

EFFECTS OF CO₂ PENETRATION ON THE PROPERTIES OF SELF-COMPACTING MORTARS WITH HIGH CONTENTS OF SUPPLEMENTARY CEMENTITIOUS MATERIALS

Data de submissão: 11/06/2024

Data de aceite: 01/07/2024

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ABSTRACT: This article evaluates the influence of accelerated carbonation on mortars with reduced amounts of cement intended for self-compacting concrete (SCC). To this end, cement was replaced by different types of supplementary cementitious materials (SCMs), including metakaolin (MK), rice husk ash (RHA) and sugar cane biomass ash (SCBA). In addition, hydrated lime (HL) was added to some mixtures to replenish the alkaline content of the system. The effects of the carbonation of the pastes with SCMs and with the addition of HL were observed using X-ray diffraction (XRD), thermogravimetry (TG) analyses and compressive strength tests. HL optimized the alkaline reserve together with MK, but did not interact well with SBCA, increasing the depth of carbonation. However, the pastes with the SCMs showed carbonation depths below the normative standards. The XRD results revealed preferential carbonation of calcium hydroxide compared to secondary C-S-H. In terms of compressive strength, the pastes with SCMs and with and without the addition of HL obtained lower results, which, however, does not make the use of these materials unfeasible since the loss of strength was not significant. The results show that the use of high contents of SCMs

and the addition of HL are important to obtain self-compacting pastes that are more resistant to CO₂ penetration, which will help to obtain more durable constructions with a lower carbon footprint.

KEYWORDS: Cleaner production. Resistance to CO₂ penetration. Sustainability. Mineral admixtures.

INTRODUCTION

The construction industry is a major consumer of natural resources, from the extraction of aggregates (sand and gravel) to the production of Portland cement, which contributes negatively to greenhouse gas emissions (Perlot et al., 2013; Mikulčić et al., 2016), accounting for over 60% of global production (Scrivener et al., 2018).

One way to reduce these impacts is to use supplementary cementitious materials (SCMs) or also called mineral admixtures, as a partial replacement for cement. When used in adequate quantities, SCMs provide improved workability, mechanical performance and durability, as well as economic benefits and reduced environmental pollution (Uysal; Yumas, 2011).

SCMs can be inert or pozzolanic materials, but each type has different effects on the properties of mortars and concretes in the fresh and hardened state and on their durability, with their advantages and disadvantages. Their advantages can be exploited by combining various SCMs, thus increasing the level of cement replacement (Le; Ludwig, 2016).

Pozzolanic materials contribute to improving binding properties, given their ability to react with the calcium hydroxide released during cement hydration and consequently produce additional hydrates that are responsible for developing mechanical strength. Nevertheless, these materials can contribute to the quality of the cement product as they help to improve the compactness of the system, which results in increased durability as porosity is reduced and attack by external agents is made more difficult (Apaza Medina, 2011).

In relation to attack by external agents, carbonation stands out because it is a common phenomenon in concrete structures. Carbonation occurs through the penetration of carbon dioxide (CO₂), present in the atmosphere in gaseous form, into the pores of concrete (Ollivier; Vichot, 2014), resulting in a reduction in the durability of reinforced concrete.

The carbonation reaction is not detrimental to the mechanical properties of common Portland cement-based materials, but it does cause a reduction in alkalinity which can lead to chemical degradation in relation to the structure of reinforced concrete steel bars (Qiu, 2020). This is due to the reaction of CO₂ with the cement's hydrated alkaline compounds, reducing the internal pH of the cement matrix.

In the alkaline environment, a protective film forms on the steel that prevents it from oxidizing. The reinforcement of reinforced concrete is said to be in a passive state. When CO₂ penetrates inside the concrete pores, contact with water produces carbonic acid

(H_2CO_3). When this carbonic acid reacts with the calcium hydroxide present in the concrete, it produces calcium carbonate (CaCO_3).

In view of the aspects related to the durability of concrete and, in particular, the effects of carbonation, this work aims to evaluate the effects of replacing Portland cement with various types of SCMs such as sugarcane bagasse ash (SCBA), rice husk ash (RHA) and metakaolin (MK) on the resistance to carbonation of mortars for self-compacting concrete. In addition, the effects of using hydrated lime (HL) (or calcium hydroxide) were also investigated.

STATE OF-THE-ART

Currently, there are several types of SCMs capable of replacing Portland cement and improving the properties of materials, including fly ash (FA), limestone filler (LF), slag and fine bagasse ash (Dossche et al., 2016). Among the SCMs obtained from ashes, sugar cane biomass ash (SBCA) and rice husk ash (RHA) stand out, as they are materials that contribute to improving viscosity and stability in the fresh state (Sua-lam; Makul, 2017).

When RHA is used in mortars, the compressive strength increases significantly due to pozzolanic reactions and the filler effect. However, the pozzolanic reaction of RHA only slightly increases the compressive strength during the early ages, i.e. at 7 and 14 days of hydration. Normally, reactions involving RHA take place significantly after 90 days of hydration (Jamil et al., 2016). The pozzolanic reaction of RHA depends on particle size, age and cement replacement percentages. The pozzolanic reaction of RHA depends on particle size, age and cement replacement percentages.

This ash has a high silica content (SiO_2), which makes it a supplementary source for use as a mineral admixture or pozzolan for the production of cementitious materials (Anjos, 2009). MK can also be used as an SCMs, as it consists of amorphous silica and alumina (Al_2O_3) and is capable of reacting with calcium hydroxide ($\text{Ca}(\text{OH})_2$) to produce new hydrated compounds such as C-S-H.

Anjos et al. (2020) produced self-compacting concrete with cement consumption of only 120 kg/m^3 and 200 kg/m^3 , with mineral admixtures of MK and FA. X-ray diffraction (XRD) showed that the main hydrated products formed were gismondin and C-S-H, and portlandite was completely consumed in some mixtures, except for those with fly ash and hydrated lime (HL).

SCMs can increase strength and electrical resistivity, however, due to their pozzolanicity, they consume calcium hydroxide, reducing the pH of the concrete and making the steel more susceptible to corrosion. Carbonation is a concrete deterioration process. Its depth of penetration is normally used to estimate the useful life of concrete. Among the most important factors that can significantly affect the carbonation resistance of concrete are the relative humidity (RH) and the water cement ratio (Elsalamawy et al., 2019).

The compressive strength of the concretes for all mix proportions increased with curing age and decreased with increasing SCBA replacement. The reduction in compressive strength at 10%, 20% and 30% cement replacement with SCBA compared to the reference concrete was 16.8%, 25% and 35.5%, respectively (Joshaghani; Moeini, 2017).

Safiuddin et al. (2010) used cement substitutions with RHA of up to 30%. The result of their study showed that increasing the content of RHA in the mixture considerably increased (by more than 10%) the compressive strength, and to a lesser extent the ultrasonic pulse velocity.

For Kannan (2018), the compressive strength at 28 and 90 days in self-consolidating concretes (SCC) with 5% to 30% metakaolin and obtained the best result with 15% replacement. He also studied the interaction with RHA and found that the ternary mixture with up to 40% replacement showed good results.

EXPERIMENTAL PROGRAM

Materials

In this research, a high early strength Portland Cement (CPV-ARI), equivalent to OPC Type III according to ASTM C150 and CEM I according to EN 197-1, was used. This cement was chosen due to its lack of pozzolanic materials, which facilitates the analysis of the synergistic effects of the different components in the production of mortars. Additionally, high purity hydrated lime (CH-I type) was used as a complementary binder to maintain the system's alkalinity and enhance the reactivity of the supplementary cementitious materials (SCMs). Natural river sand with a maximum diameter of 2.40 mm, a fineness modulus of 2.20 and a specific gravity of 2.65 kg/dm³ was used as fine aggregate.

Three types of SCMs were used to produce the mortars:

- Sugarcane bagasse ash (SCBA): this ash comes from burning sugarcane bagasse in the municipality of Arez (RN-Brazil). Before use, the SCBA was dried in an oven at 100 °C for 24 hours and then subjected to a particle size reduction process using a ball mill for 3 hours.
- Rice husk ash (RHA): derived from burning rice husks to generate energy at a factory in Alegrete (RS-Brazil). The combustion temperature used to produce RHA was approximately 700°C.
- Metakaolin (MK): commercial product supplied by Metafort (Ipojuca - Pernambuco, Brazil), orange in color and moderately reactive. A 20% MK content was used in all the pastes in order to maintain mechanical strength and further reduce the cement content.

The specific surface area (SSA) of the Portland cement and the SCMs was determined using the Brunauer, Emmett, and Teller (BET) method. For this test, one gram of each material was taken and dried in an oven. The results are presented in Table 1.

Materials	SSA (m ² /g)
Cement	1.70
SCBA	54.26
RHA	16.04
MK	16.11
HL	10.64

Table 1. Specific surface area (SSA) of cement, hydrated lime and SCMs

The crystalline phases of the materials were identified by X-ray diffraction (XRD) using a diffractometer with CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) operating at an applied voltage of 45 kV, a power of 1000 W and a current of 25 mA. Measurements were carried out over a range of 5-70° 2 θ , with a step time of 0.5 s and a nitrogen filter. The crystalline phases were identified using High Score Plus software and the results can be seen in Fig. 1.

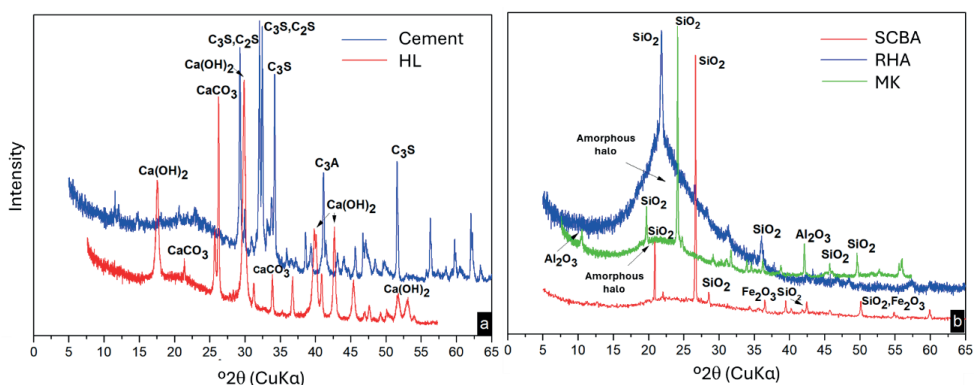


Figure 1. X-ray diffraction patterns of (a) cement, hydrated lime and (b) SCMs.

The chemical composition of the materials in terms of the main oxides was determined by X-ray fluorescence (XRF) and the results can be seen in Table 2.

Main Oxides (%)	CEM	HL	SBCA	RHA	MK
CaO	61.39	97.20	2.47	0.94	0.45
SiO ₂	19.13	1.32	60.04	92.42	53.92
Fe ₂ O ₃	3.65	0.47	25.78	0.09	9.90
SO ₃	3.26	0.13	0.34	0.13	0.22
Al ₂ O ₃	3.99	0.40	3.08	2.01	30.34
MgO	3.15	0.00	1.02	0.00	0.00
K ₂ O	0.52	0.40	3.39	0.00	2.25
Others	4.91	0.08	3.88	4.40	2.92

Table 2. Chemical oxides (wt. %) of cement (CEM), hydrated lime and SCMs

Fig. 2 shows the results of the reactivity test of the SCMs using the direct compressive strength method determined by ABNT NBR 5751 (2015).

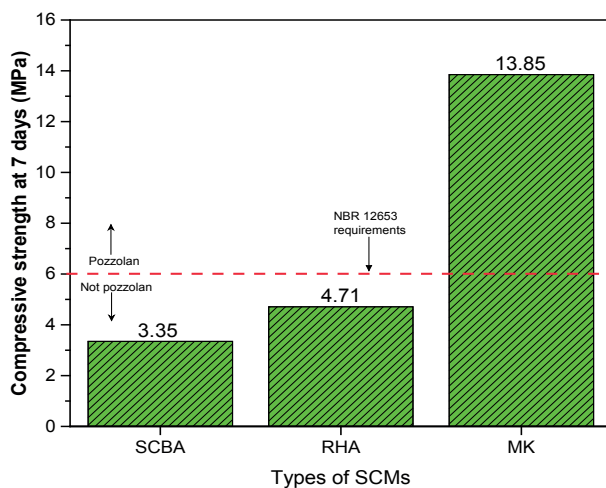


Figure 2. Pozzolanic activity with lime from SCBA, RHA and MK

It can be seen that the metakaolin had a strength above 6 MPa, which is a necessary requirement to attribute pozzolanicity to the materials investigated, according to ABNT NBR 12653 (2014). Although the RHA did not achieve a value of 6 MPa, the results are consistent with those presented in the next section of this chapter and with findings from previous studies (Fapohunda et al., 2017).

Mortar Mixing Design

The mixture proportions were defined with the aim of producing low-cement mortars for self-compacting concrete (SCC). The reference mix (REF) was defined from a self-compacting concrete previously tested in the laboratory (Diniz, 2018; Diniz et al., 2022), removing only the coarse aggregate from its composition. Table 3 shows the mixing ratio by mass of each mortar. Because the SCMs act as viscosity modifiers and for the mortars to reach a consistency of 300 mm \pm 50mm, a superplasticizer (SP) additive such as Glenium SCC 160 (BASF) was used.

Sample	Materials (Kg/m ³)							%SP
	CEM	SCBA	RHA	MK	HL	Sand	Water	
REF	719.90	-	-	-	-	1180.64	316.76	0.10
S40	411.82	274.55	-	-	-	1125.65	302.00	3.50
S40L10	373.47	248.98	-	-	62.25	1122.91	301.27	0.60
R40	415.52	-	277.01	-	-	1135.75	304.71	2.10
R40L10	376.51	-	251.01	-	62.75	1132.04	303.72	0.95
M40	424.09	-	-	282.73	-	1159.18	311.00	1.10
M40L10	383.53	-	-	255.69	63.92	1153.16	309.38	0.10

Table 3. Mix design of mortars for self-compacting concrete (SCC)

Molding mortars for SCC

For each mortar, 15 cylindrical samples measuring 5x10 cm² and 15 cubic samples measuring 5x5x5 cm³ were made. The mixing sequence for the materials was based on ABNT NBR 13281 (2005), but some adaptations were necessary. Initially, half the sand and water were mixed for 30 seconds. Next, cement, MK, the rest of the sand and 30% of the water were mixed for 60 seconds. Next, 50% of the additive was added and the mixture was homogenized for 90 seconds. Finally, the SCBA or RHA, together with the lime and the rest of the water (2%) and the SP additive (50%) were mixed for 120 seconds. After mixing, the mortars were placed in the molds mentioned above. After 24 hours of molding, the samples were demolded, identified and immersed in water saturated with lime for 28 days. After this period, the samples were dried in the open air for 14 days and then placed in the carbonation chamber.

Characterization in the hardened state

Fig. 3 shows a schematic drawing of the chronology of the tests and techniques used, as well as the respective environments used in each analysis. The following topics highlight all the protocols used in each test and technique.

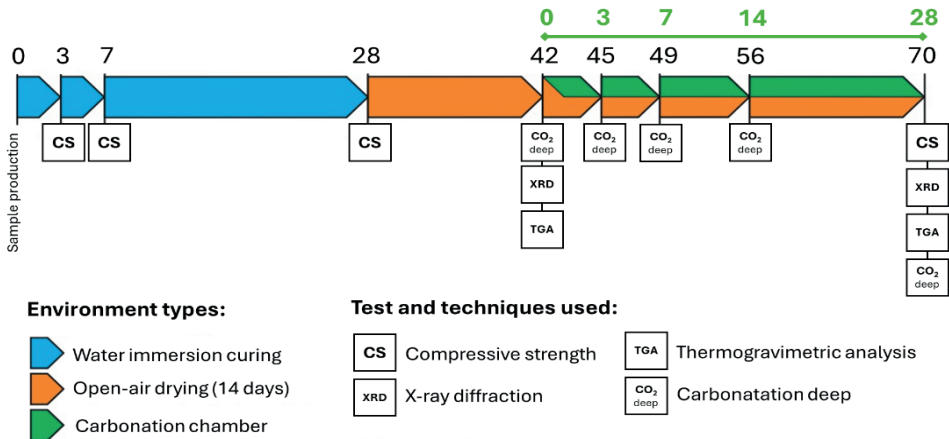


Figure 3. Schematic drawing of the chronology of the tests and techniques used

Compressive strength test

For this test, three cylindrical specimens were made for each age and broken in axial compression at 3, 7, 28 and 70 days from the date of molding. The compressive strength test was carried out based on the recommendations of ABNT NBR 13279 (2015).

Microstructural analysis

Microstructural analysis was carried out using XRD and thermogravimetric analysis (TGA) techniques. The samples were collected from specimens that were pulverized using a porcelain mortar with a Gauss hand. After this procedure, the samples were sieved using a 0.075 mm sieve (#200 mesh) and the passing material (below 0.075 mm) was used in the XRD and TGA analyses. The operating parameters for XRD were the same as those described in section 3.1 and for TGA, a Shimadzu DTG-60H thermal analyzer was used, operating at a heating rate of 10°C/min under a nitrogen atmosphere and a gas flow of 50 mL/min. No method of stoppage of the reactions was used in order to preserve the microstructure (Taylor, 1997).

The analyses were carried out before and after the samples entered the carbonation chamber. Nevertheless, the use of these microstructural analysis techniques allowed us to infer the influence of carbonation on the formation of hydrated products in mortars with the incorporation of various SCMs and with and without the addition of HL.

CO₂ Penetration depth

After curing and drying at 42 days, the carbonation test was carried out at 5 different ages, counted from the day they entered the chamber: 0 days (when entering the chamber) and 3, 7, 14 and 28 days (after entering the chamber). The CO₂ concentration in the chamber was 10% ± 0.5%, the temperature was 20 ± 2°C and the relative humidity was 55 ± 5%. Three cubic specimens were made for each age. To quantitatively determine the depth of CO₂ penetration, measurements were taken in the areas where there was no change in color using a caliper.

RESULTS AND DISCUSSIONS

Microstructural analysis

The results of the microstructural analysis using XRD and TGA techniques can be seen in Figs. 4 to 10. Fig. 4a shows that the portlandite peaks of the REF sample decrease after carbonation in the chamber.

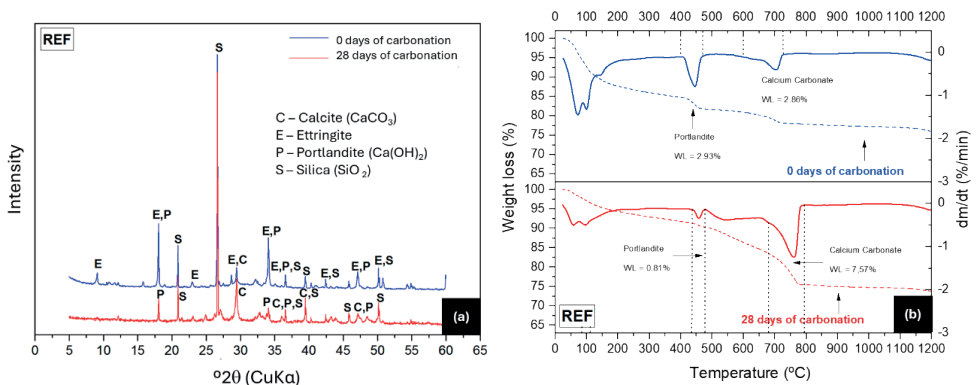


Figure 4. (a) XRD patterns and (b) TGA results of the REF sample

Fig. 4b shows that the weight loss (WL) due to the decomposition of portlandite and calcite reduces and increases after carbonation, respectively. This shows that carbonation is taking place as portlandite is converted into calcite. The results also reveal the permanence of peaks and thermal events related to C-S-H, which may indicate the preferential reaction of CO₂ with calcium hydroxide. This reaction, according by Del Bosque et al. (2020), occurs because thermodynamically it has priority over the decomposition of C-S-H.

In the S40 samples, the XRD patterns also indicated that the intensity of the portlandite peaks decreased, and the calcite phase appeared after carbonation, as can be seen in Fig. 5a. Carbonation was also indicated in the hydrated aluminates, as these compounds were present in greater quantities due to the incorporation of SCBA. It can also be seen that different compounds are formed, such as hydrated calcium silicoaluminate.

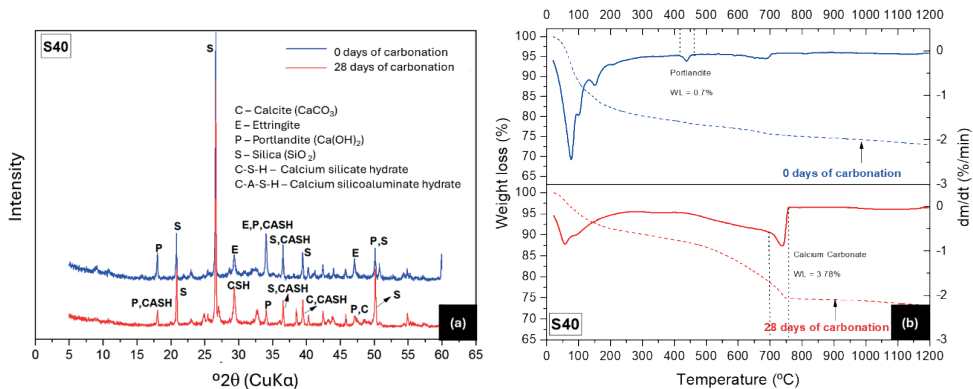


Figure 5. (a) XRD patterns and (b) TGA results of the S40 sample

The TGA results shown in Fig. 5b followed the same trend as the XRD results. There was a reduction in the loss of mass in the aluminate and portlandite zones, as well as a considerable increase in the weight loss associated with the decomposition of calcium carbonate after the carbonation process.

For the S40L10 sample, the behavior was similar to that of the S40 sample, since there was a reduction in the portlandite and aluminate peaks and, after carbonation, calcite formation, as shown in Fig. 6a.

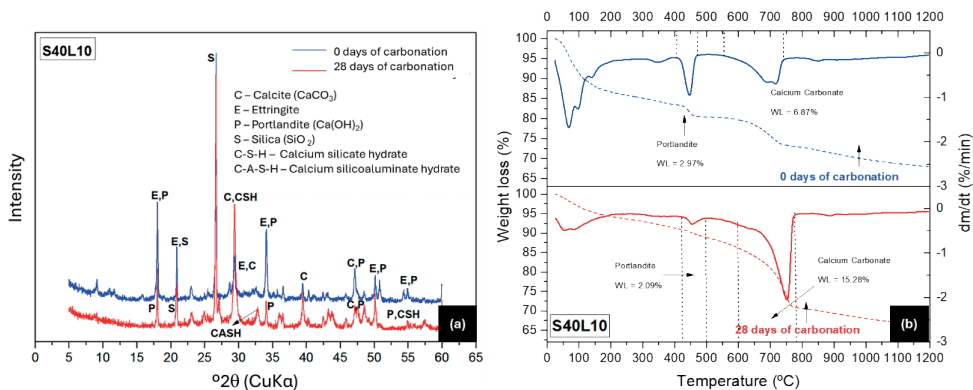


Figure 6. (a) XRD patterns and (b) TGA results of the S40L10 sample

The XRD patterns also revealed the presence of the vaterite phase, an unstable crystal formed at the beginning of calcite formation, indicating that the carbonation reaction is taking place. However, the presence of HL increased the intensity of the portlandite peaks compared to S40, indicating greater formation of this crystal.

This greater amount of portlandite resulted in greater production of calcium carbonates due to greater weight loss compared to sample S40, as shown in Fig. 6b. The addition of HL, therefore, together with the lower reactivity of SCBA may explain why the carbonation of sample S40L10 was higher than that of S40. Thus, portlandite was available throughout the cementitious matrix and because it reacted easily with CO₂ it allowed carbonation to progress. It is important to note that this may have been facilitated by the greater porosity of the mixtures with SCBA, since the mechanical strength of the mortars with these SCMs was lower than that of the mortars with RHA and MK, as will be discussed below.

In the sample with RHA incorporation (Fig. 7a), the results showed that, even at 0 days, the intensity of the portlandite peaks is low, there is a greater quantity and intensity of the C-S-H peaks, compared to the previous samples, and there is also formation of hydrated calcium silicoaluminates. These three factors can be attributed to the greater reactivity of RHA.

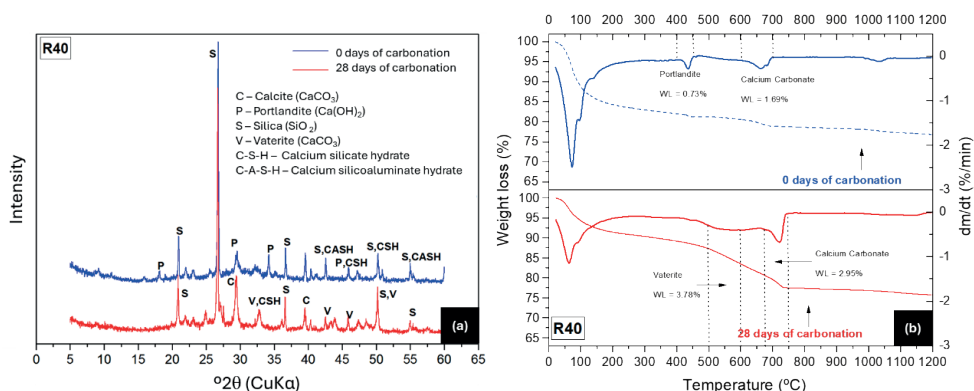


Figure 7. (a) XRD patterns and (b) TGA results of the R40 sample

The intensity of the calcite peaks is also low, indicating that the consumption of portlandite in the pozzolanic reaction reduces its availability to react with CO₂. The TGA validates this claim, where there is a reduction in portlandite, an increase in calcite and the appearance of vaterite, as shown in Fig. 7b. However, the loss of mass related to the decomposition of hydrates such as C-S-H is reduced after carbonation, indicating that they were consumed in the reaction. This effect is undesirable since C-S-H is the main hydrated product of cement, which makes carbonation more aggressive.

The microstructural analysis of sample R40L10 can be seen in Fig. 8a and Fig. 8b. The results show behavior similar to that of sample R40, but the intensity of the calcite peaks is significantly higher (Fig. 8a). This proves that the presence of HL favors the formation of both calcite and vaterite, as occurred in sample S40L10. The presence of HL causes carbonation to take place over a longer period, as there are lower intensity peaks in the vaterite phase. The TGA results revealed an increase in mass loss related to calcite decomposition, proving that carbonation has occurred (Fig. 8b).

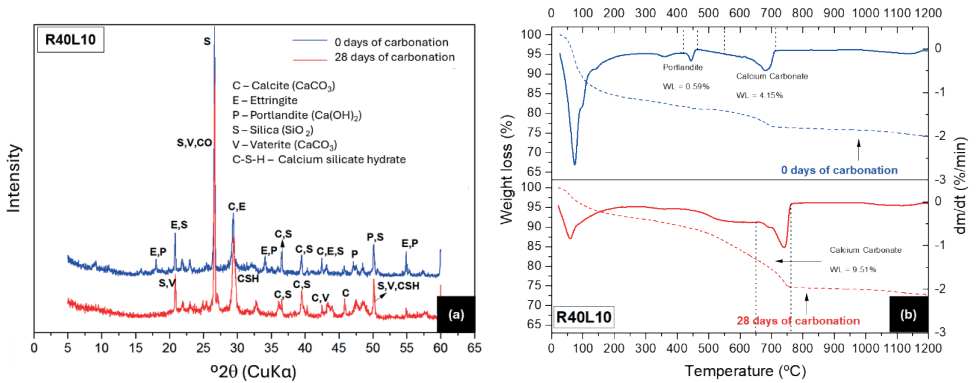


Figure 8. (a) XRD patterns and (b) TGA results of the R40L10 sample

Fig. 9a shows that the diffractogram of the sample incorporating MK (M40) has few peaks related to portlandite, which indicates that this hydrated product is consumed by the pozzolanic ratio. In this mixture, the peaks of this crystal are less intense, which may indicate an even smaller amount. This result can be attributed to the greater reactivity of MK compared to the other SCMs. In addition, the XRD results also revealed the presence of new aluminates, due to the composition of MK. As the availability of portlandite is reduced by the pozzolanic reaction, C-S-H becomes the main target for reacting with CO_2 . This hypothesis can be observed at $37^\circ 2\theta$ since the C-S-H is all converted into calcite.

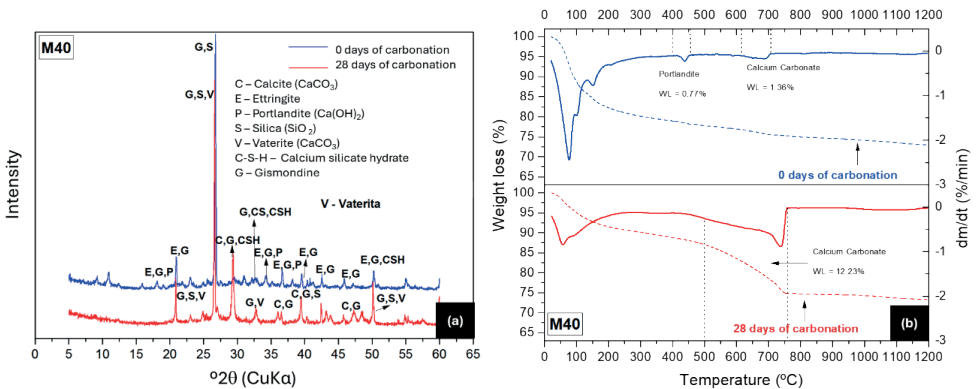


Figure 9. (a) XRD patterns and (b) TGA results of the M40

The results of the microstructural analysis of sample M40L10 follow the same trend as the results of sample R40L10, as shown in Fig. 10a. It was observed that the intensity of the peaks associated with calcite is greater due to the presence of HL. Vaterite is also present, again indicating that HL contributes to the development of carbonation reactions. However, in the samples with MK, there is greater carbonation of the hydrated aluminates due to the composition of these SCMs. TGA also showed that the thermal event associated with the decomposition of aluminates does not appear in the carbonated sample (Fig. 10b).

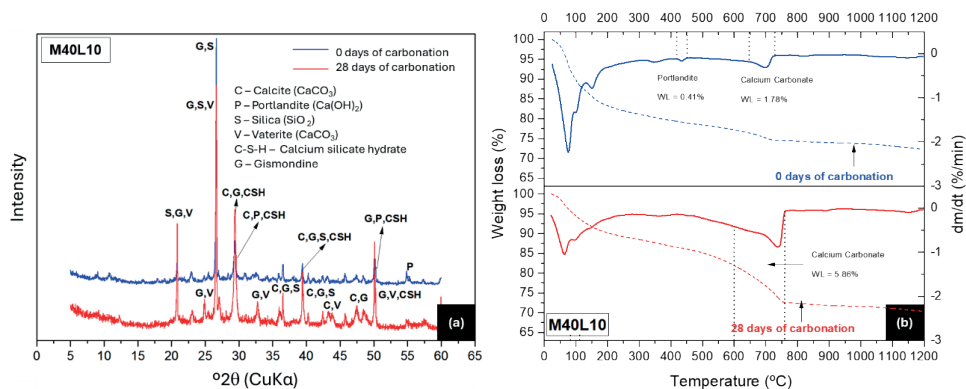


Figure 10. (a) XRD patterns and (b) TGA results of the M40L10

These results show that the addition of HL favors the development of the carbonation reaction. However, the CO_2 penetration depth results show that the samples with RHA and MK have equivalent values with and without the addition of HL. This shows that the negative effect of HL is greater when the SCMs have low reactivity, as is the case with SCBA.

CO_2 Penetration depth

The samples were inserted into the carbonation chamber and at 3, 7, 14 and 28 days they were removed to measure the depth of carbonation using the phenolphthalein spray technique. The results of this quantitative analysis of CO_2 penetration in the samples can be seen in Fig. 11. In general, the results showed that the carbonation front of the mortars with the SCMs is smaller compared to the reference mortar (REF) at all ages, except for sample S40. The reason for this is that due to the pozzolanic reaction of the SCBA, the portlandite content is reduced and, as a result, the pH of the solution decreases and CO_2 penetration is greater. There is also the possibility that there was a drop in CO_2 concentration inside the chamber, which affected the results and should therefore be disregarded.

On the other hand, the mortars with RHA had the lowest carbonation depths than the mortars with MK. This result is consistent with the compressive strength results, indicating that the 40% mass replacement of RHA contributes to obtaining a more cohesive matrix. In addition, the results for the MK mortars, with and without the addition of HL, were similar, indicating that they can also be used in this proportion of cement substitution.

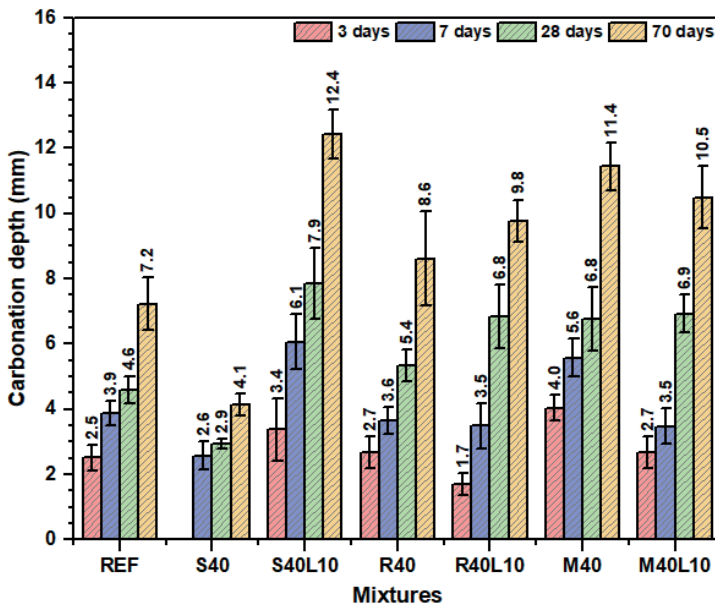


Figure 11. Depth of carbonation (mm) of mortars with SCMs and with the addition of HL

With regard to the effects of adding HL, it was observed that the addition of HL to the mortars with RHA and SCHA resulted in an increase in the depth of carbonation, while there was no significant influence on the mortars with MK. This indicates that the addition of HL not only raises the alkaline reserve, but also increases the rate of carbonation of the mortars.

However, it is important to note that all the mortars had a penetration depth of less than 25 mm. This value is the minimum cover for structural concrete in Brazil, according to the standards established in ABNT NBR 6118 (2023). Therefore, this physical barrier of the cover (space between the outer face of the structural element and the steel) is already efficient and validates the use of these materials since it helps to prevent corrosion.

Compressive strength

The results of the compressive strength tests of the mortars at 3, 7, 28 and 70 days of hydration are shown in Fig. 12. The reference sample (REF) showed the best results due to its higher cement content. However, the mortars incorporating RHA and MK showed results close to those of the REF mortar, which indicates the viability of using these SCMs. The reason for this behavior is due to the reactivity of these materials and, therefore, their ability to react with the calcium hydroxide released by cement hydration and result in the formation of additional C-S-H. As a result, mechanical performance is improved.

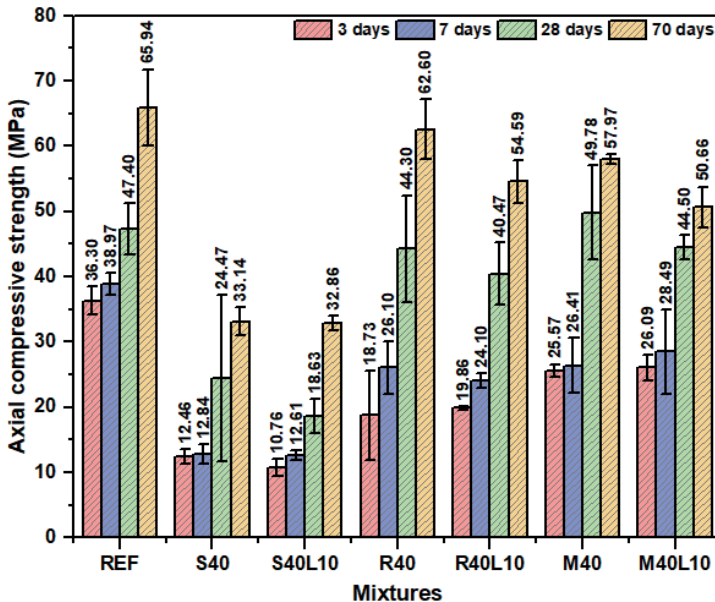


Figure 12. Compressive strength of mortars with SCBA, RHA, MK and without HL.

However, the SCBA mortars showed lower results than the other mortars, indicating that the low reactivity of these SCMs is the main factor responsible for this behavior.

CONCLUSIONS

This study evaluated the effects of incorporating different types of supplementary cementitious materials (SCMs), including metakaolin (MK), rice husk ash (RHA) and sugar cane biomass ash (SCBA), as well as the addition of hydrated lime (HL), on the carbonation resistance of mortars intended for self-compacting concrete (SCC). The following conclusions can be drawn from the results.

- In the microstructural analysis, either by XRD or TGA, the results revealed that there was a consumption of Portlandite and an increase in calcium carbonate after 28 days of carbonation. This behavior remained uniform in all the samples.
- The reference mix (REF) was the only one in which calcium hydroxide remained even after the full period in the accelerated carbonation chamber, revealing that the cement is highly resistant to degradation caused by CO_2 .
- In the REF, it was also observed that in the XRD patterns, the peaks associated with C-S-H remained even after the carbonation process, confirming that this process significantly involves calcium hydroxide. On the other hand, the opposite behavior was observed in the samples with SCMs, as the intensity of the C-S-H peaks decreased after 28 days of carbonation, indicating that due to the reduced availability of calcium hydroxide (consumed by the pozzolanic reaction), this phase also tends to carbonate.

- The depth of carbonation in all the mixtures did not reach the minimum 25 mm of cover as established in the normative standards. In practice, this prevents the carbonation front from reaching the steel during the useful life of structural elements built with SCC derived from these mortars.
- All the mixtures had good compressive strengths, with values above 30 MPa at 28 days. However, only the mortars with RHA and MK showed compressive strength values close to those of the REF mortar.

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