacement levels is fundamentally governed by the dilution effect. Because a significant portion of the binder is substituted, the absolute quantity of Portland cement clinker is reduced, setting a ceiling on the total volume of primary structural hydration products (C-S-H) gel that can be generated. However, achieving strengths exceeding **31MPa** at a **15%** industrial waste replacement level remains highly promising for sustainable, non-structural, or moderately loaded structural engineering applications.

**Table IV.4 Compression strength at 28 days.**

| Specimen   | P (kN)       | $f'_c$ (MPa) | $f'_c/f'_{c0}$ |
|------------|--------------|--------------|----------------|
| C-28-0-1   | 61.8         | 38.68        | -              |
| C-28-0-2   | 66.36        | 41.53        | -              |
| C-28-0-3   | 62.63        | 39.19        | -              |
| <b>Avg</b> | <b>63.59</b> | <b>39.8</b>  | <b>1</b>       |
| C-28-5-1   | 64.29        | 40.23        | -              |
| C-28-5-2   | 61.51        | 38.5         | -              |
| C-28-5-3   | 61.05        | 38.21        | -              |
| <b>Avg</b> | <b>62.28</b> | <b>38.98</b> | <b>0.98</b>    |
| C-28-10-1  | 51.64        | 32.31        | -              |
| C-28-10-2  | 48.94        | 30.63        | -              |
| C-28-10-3  | 54.18        | 33.91        | -              |
| <b>Avg</b> | <b>51.59</b> | <b>32.28</b> | <b>0.81</b>    |
| C-28-15-1  | 50.84        | 31.82        | -              |
| C-28-15-2  | 49.24        | 30.82        | -              |

|           |       |       |      |
|-----------|-------|-------|------|
| C-28-15-3 | -     | -     | -    |
| Avg       | 50.04 | 31.32 | 0.79 |

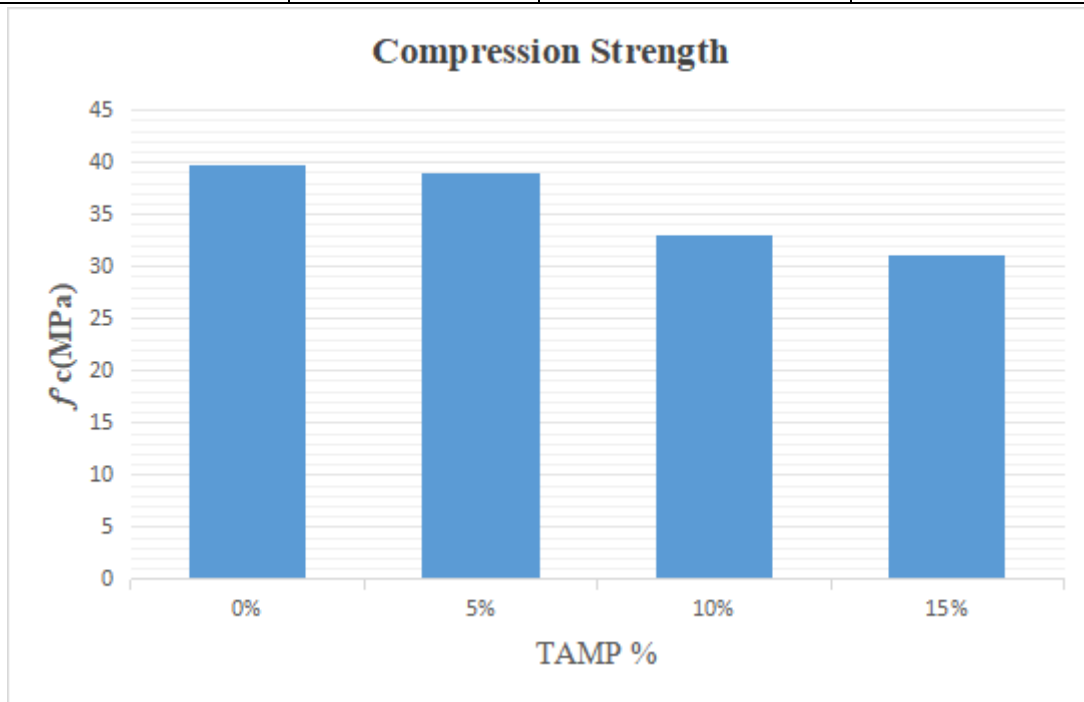


Figure IV.3 Compressive strength at 28 days.

#### IV.3.1.4. Characterization of Compressive Failure Modes

Beyond the quantitative mechanical data, the physical failure modes of the mortar specimens were monitored during testing. For all the mixtures (0%, 5%, 10%, 15%), the specimens exhibited typical semi-explosive failure, resulting in a classic hourglass or double-cone shape, indicating a highly rigid and homogeneous internal matrix.

#### IV.4. Flexural tensile strength

The flexural tensile strength of the different mortar mixtures was evaluated at 28 days in accordance with EN 196-1. Interestingly, the results show a different trend compared to the compression test: as the percentages of thermally activated marble powder (TAMP) replacement increases to 10% and 15%, the flexural tensile strength of the mortar generally increases compared to the control (0%), while a slight drop is observed at 5%.

##### IV.4.1. Analysis and Discussion of 28-Day Flexural Tensile Strength

While compressive strength is the primary indicator of a mortar's load-bearing capacity, flexural tensile strength  $f_t$  is critical for evaluating its resistance to cracking, bending, and environmental stresses. The experimental results and corresponding graphical representations for the 28-day flexural tests are presented in **Table IV.5** and **Figure IV.4**, respectively.

The control mixture (0% TAMP) achieved a baseline flexural strength of 5.612MPa. With a 5% TAMP replacement, a minor decrease to 5.244MPa was observed, representing a 6.6% reduction in bending capacity.

Intriguingly, higher replacement percentages yielded a reversal in this trend, showing an enhancement in tensile performance. At 10% TAMP replacement, the flexural strength increased to 5.830MPa, marking a 3.9% improvement over the pure cement control. This elevated performance was sustained at the 15% replacement level, which reached 5.810MPa (a 3.5% increase compared to the control).

This distinct behavior—where higher TAMP contents compromise compressive strength but optimize flexural strength—can be explained by the physical mechanisms governing tensile resistance in cementitious matrices. Flexural capacity is highly dependent on the quality of the Interfacial Transition Zone (ITZ) between the binder paste and the sand aggregates. The ultra-fine grain size of the ground marble powder exerts a powerful micro-filler effect, packing into micro-voids that cement grains cannot reach. This densifies the ITZ and limits micro-crack propagation under tensile loading. Furthermore, the angular morphology of the processed marble particles may enhance mechanical interlocking within the matrix, contributing to a higher resistance against bending forces despite the lower absolute cement content.

**Table IV.5 Flexural tensile strength at 28 days**

| Specimen | P (kN) | $f_t$ (MPa) | $f_t/f_{t0}$ |
|----------|--------|-------------|--------------|
| T-28-0-1 | 2.326  | 5.454       | -            |
| T-28-0-2 | 2.526  | 5.921       | -            |
| T-28-0-3 | 2.328  | 5.460       | -            |

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|            |              |              |          |
|------------|--------------|--------------|----------|
| <b>Avg</b> | <b>2.393</b> | <b>5.612</b> | <b>1</b> |
| T-28-5-1   | 2.355        | 5.522        | -        |
| T-28-5-2   | 2.168        | 5.085        | -        |
| T-28-5-3   | 2.185        | 5.125        | -        |
| <b>Avg</b> | <b>2.236</b> | <b>5.244</b> | 0.934    |
| T-28-10-1  | 2.532        | 5.938        | -        |
| T-28-10-2  | 2.436        | 5.712        | -        |
| T-28-10-3  | 2.49         | 5.840        | -        |
| <b>Avg</b> | <b>2.486</b> | <b>5.830</b> | 1.039    |
| T-28-15-1  | 2.473        | 5.800        | -        |
| T-28-15-2  | 2.283        | 5.353        | -        |
| T-28-15-3  | 2.677        | 6.277        | -        |
| <b>Avg</b> | <b>2.478</b> | <b>5.810</b> | 1.035    |

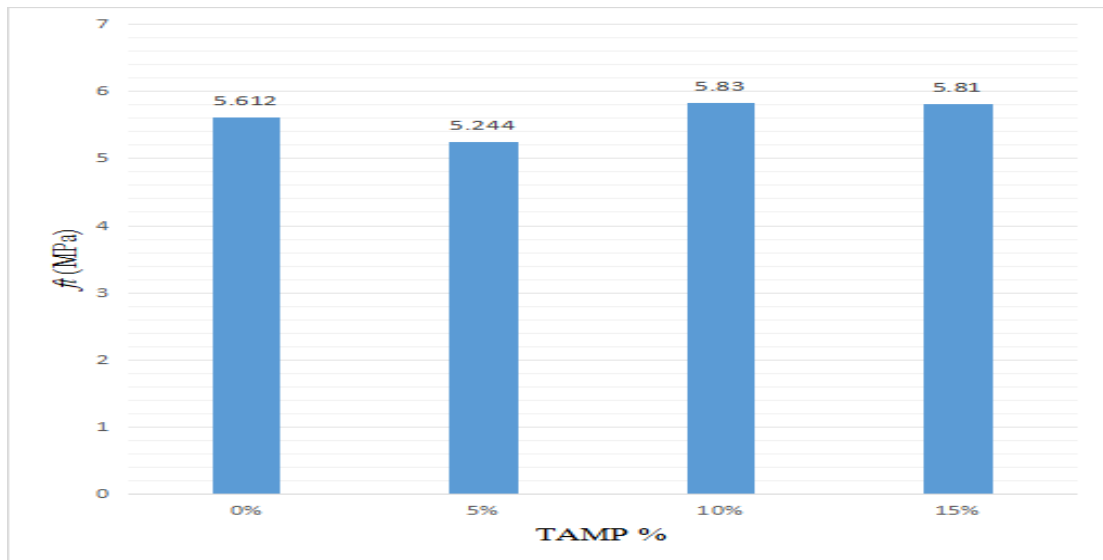


**Figure IV.4 Flexural tensile strength at 28 days.**

### IV.4.2. Characterization of Flexural Failure Modes

Under three-point or four-point bending, all mortar specimens across all replacement levels (0% to 15% TAMP) displayed a classic brittle tensile fracture. The failure was characterized by a single, clean vertical crack propagating upwards from the bottom face of the prism, originating precisely within the middle third span where tensile stresses reach their maximum.

For the 10% and 15% TAMP mixtures, visual inspection of the fractured cross-section revealed a highly dense, uniform surface with no visible air voids or agglomerations. The clean break across the sand aggregates, rather than pulling out around them, visually validates the enhanced aggregate-paste bonding and densified Interfacial Transition Zone (ITZ) attributed to the micro-filler effect of the fine marble powder particles.

### IV.5 Water Absorption by Immersion at 28 Days

Water absorption by immersion is a crucial durability indicator, as it reflects the open porosity and volume of interconnected capillary pores within the hardened mortar matrix. The experimental data illustrating the impact of TAMP substitution on the 28-day water absorption capacity is presented in **Figure IV.5**.

The control mixture (0% TAMP) exhibited the lowest water absorption value at 6.04%. Upon substituting cement with 5% TAMP, the absorption capacity experienced a minor increase to 6.33%. As the TAMP substitution level was further increased to 10% and 15%, the absorption stabilized, dropping marginally to a constant value of 6.27% for both mixtures.

The initial increase in absorption from 6.04% to 6.33% is primarily governed by the cement dilution effect. Because a fraction of the Portland cement clinker is removed, the absolute volume of primary hydration products (C-S-H) gel drops slightly, leaving behind a marginally more porous paste structure.

However, the stabilization and slight drop observed at 10% and 15% substitution levels highlights the progressive efficacy of the micro-filler effect. The ultra-fine particles of the ground, thermally activated marble powder physically segment and segment the capillary pore structure. By filling the interstitial voids between sand particles and cement hydration

products, the TAMP grains disconnect the capillary networks, reducing pore tortuosity and preventing further water ingress. This dual-action balance between the dilution effect and pore-blocking mechanism ensures that higher industrial waste replacements do not critically compromise the durability or permeability characteristics of the developed material.

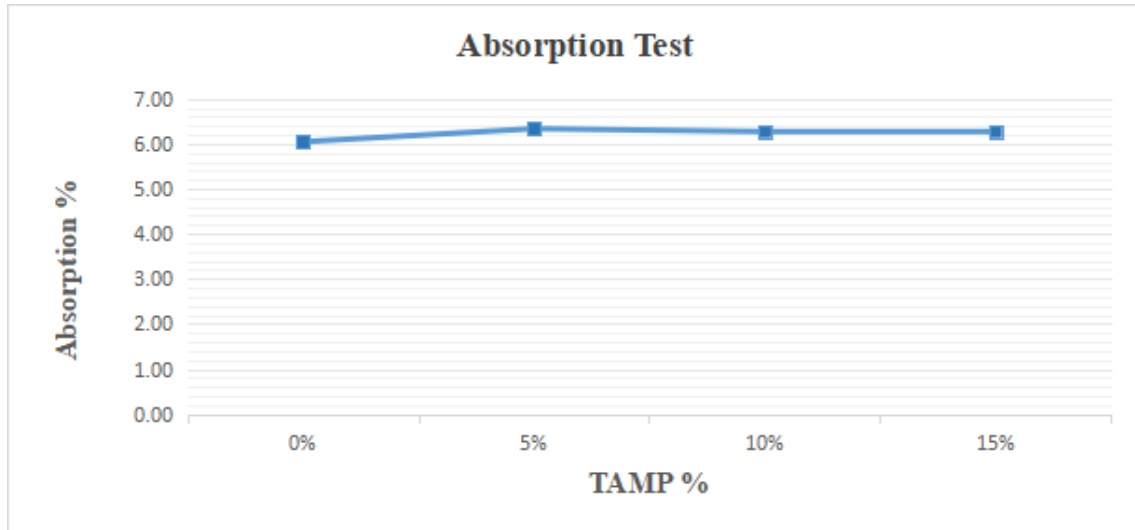


**Figure IV.5 Influence of TAMP replacement percentage on water absorption by immersion at 28 days.**

### IV.6 Durability Evaluation via Weight Loss Over Time

Evaluating the long-term mass loss of cementitious matrices under aggressive exposure conditions provides essential insight into the durability, chemical stability, and environmental resistance of the modified mortars. The experimental progression of cumulative mass loss (%) recorded over a 60-day testing interval is illustrated in Figure IV.6.

The chronological profile of the degradation reveals three distinct kinetic phases common to all mixtures. Phase one, spanning from day 0 to day 15, is characterized by a severe, rapid acceleration in mass loss across all specimens. Following this initial exposure window, phase two exhibits a pronounced stabilization and plateau between days 15 and 45, where the degradation rate decelerates significantly. The final phase, occurring from day 45 to day 60, shows a marginal, uniform decline in the total recorded mass loss percentage,

indicating a mitigation in active leaching or the complete sloughing off of deteriorated surface compounds.

A clear stratified trend emerges regarding the binder composition: the magnitude of material degradation is directly proportional to the TAMP replacement percentage. The control mixture (0% TAMP) demonstrated the highest chemical resilience, maintaining the lowest mass loss profile throughout the 60-day cycle, peaking at approximately 3.3%. The 5% and 10% TAMP variations track closely together, exhibiting a moderate increase in vulnerability. In contrast, the 15% TAMP replacement mixture experienced the most substantial degradation, establishing a clear peak mass loss near 4.5%.

This increased susceptibility to mass loss at higher substitution rates is fundamentally linked to the chemical mineralogy of the alternative binder. The thermally activated marble powder introduces higher concentrations of calcium carbonate  $\text{CaCO}_3$  into the matrix. When subjected to aggressive chemical environments,  $\text{CaCO}_3$  undergoes rapid dissolution compared to the structurally resilient silicate networks of the primary calcium silicate hydrate (C-S-H) gel. Consequently, while the micro-filler effect of TAMP successfully optimizes physical attributes like flexural strength, its chemical composition increases the rate of surface leaching when high replacement dosages (15%) are deployed in aggressive environments.

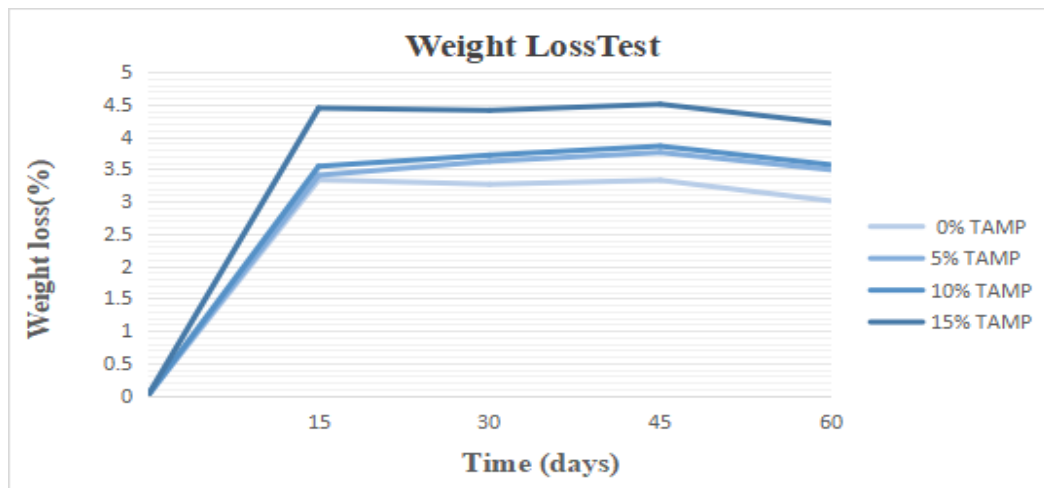


**Figure IV.6 Evolution of mortar mass loss (%) up to 60 days across varying TAMP replacement levels.**

### IV.7. Conclusion

This chapter provided a comprehensive analysis of the fresh, mechanical, and durability attributes of mortar mixtures incorporating Thermally Activated Marble Powder (TAMP) as a partial replacement for cement at dosages up to 15%. Evaluating the binder in its fresh state revealed that the inclusion of TAMP subtly enhances workability characteristics. This behavior is directly supported by the material's bulk Blaine fineness of **1075cm<sup>2</sup>/g**; being physically coarser than standard cement grains, the ground marble reduces the cumulative surface area of the binder matrix, minimizing localized water adsorption. The particle morphology optimized the particle packing of the system, which lowered the water requirement for normal standard consistency from a water-to-binder W/B ratio of **0.28** for the control to a stable **0.27** across all replacement levels. Concurrently, despite the reduction of active clinker phases and the lower bulk surface area, the high surface energy of the individual TAMP grain surfaces offered active sites for heterogeneous nucleation. This mechanism effectively accelerated early-age hydration kinetics, resulting in a marginal shortening of both the initial and final Vicat setting times.

The mechanical characterization highlighted a compelling contrast between the compressive and tensile performance of the modified matrices over time. Under compressive loading, a systematic strength deficit was observed at early ages (**3 days**) due to the dominant clinker dilution effect at higher dosages. However, significant strength development was recorded by **day 7**, where the **5%** TAMP mixture completely recovered its early deficit to match the control strength baseline of **27.84 MPa**. By the standard 28-day benchmark, the **5%** replacement mix maintained a high relative strength ratio of 0.98 (**38.98 MPa**), verifying its structural equivalence. Conversely, the **28-day** flexural tensile strength exhibited a complete trend reversal, with the **10%** and **15%** replacement mixtures outperforming the pure cement control by **3.9%** and **3.5%**, respectively. This specific mechanical enhancement is fundamentally driven by the physical micro-filler effect and particle interlocking of the fine sieved fractions, which significantly densified the Interfacial Transition Zone (ITZ) between the binder paste and the sand aggregates.

In the end, evaluating long-term durability revealed the practical limits and local chemical weaknesses caused by changing the mineral **structure**. Looking at water absorption by immersion, open porosity ticked up slightly at **5%** replacement because the cement was diluted. However, it flattened out completely at **6.27%** for both the 10% and **15%** mixes. This flattening shows that the ultra-fine marble powder physically cuts off, twists, and breaks apart the internal capillary channels, keeping water from soaking in deep. Even so, a **60-day** cycle in harsh environments showed that mass loss scales directly with TAMP content. The **15%** mix took the biggest hit, losing nearly **4.5%** of its mass. This breakdown happens because calcium carbonate  $\text{CaCO}_3$  leaches easily in aggressive conditions, whereas the primary cement hydration products form much tougher, highly stable silicate networks.

Ultimately, while high substitution levels introduce compromises in absolute compressive strength and chemical durability under direct exposure, a **5%** TAMP replacement level emerges as an ideal performance threshold that preserves structural integrity. Furthermore, the notable optimization of flexural tensile capacity and the stabilization of capillary porosity up to a **15%** dosage validate the deployment of thermally activated marble powder as an innovative, environmentally sustainable, and technically viable alternative binder for modern civil engineering applications.

# Conclusion

## Conclusion

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The present study investigated the feasibility of recycling industrial marble waste as an alternative binder reinforcement in cement mortars, with the dual objective of addressing a severe environmental challenge and exploring potential improvements in the performance of cementitious composites. Discarded marble residues and fine sawing slurries are among the most prevalent and disruptive industrial pollutants, with millions of tons dumped annually worldwide. Their unmanaged accumulation, coupled with the threat they pose to groundwater chemistry and soil fertility, makes them an urgent target for innovative waste valorization strategies. Incorporating thermally activated marble powder (TAMP) into mortars represents a promising pathway that combines sustainable waste management with advancements in construction materials.

The experimental program was designed in accordance with national and international standards, ensuring the reliability and comparability of results. A Portland-limestone cement (CEM II/A-L 42.5 N, NA 442) was used to produce control and modified mortar mixtures with substitution rates of 5%, 10%, and 15% by weight. Fresh state properties were checked using standard water consistency and Vicat setting time tests. Compressive strength development was evaluated at 3, 7, and 28 days using prismatic specimens, while flexural tensile behavior was assessed through mechanical bending tests. Long-term durability was monitored via water absorption by total immersion and evaporation mass loss over a 60-day cycle. A systematic nomenclature was adopted to classify specimens based on curing age and TAMP dosage, and results were analyzed with reference to both standard requirements and relevant literature.

The fresh state evaluations demonstrated that blending TAMP explicitly optimizes mortar workability. Because the processed marble powder possesses a lower specific surface area ( $1075 \text{ m}^2/\text{g}$ ) relative to industrial cement, it demands less water to achieve normal consistency, stabilizing the W/B ratio down to 0.27 across all modified formulations. Concurrently, the high surface energy of the calcined marble particles accelerated the process of heterogeneous nucleation, which caused a minor reduction in both initial and final setting times despite the lower active clinker content.

The compressive strength results confirmed the normal development of strength with curing age for all mixtures. The addition of 5% TAMP consistently maintained excellent structural

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performance, retaining 98% of the control mix's strength (38.98 MPa) at the standard 28-day curing mark. At early hydration ages (3 days), higher substitution levels (10% and 15%) introduced an expected mechanical deficit due to the dominance of the dilution effect and slower initial hydration kinetics. However, this gap closed significantly by day 7 as mid-term thermal activation pathways began forming secondary hydration products that reinforced the concrete matrix, stabilizing at structurally viable values above 31 MPa at 28 days.

In contrast to the compressive trends, the flexural tensile capacity results revealed a distinct mechanical upgrade when utilizing higher TAMP dosages. The 10% and 15% modified blends yielded explicit structural increases in 28-day flexural strength, outperforming the pure cement control mix by 3.9% and 3.5%, respectively. This enhanced performance stems directly from the micro-filler effect of the ultra-fine marble dust, which successfully packed into the micro-voids of the cement paste matrix. This physical action densified the Interfacial Transition Zone (ITZ) between the cement paste and aggregate particles, creating superior mechanical interlocking a finding corroborated by the visual inspection of fractured sample faces, which displayed highly uniform, compact structures free from macroscopic air voids.

Evaluating long-term durability revealed the practical limits and local chemical weaknesses caused by altering the binder structure. Looking at water absorption by total immersion, open capillary porosity ticked up slightly at a 5% replacement level due to binder dilution. However, the total absorption rates flattened out and decreased at the 10% and 15% limits to a steady 6.27%. This stabilization shows that the fine marble particles succeed in physically interrupting and disconnecting internal capillary path networks. Even so, a 60-day mass loss exposure under ambient laboratory environments showed that mass degradation scales up as TAMP content increases. The 15% mix experienced the highest degradation, losing nearly 4.5% of its total mass. This breakdown happens because calcium carbonate ( $\text{CaCO}_3$ ) structures leach out easily under aggressive conditions, whereas standard cement hydration products build much tougher, highly stable silicate (C-S-H) networks.

Ultimately, while high substitution levels introduce compromises in absolute compressive strength and chemical durability under direct environmental exposure, a 5% TAMP replacement level emerges as an ideal performance threshold that preserves structural integrity. Furthermore,

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the notable optimization of flexural tensile capacity and the stabilization of capillary porosity up to a 15% dosage validate the deployment of thermally activated marble powder as an innovative, environmentally sustainable, and technically viable alternative binder for modern civil engineering applications. The study thus contributes to the growing body of research on sustainable construction materials, illustrating how innovative recycling strategies can simultaneously improve structural performance and reduce environmental impacts.

### Practical Recommendations

Based on the results obtained, the following recommendations can be made:

The optimum dosage of thermally activated marble powder for structural applications is approximately 5% by weight of cement, which preserves maximum compressive strength while lowering the carbon footprint.

Dosages up to 15% are highly suitable for engineering applications where flexural performance and capillary pore blocking are important, such as paving blocks, precast architectural panels, lightweight rendering mortars, and non-structural building components.

Higher binder dosages (>15%) should be avoided unless alternative thermal activation temperatures or finer grinding cycles are developed, as they tend to aggravate the dilution effect and cause early strength drops.

Before large-scale implementation, further studies should evaluate additional durability properties (such as drying shrinkage, carbonation resistance, freeze-thaw behavior, and sulfate attack resistance) to guarantee long-term performance.

From an environmental perspective, utilizing processed stone residues in construction can be integrated into industrial waste management programs, contributing to waste reduction, lower greenhouse gas emissions, and sustainable construction practices.

By implementing these recommendations, industrial marble recycling in cementitious matrices represents a practical, eco-efficient, and technically viable solution that aligns with the principles of the circular economy and global sustainable development goals.

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