

# The influence of metakaolin on the autogenous and drying shrinkage of internally cured concrete

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**Abstract.** Internal curing might be considered crucial for applications, where High-Performance Concrete (HPC) mixtures are needed, to mitigate or even eliminate the development of autogenous shrinkage and thus the formation of early-age microcracks that diminish the durability of concrete. Lightweight aggregates act as a water reservoir that provides water to unhydrated cement particles when needed, has already been proven an effective curing mechanism. On the other hand, adding supplementary cementitious materials could be a subsidiary strategy to prevent autogenous shrinkage, although in some cases opposite results were reported. Even though metakaolin is well known for its pozzolanic activity and contribution to enhancing concrete's mechanical and durability properties, its effect on autogenous shrinkage needs further investigation. This research examines the individual effects of pumice and metakaolin, as well as their combined influence on autogenous and drying shrinkage. The findings indicate that while metakaolin slightly increases early-age shrinkage, it contributes to a reduction in drying shrinkage at later stages.

## 1 Introduction

High-performance concrete (HPC) is known for its exceptional mechanical and durability properties. Its key characteristics include a dense microstructure, supplementary cementitious materials, and a very low water-to-cement ratio (w/c) [1]. During the early stages of cement hydration, chemical shrinkage occurs, leading to self-desiccation [2]. In the absence of an external water source, cement particles draw water from the partially filled capillary pores, forming menisci in the pore solution. This process generates autogenous stresses and strains, which become more pronounced as the w/c ratio decreases, potentially resulting in microcrack formations due to autogenous shrinkage [3]. Traditional curing methods, such as burlap or membrane curing, are ineffective for HPC, because water can only penetrate to a limited depth due to its dense microstructure. This limitation can be addressed by incorporating pre-wetted porous or physically adsorbed water materials into the concrete mix, which serve as dispersed water reservoirs, supplying water to unhydrated cement particles. This approach, known as internal curing, has been widely studied in the literature [4, 5].

The two most commonly used internal curing agents are lightweight aggregates (LWA) [6] and superabsorbent polymers (SAP) [7]. Both types of materials have been proven effective in reducing or even eliminating autogenous shrinkage. However, factors such as their chemical or physical properties, size distribution and replacement ratio, significantly influence the overall performance of the internally cured mixture. Some studies have reported improvements in

the mechanical and durability properties of concrete, while others have observed opposite results when incorporating internal curing agents of the same type [8].

Pumice is a natural lightweight aggregate formed in volcanic regions and has already proven its effectiveness in lightweight concrete production [9]. Its highly porous structure allows it to store and transport water within the concrete matrix, making it a valuable material for internal curing and yielding promising results. Studies have reported a significant reduction or even elimination of the autogenous shrinkage along with slight improvements in mechanical properties with minimal impact on durability [10, 11]. Additionally, pumice enhances concrete microstructure by refining the pore structure and improving the interfacial transition zone between pumice and cement paste [12].

Metakaolin is a supplementary cementitious material (SCM) with a well-established positive influence on both the mechanical and durability properties of concrete [13]. However, its effect on shrinkage-related properties requires further investigation, as discrepancies among different studies have been reported and additional justification is needed. Weng et al. [14] investigated the influence of metakaolin, among other SCMs, on shrinkage behavior and found that the partial replacement of cement with metakaolin led to an increase in autogenous shrinkage. This was attributed to its higher specific surface area, which contributes to a denser, more refined microstructure, that may hinder the water movement between the matrix and the curing environment. On the other hand, Gleize et al. [15] demonstrated that the

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reduction in autogenous shrinkage was mainly linked to the pozzolanic activity of metakaolin and not to the reduction of the cement content, although further justification is required. Brooks et al. [16] also found that early-age autogenous shrinkage decreased at higher replacement levels.

Regarding drying shrinkage, the majority of research studies conclude that metakaolin reduces drying shrinkage, due to the denser microstructure it creates as a result of its pozzolanic activity, which limits water migration from the internal pores to the environment [17, 18, 19, 20]. However, some studies have reported an adverse effect of metakaolin on drying shrinkage, possibly linked to the higher content of C-S-H gels [21], the increased porosity from the incorporation of metakaolin [22] and the redistribution of water between cement and metakaolin [23]. Despite the importance of restrained shrinkage, which better represents real structural behavior, only limited research has been conducted, which reported a slightly negative effect of metakaolin [24, 25].

This study examines the individual performances of fine pumice, as an internal curing agent, and metakaolin in terms of mechanical and shrinkage-related properties by replacing normal-weight limestone sand and cement, respectively. It also provides useful insights into the combined performance of these two materials in achieving internal curing.

## 2 Experimental

### 2.1 Materials

Portland cement CEM I 52,5R was used in this study. Crushed limestone aggregates with nominal grades of 0-4 mm, 4-8 mm and 8-16 mm, having a specific gravity of 2.7 kg/m<sup>3</sup> were also used. Additionally, metakaolin, provided by Imerys S.A., was incorporated as a supplementary cementitious material. Furthermore, pumice sand with a nominal grade of 0-3 mm, which originates from Yali Island, Greece, was also used as the internal curing agent. The specific gravity and the water absorption of pumice sand are 1.7 kg/m<sup>3</sup> and 16%, respectively. Finally, a polycarboxylate-based superplasticizer was used in all mixtures.

### 2.2 Mix design

Four concrete mixtures were prepared with a constant water-to-binder (w/b) ratio of 0.27. All mixtures consist

of high amount of cement and limited water to make the effects of autogenous shrinkage more pronounced. An extra amount of water was incorporated into the mixture via the pumice aggregates to mitigate the autogenous shrinkage. The required amount of water and consequently the amount of pumice (PU) that partly replaces the conventional limestone sand (LS), was determined using the following equation, according to ASTM C 1761 [26]:

$$M_{PU} = \frac{C_f C_S a_{max}}{SW_{PU}} \quad (1)$$

where  $M_{PU}$  is the required mass of oven-dry pumice,  $C_f$  is the cementitious materials content,  $C_S$  is the chemical shrinkage of cementitious materials,  $a_{max}$  is the maximum potential degree of hydration,  $S$  is the degree of saturation of pumice and  $W_{PU}$  is the mass of the released water from pumice at a relative humidity of 94% expressed as a fraction of the oven dry mass. To reach the maximum degree of saturation, pumice sand was pre-wetted in part of the mixing water inside a sealed container for three days before mixing.

To study the effect of metakaolin on both the reference, and prone to autogenous shrinkage, mixture (AS) and the internally cured mixture (IC), two additional mixtures (ASM, ICM) were designed. In these mixtures, metakaolin replaced 10% of the cement by weight, allowing for a comprehensive analysis of its influence on shrinkage behaviour. The detailed proportions of the mixtures are presented in Table 1.

### 2.3 Test methods

After mixing, the fresh properties of the mixtures were determined. Slump, wet density and entrapped air content were measured according to EN 12350-2 [27], EN 12350-6 [28] and EN 12350-7 [29], respectively.

Compressive strength tests were conducted according to EN 12390-3 [30] using cubic specimens with an edge length of 100 mm. After casting, the specimens were covered with wet burlap for 24 hours until demolding. They were then sealed with a plastic membrane to prevent any loss of the internal moisture of the concrete matrix to the environment and they were stored in a controlled temperature chamber ( $20 \pm 1^\circ \text{C}$ ). The compressive strength was measured at 7, 28 and 90 days after mixing using three specimens for each day.

The determination of drying shrinkage was carried out according to ASTM C157 [31]. For each mixture,

**Table 1.** Mix proportions (kg/m<sup>3</sup>)

Mixture	Cement	Metakaolin	Water	LS 0-4	LS 4-8	LS 8-16	PU	Superplasticizer
AS	600	-	162	890	270	630	-	5.04
ASM	540	60	162	890	270	630	-	6.42
IC	600	-	162	568	270	630	203	5.16
ICM	540	60	162	568	270	630	203	6.12

three prismatic specimens with dimensions 100 x 100 x 500 mm<sup>3</sup> were prepared, with two metal heads attached at the ends. After casting, the specimens were covered with wet burlap and left for 24 hours after which they were removed from the molds. Immediately after demolding, their length was measured using a dial indicator. At the same time, the mass of the specimens was also recorded, and these values are considered as the initial reference length and mass values of the specimens, respectively. The specimens were then stored in a room at a constant temperature of 20 ± 2 °C and relative humidity of 50 ± 5%. Length and mass measurements were repeated at 3, 7, 14, 21, 28, and 56 days after mixing.

The behaviour of the different mixtures regarding the autogenous and drying shrinkage was studied according to ASTM C1581-04 [32]. For each mixture, two ring specimens were produced. The ring moulds comprise an inner steel ring with an outer diameter of 330 ± 3 mm, a thickness of 13 mm and a height of 150 ± 6 mm along with an outer steel ring with an internal diameter of 405 ± 5 mm and the same height. Prior to casting, both rings were fixed on a frictionless, non-absorptive polyurethane sheet base. Immediately after mixing, the ring was filled with concrete without applying any compaction as the mixtures exhibited high flowability. The rings were then covered with wet burlap to avoid any loss of moisture and both rings were detached from the base to allow free movement. After 24 hours, the outer ring was removed, and the top surface was sealed with impermeable tape to prevent moisture loss from the top surface. The ring specimens were stored in a room with a constant temperature of 20 ± 2 °C and relative humidity of 50 ± 5% until the first crack formation. Four strain gauges, that were attached to the inner surface of the inner ring, recorded the strain due to the shrinkage of the concrete specimen right after its casting. The experimental setup for this test procedure is illustrated in Figure 1.



**Figure 1.** Specimen arrangement for the restrained ring test.

### 3 Results and discussion

#### 3.1 Fresh properties

The slump, wet density and entrapped air content results are presented in Table 2. All mixtures exhibit good rheological behaviour despite their low water-to-binder ratio. Mixtures with metakaolin demanded more superplasticizer to reach the same slump values. This slight reduction in workability is attributed to metakaolin's higher specific surface and the irregular shape of its particles [33]. The incorporation of pumice did not affect the workability, as it was pre-wetted before mixing, preventing it from absorbing or releasing additional water. Minor variations in wet density are due to differences in the specific gravity of metakaolin and pumice sand compared to cement and limestone sand, respectively. The entrapped air content remains consistent at low levels across all mixtures, even though no compaction was applied.

**Table 2.** Fresh properties of concrete mixtures.

Test	AS	AS M	IC	ICM
Slump(mm)	190	200	>220	>220
Wet density(kg/m <sup>3</sup> )	2425	2406	2338	2314
Air content (%)	2.8	3.2	2.6	3.3

#### 3.2 Compressive strength

The compressive strength results are presented in Figure 2. First, all mixtures can be classified as high-performance concrete, as they exceeded 80 MPa in strength within the first seven days. By 28 days, their strength approaches or, in some cases, surpasses 100 MPa. The partial replacement of cement with 10% metakaolin appears to have no significant effect at early ages, as both metakaolin-modified and unmodified mixtures exhibit similar strength levels at seven days. However, by 28 days, the pozzolanic activity of metakaolin becomes more pronounced, leading to a 10% strength increase in the reference mixtures and a 6% increase for the internally cured mixtures. Beyond 28 days, the pozzolanic effects appear to moderate, with strength gains between reference and internally cured mixtures settling at 8% and 3%, respectively. Additionally, metakaolin contributed to a 17% strength increase between the 7-day and 28-day marks, compared to an 8% increase in reference mixtures without metakaolin. For internally cured mixtures, these figures were even higher, with strength gains of 20% and 13%, respectively.

The comparison between the reference mixtures and those with internal curing is particularly interesting. At seven days, a slight reduction in strength of 4–6% is observed when conventional limestone sand is replaced with fine pumice. This decrease is expected as pumice, like other natural LWA, has lower strength than normal-weight aggregates. However, this initial strength reduction tends to diminish over time and, in some cases, even reverses. This phenomenon is also reported by Zhutovsky et al. [10]. It appears that the prevention

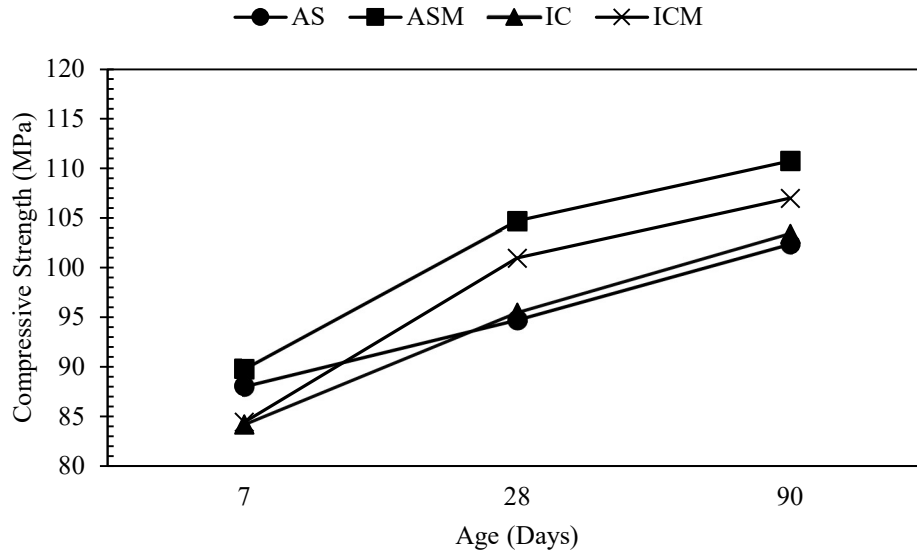


Figure 2. Compressive strength of concrete mixtures at different ages.

of autogenous shrinkage and subsequent microcracking by internal curing helps counterbalance the negative impact of pumice's lower strength. Another key factor contributing to the higher strength development rate in internally cured mixtures is the extended hydration of cement. This is facilitated by the additional water released from the pumice pores during curing, enhancing cement hydration and overall strength development [3]. This positive behavior of fine pumice as an internal curing agent was also reported by Lura et al. [34]. Additionally, the selection of fine pumice, which acts as smaller and better-distributed water reservoirs within the matrix, contributes to the good mechanical response of the internally cured mixtures [35]. With the combination of metakaolin and pumice, it was possible to achieve an internally cured mixture with compressive strength at 28 days, by 7 % higher compared to the reference mixture.

### 3.3 Drying shrinkage and mass loss

The drying shrinkage and percentage mass change of the specimens are presented in Figures 3 and 4, respectively. Since measurements started 24 hours after mixing, the results concern mainly the drying and not the autogenous shrinkage [36]. As early as the first three days, a 50% increase in strain is observed with the addition of pumice. This variation fluctuates over time but remains within the 20-40% range. The increase in drying shrinkage due to the incorporation of fine pumice has also been reported by Costa et al. [37] and Zhutovsky et al. [38], who replaced part of the conventional aggregates with lightweight expanded clay

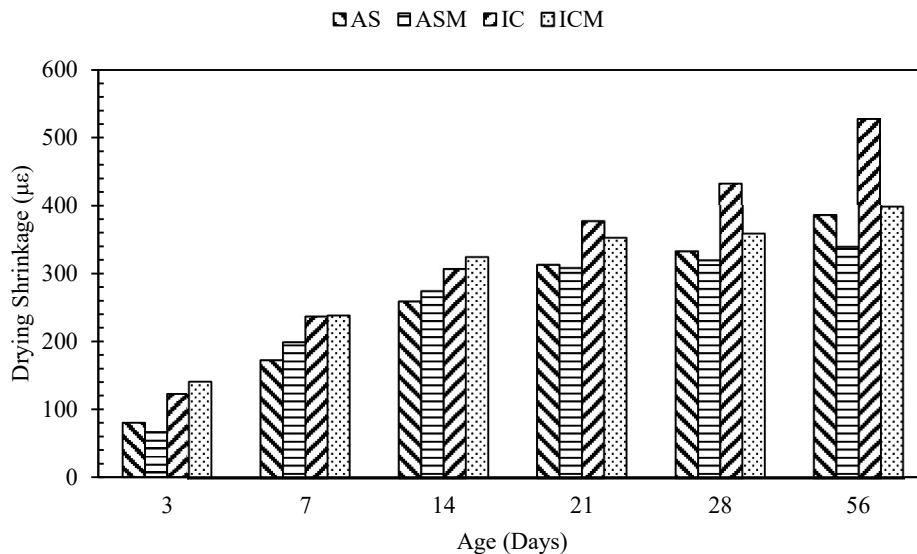
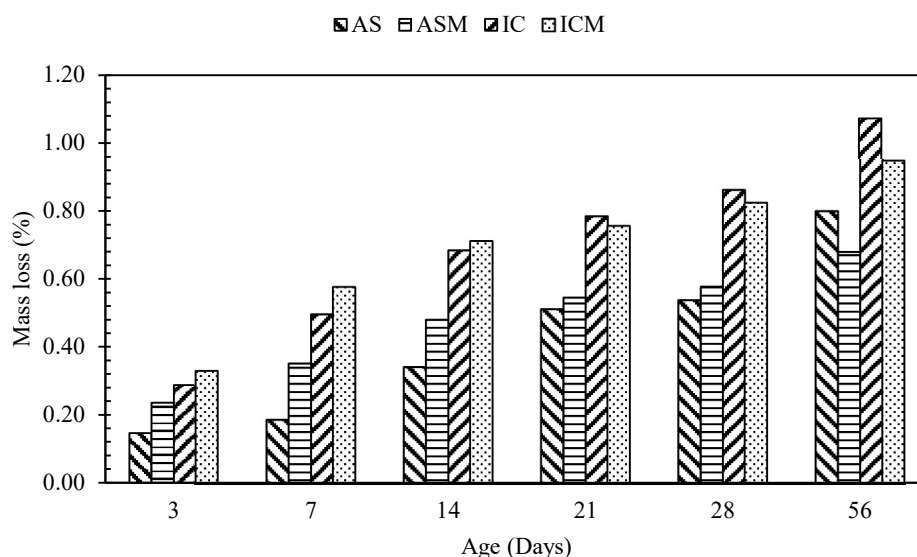


Figure 3. Drying shrinkage of the different concrete mixtures at various ages.



**Figure 2.** Mass loss percentage of the different concrete mixtures at various ages.

and pumice, respectively. These studies attributed the increase in shrinkage to the lower stiffness of the LWA, which imposes less restraint on the tendency of the cement paste to shrink during drying. However, other studies found that partially replacing conventional aggregates with pre-wetted LWA can reduce drying shrinkage [39]. This difference may be due to that these studies let specimens to be cured in moist environments for seven days before starting strain measurements. Nevertheless, the increase in shrinkage is offset by the benefits of internal curing and it should not be considered a limiting factor for the utilization of the pumice.

Regarding the effect of metakaolin on drying shrinkage, results indicate no impact during the first three weeks, as strain values remain nearly identical between mixtures with and without metakaolin. Afterward, it seems that metakaolin tends to reduce the rate at which drying shrinkage increases over time. Some studies have reported an increase in drying shrinkage due to the incorporation of metakaolin, attributing it to the redistribution of water within the concrete matrix [20]. The current results are in accordance with the similar findings in the literature [19]. Güneysi et al [20] concluded that the pozzolanic reactions occur before 28 days and the formed products enhance concrete microstructure, by refining the pore structure and decreasing the capillary. This enhanced microstructure impedes water migration from capillary pores to the environment. The present study supports this theory, as the reduction in strain rate begins after three weeks, when sufficient calcium hydroxide has been produced by cement hydration to initiate pozzolanic reactions. Furthermore, strain rate reduction is more profound in the internally cured mixtures, which can be attributed to the higher calcium hydroxide concentration resulting from the enhanced hydration of unhydrated cement particles and internal curing water.

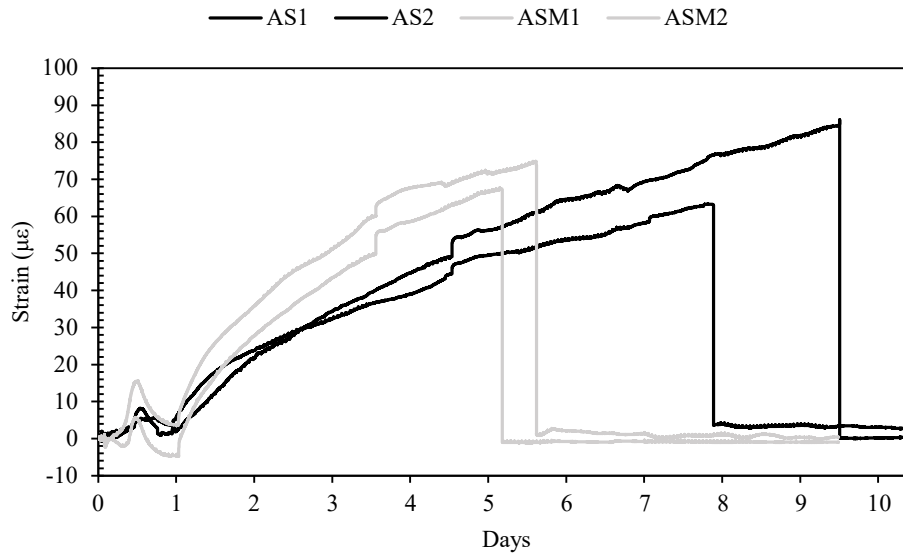
In terms of drying shrinkage, metakaolin and fine pumice appear to complement each other well. By 28 days, the internally cured mixture exhibits drying shrinkage values similar to the reference mixture,

indicating that the negative impact of pumice on drying shrinkage can be mitigated by the incorporation of metakaolin. The mass loss results follow a similar trend to drying shrinkage. It is also observed that the water loss of the mixtures with metakaolin is reduced after three weeks, due to the densification of the microstructure.

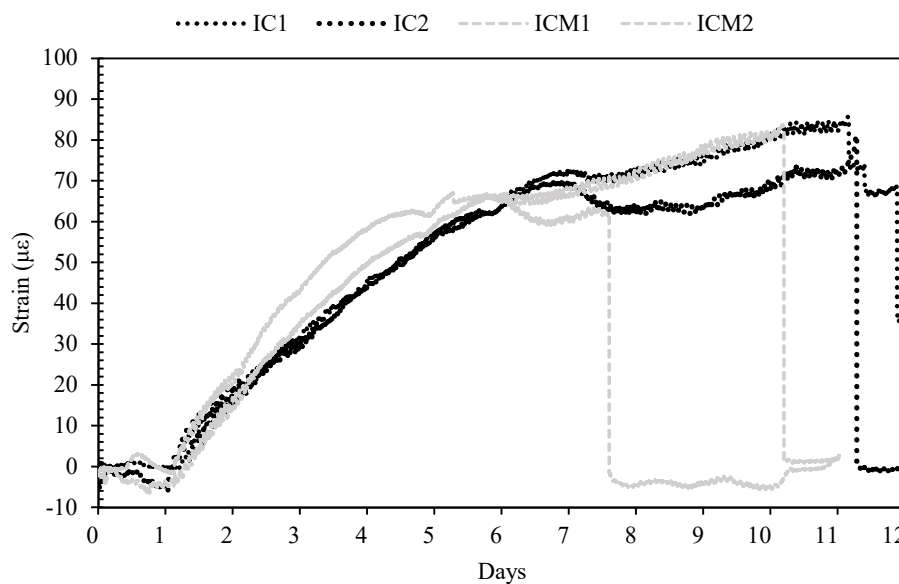
### 3.4 Restrained shrinkage

Concrete begins to shrink immediately after casting. As it contracts, tensile stresses develop due to the restraint imposed by the inner steel ring, potentially leading to its deformation. The greater the shrinkage effects, the higher the tensile stresses in the concrete, as the steel ring restricts free deformation. If these tensile stresses exceed the concrete's tensile strength, cracking occurs, causing the specimen to lose its confinement effect on the ring, resulting in an instantaneous reduction in recorded strain.

Figures 5 and 6 illustrate the strain evolution of the inner steel ring for the reference and the internal curing mixtures, respectively. Curves with the same colour and line style correspond to the two specimens of each composition. The deformations that develop are attributed to chemical, autogenous, and drying shrinkage. In reference mixtures, a slight peak in deformation is observed during the first day of monitoring, whereas this peak is significantly reduced or even absent in the internally cured mixtures. This observation indicates the effectiveness of the internal curing mechanism in mitigating autogenous shrinkage, since autogenous shrinkage effects tend to peak during the first 24 hours of curing, while their rate significantly drops, afterward. After the first day, when the outer ring is removed, drying shrinkage becomes the dominant factor influencing deformation, which steadily increases, until cracking occurs. Cracking happens instantaneously, resulting in a vertical through-crack of considerable width. The most significant contribution of the utilization of pre-wetted pumice concerns the delay of the cracking time. This cracking delay is around 30%



**Figure 5.** Restrainted shrinkage of the reference mixtures without pumice.



**Figure 6.** Restrainted shrinkage of the internally cured mixtures with pumice.

for mixtures without metakaolin and around 50% for mixtures containing metakaolin, similar to findings in previous studies on internal curing using LWA [40, 41]. After the first 24 hours, a slight reduction in the strain development rate is observed in internally cured mixtures. Regarding the maximum strain reached before cracking, no significant differences were found among mixtures, aligning with the similar compressive strength values obtained.

On the other hand, metakaolin seems to have an adverse effect regarding restrained shrinkage. The incorporation of metakaolin leads to a decreased cracking time for the reference and the internal curing mixtures with approximate values of 25% and 13%, respectively. At the same time, an increase of 25% in strain development rate after the first 24 hours is reported for both the reference and the internal curing mixtures. While metakaolin improves drying shrinkage resistance, as previously discussed, it leads to higher shrinkage when concrete is subjected to drying under

restrained conditions. Limited related studies report similar results, but experimental justification remains undocumented [42, 25]. Consistent with previous experimental results, the combination of pumice and metakaolin exhibits better performance in terms of cracking latency than the reference mixture, even though the optimum mixture, in this case, is the one containing only pumice.

## 4 Conclusions

This study investigates the internal curing properties of different concrete mixtures using pumice as an internal curing agent and focuses on the effect of metakaolin on shrinkage related properties and its contribution to the internal curing. Among standard fresh and hardened properties tests, drying and restrained shrinkage tests were conducted. The following conclusions were drawn from the results obtained:

1. Since pumice was already pre-wetted before adding into the mixture, it did not affect the rheology. Metakaolin on the other hand increased the demand for superplasticizer.
2. The replacement of limestone with pumice sand led to a slight decrease in compressive strength at early ages. However, this decrease was eliminated after 28 days. Metakaolin enhanced the compressive strength of the mixtures, thanks to its pozzolanic activity.
3. Pumice increased drying shrinkage, particularly at early ages. However, metakaolin had no impact during the first three weeks and later significantly reduced the shrinkage development rate. Thus, internally cured concrete can be designed without adverse effects on drying shrinkage by incorporating metakaolin.
4. Internal curing proved effective in limiting the strain during the first day of the restrained shrinkage test due to autogenous shrinkage. It was also able to prolong the cracking time. However, metakaolin was found to slightly accelerate this time. Yet mixture with pumice and metakaolin still exhibited better performance than the reference mixture.
5. The fact that metakaolin reduced the time to crack even though it exhibited lower drying shrinkage needs further research.
6. Metakaolin can be effectively utilized in internally cured concrete to counteract the negative effects of LWA used as internal curing agents.

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