

## Evaluation of self-cleaning lime mortars containing titanium dioxide (TiO<sub>2</sub>) and metakaolin.

C. M. Lasta<sup>1\*</sup> , A. S. Fonseca<sup>1</sup> , F. Tiecher<sup>2</sup> 

\*Contact author: [charlesmichellasta@gmail.com](mailto:charlesmichellasta@gmail.com)

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### ABSTRACT

This study evaluated the performance of lime mortars modified with titanium dioxide (TiO<sub>2</sub>) and metakaolin (MK), with emphasis on compressive strength and self-cleaning capacity. Six different formulations were prepared and tested for consistency, mechanical strength, and color variation after exposure to natural weathering. The results indicated that TiO<sub>2</sub> improved both self-cleaning performance and compressive strength, with a statistically significant effect on color variation ( $\Delta E$ ). The presence of MK had no immediate effect on strength within the evaluation period. The originality of the study lies in the combined assessment of TiO<sub>2</sub> and MK in lime mortars exposed to different microclimatic conditions. It was concluded that TiO<sub>2</sub> contributes to the functional performance of mortars.

**Keywords:** lime mortar; titanium dioxide; photocatalysis; building materials.

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<sup>1</sup> Master's student, Graduate Program in Architecture and Urbanism, Atitus Educação, Passo Fundo, Brazil.

<sup>2</sup> Professor, PhD, Graduate Program in Architecture and Urbanism, Atitus Educação, Passo Fundo, Brazil.

### Contribution of each author

In this study, the authors Lasta and Fonseca contributed to conducting the experiments, exposing the samples, preparing the photographic record, producing the graphs, and writing the manuscript, accounting for 50% each in these activities. The experimental program was designed and supervised by Tiecher, who was also responsible for the use of the measurement and testing equipment. The discussion of the results, conclusions, and revision of the manuscript were carried out by all three authors, with an approximate contribution of 33% each.

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### Discussions and subsequent corrections to the publication

Any dispute, including the replies of the authors, will be published in the first issue of 2027 provided that the information is received before the closing of the third issue of 2026.

## **Evaluación de la autolimpieza de morteros de cal a partir de mezclas con dióxido de titanio (TiO<sub>2</sub>) y metacaolín.**

### **RESUMEN**

Este estudio evaluó el desempeño de morteros de cal modificados con dióxido de titanio (TiO<sub>2</sub>) y metacaolín (MK), con énfasis en la resistencia a la compresión y la capacidad autolimpiante. Se moldearon seis formulaciones distintas, que fueron ensayadas en términos de consistencia, resistencia mecánica y variación de color después de la exposición a la intemperie natural. Los resultados indicaron que el TiO<sub>2</sub> mejora tanto la autolimpieza como la resistencia a la compresión, con un efecto estadísticamente significativo sobre la variación de color ( $\Delta E$ ). La presencia de MK no tuvo un efecto inmediato sobre la resistencia. La originalidad del estudio radica en la evaluación conjunta de TiO<sub>2</sub> y MK en morteros de cal expuestos a diferentes microclimas. Se concluye que el TiO<sub>2</sub> contribuye al desempeño funcional de los morteros.

**Palabras clave:** mortero de cal; dióxido de titanio; fotocatalisis; materiales de construcción.

## **Avaliação da autolimpeza de argamassas de cal a partir de misturas com dióxido de titânio (TiO<sub>2</sub>) e metacaulim.**

### **RESUMO**

Este estudo avaliou o desempenho de argamassas de cal modificadas com dióxido de titânio (TiO<sub>2</sub>) e metacaulim (MK), com foco na resistência à compressão e capacidade de autolimpeza. Foram moldadas seis formulações distintas, testadas quanto à consistência, resistência mecânica e variação de cor após exposição ao intemperismo natural. Os resultados indicaram que o TiO<sub>2</sub> melhora tanto a autolimpeza quanto a resistência à compressão, com efeito estatisticamente significativo sobre a variação de cor ( $\Delta E$ ). A presença de MK não teve impacto imediato na resistência. A originalidade do estudo está na avaliação conjunta de TiO<sub>2</sub> e MK em argamassas de cal expostas a diferentes microclimas. Conclui-se que o TiO<sub>2</sub> contribui para o desempenho funcional das argamassas.

**Palavras-chave:** argamassa de cal; dióxido de titânio; fotocatalise; materiais de construção.

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## 1. INTRODUCTION

Lime mortar is one of the oldest construction materials and has been widely used in buildings and restoration due to its compatibility with historic materials and ease of application, with little change over the centuries. In addition, the use of cement in restoration is discouraged in many cases because of the irreversible damage it may cause to the stone matrix (Saeli et al., 2017). However, the exclusive use of air lime presents challenges, such as long curing times and low resistance to moisture and weathering agents, which has motivated research into new formulations better suited to restoration applications (Mattos and Gonçalves, 2019).

One alternative is the addition of pozzolans, since these materials improve the properties of binder composites by increasing durability and mechanical strength, as demonstrated in historic and traditional structures, including aqueducts and bridges (Aggelakopoulou et al., 2011).

With increasing pollution, especially in urban areas, construction materials are more severely affected and exhibit earlier signs of deterioration, such as dark stains caused by soot from vehicle engines, resulting in the loss of the aesthetic value of buildings (Pozo-Antonio and Dionísio, 2017). Environmental agents, such as moisture and UV radiation, can also compromise the durability and appearance of mortars, increasing maintenance requirements. In addition, air quality directly affects human health, making it essential to develop self-cleaning and pollutant-reducing materials that help reduce harmful emissions (Bhagyamma et al., 2023). In parallel, the use of antimicrobial and self-cleaning materials has emerged as a promising strategy to mitigate the growth of microorganisms on surfaces, thereby discouraging the spread of diseases in critical environments (Ramadan et al., 2023).

In this context, titanium dioxide ( $\text{TiO}_2$ ) stands out as one of the main photocatalytic agents used to improve the performance of mortars and concretes. Photocatalysis, a process activated by solar or UV radiation, enables  $\text{TiO}_2$  to degrade pollutants and microorganisms, thus contributing both to self-cleaning and improved air quality (Bhagyamma et al., 2023). In addition,  $\text{TiO}_2$  is valued for its chemical stability, biocompatibility, and low cost, making it a promising material for applications aimed at reducing environmental pollution and maintenance costs (Saeli et al., 2017; Gutiérrez et al., 2020). When added to mortars,  $\text{TiO}_2$  also improves mechanical strength and durability, especially in aggressive environments such as saline and acidic waters (Daniyal et al., 2019). These advances indicate that  $\text{TiO}_2$  may be an important component in the formulation of more sustainable and efficient construction materials.

Another noteworthy addition is metakaolin (MK), a pozzolan known for its ability to react with calcium hydroxide, forming compounds that increase the mechanical strength and durability of concretes and mortars, while also contributing to more sustainable construction practices because of the lower  $\text{CO}_2$  emissions associated with its production compared with Portland cement, for example (Mattos and Gonçalves, 2019). In addition, studies have shown that the incorporation of MK into mixtures containing  $\text{TiO}_2$  can improve long-term photocatalytic performance (Lara et al., 2023). Metakaolin therefore not only improves the physical structure of the mortar, but also enhances the self-cleaning and air-purifying effects provided by  $\text{TiO}_2$ , making it a promising This study evaluates the self-cleaning behavior of lime mortars modified with titanium dioxide ( $\text{TiO}_2$ ) and metakaolin (MK), with emphasis on the effects of each additive on the functional and aesthetic properties of the mortars. The research provides comparative data on the effectiveness of these mixtures, highlighting their potential to improve durability and reduce maintenance requirements in applications exposed to polluting agents.

## 2. EXPERIMENTAL PROGRAM

The present study aims to evaluate the performance of lime mortars containing metakaolin (MK) and titanium dioxide ( $\text{TiO}_2$ ) with respect to self-cleaning capacity and compressive strength. To this end, six mortar compositions were prepared with different  $\text{TiO}_2$  and MK contents, as described below.

### 2.1 Materials

For the development of the experimental program, sand–lime mortars were produced without  $\text{TiO}_2$  addition and with 5% and 10%  $\text{TiO}_2$  addition, based on the mass of lime. The addition of metakaolin to the mixtures was also evaluated, at contents of 5% and 10%. The compositions investigated are presented in Table 1.

Table 1. Mortar compositions.

Mortar ID	Mix proportion				W/B Ratio
	Lime	MK	Sand	$\text{TiO}_2$	
A01	1	-	4	-	0,9
A02	1	-	4	0,05	0,9
A03	1	-	4	0,10	0,9
A04	0,85	0,15	4	-	0,9
A05	0,85	0,15	4	0,05	0,9
A06	0,85	0,15	4	0,10	0,9

Figure 1 shows the appearance of each mixture in the fresh state. The mortars became visibly whiter as the  $\text{TiO}_2$  content increased.

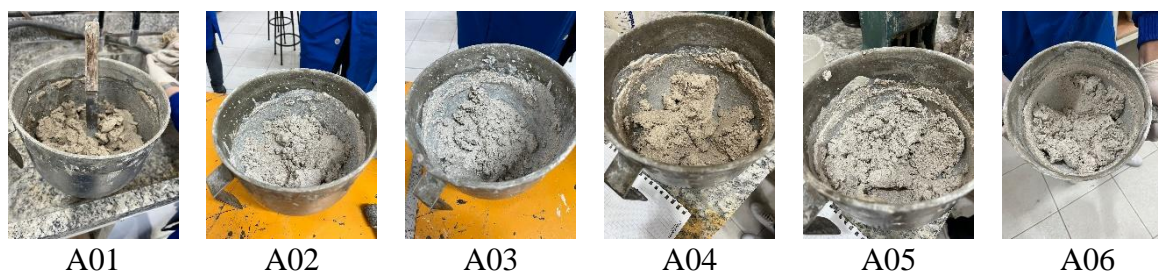


Figure 1. Mixtures in the fresh state.

### 2.2 Mortar preparation and specimen casting.

The mixing process was carried out using a bench mortar mixer, always following the same mixing sequence, in accordance with NBR 7215 (ABNT, 1996).

After mixing, a consistency test was performed according to NBR 13276 (ABNT, 2016) to evaluate the workability and flowability of the mortars based on spread measurements.

Subsequently, specimens measuring  $4 \times 4 \times 4$  cm were cast for compressive strength evaluation. Six specimens were prepared for each mortar type for this test.

Self-cleaning performance was evaluated using mortar plates measuring  $10 \times 10 \times 2.5$  cm, with one plate cast per mixture, totaling six plates. It is acknowledged that, although this approach provides quantitative data on color variation, it has limitations in terms of statistical

representativeness because only one sample was used for each formulation. The results should therefore be interpreted in light of this methodological limitation. The mixing and casting process followed the steps below:

1. The specimens were cast in two layers, and each layer was compacted with 20 blows using a manual tamper to ensure good material distribution;
2. After casting, the specimens were cured for 28 days in an environment with controlled temperature and humidity.

## 2.3 Evaluation methods.

### 2.3.1 Compressive strength test.

Compressive strength was evaluated according to the procedures described in NBR 13279 (ABNT, 2005), using an *Emic* hydraulic press with a load capacity of 220 kN.

### 2.3.2 Self-cleaning evaluation .

For self-cleaning assessment, the mortars were exposed to natural weathering while facing east, in the cities of Canoas (samples with MK + TiO<sub>2</sub>) and Montauri (samples with TiO<sub>2</sub> only), both located in the state of Rio Grande do Sul, Brazil, but with different microclimates in terms of environmental pollution. The distribution of samples between the two locations was defined to prioritize the evaluation of different formulations under distinct climatic conditions, thereby maximizing the diversity of exposure environments within the available number of samples. Canoas is a municipality with 347,657 inhabitants (IBGE, 2022 (01)), located in the metropolitan region of the state, and therefore exhibits higher levels of atmospheric pollution. Montauri is a municipality with 1,499 inhabitants (IBGE, 2022 (02)), located approximately 200 km from Porto Alegre, and its microclimate resembles that of a rural area (Figure 2).

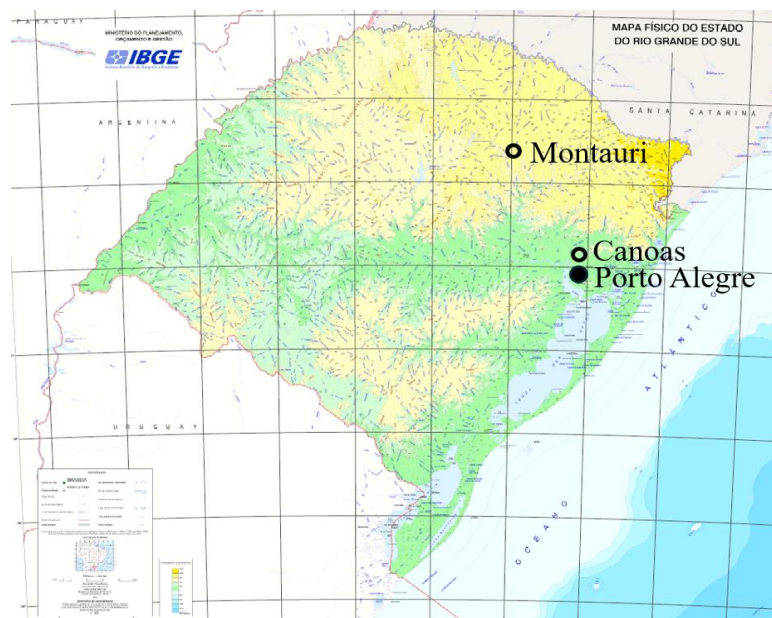


Figure 2. Cities where the samples were exposed

Source: IBGE, 2010

Before exposure to the respective microclimates, the samples were stained with the following agents, as shown in Figure 3:

- Red wine (1);
- Olive oil (2);
- Coal ash (3);
- Rust (wet steel wool) (4).

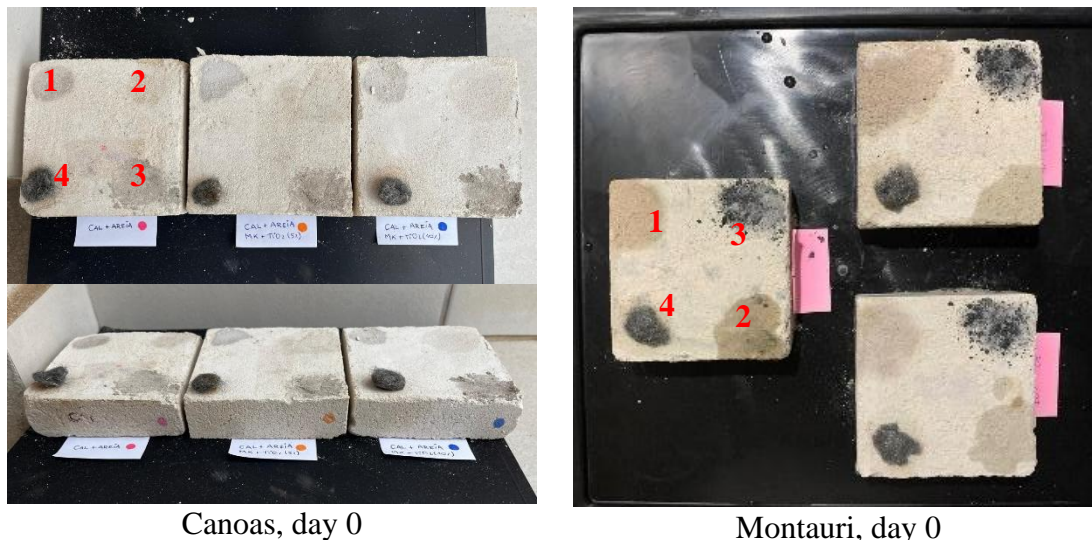


Figure 3. Staining of the samples.

Exposure to climatic conditions lasted 50 days, and the samples were monitored weekly through photographic records. After this period, color was evaluated using a portable spectrophotometer, model CM-500d, manufactured by *Minolta*. The spectrophotometer was used for colorimetric characterization through sample readings with the aid of *Cyberchrome OnColor* software (Austria, 2015), which detects and quantitatively compiles the generated data for subsequent analysis and interpretation.

Color variation (dirtiness) was measured using the *Lab\** system, which is based on a scale that considers colors as combinations of three receptor color types: red, yellow, and blue. In this scale, the spectrophotometer results indicate:

- *L\**: lightness
- *a\**: red/green coordinate
- *b\**: yellow/blue coordinate

### 3. RESULTS

#### 3.1 Evaluation of mixture consistency

Figures 4 and 5 present the average spread values measured for the mortars in the consistency test; the appearance of the mixtures before and after testing can also be observed.

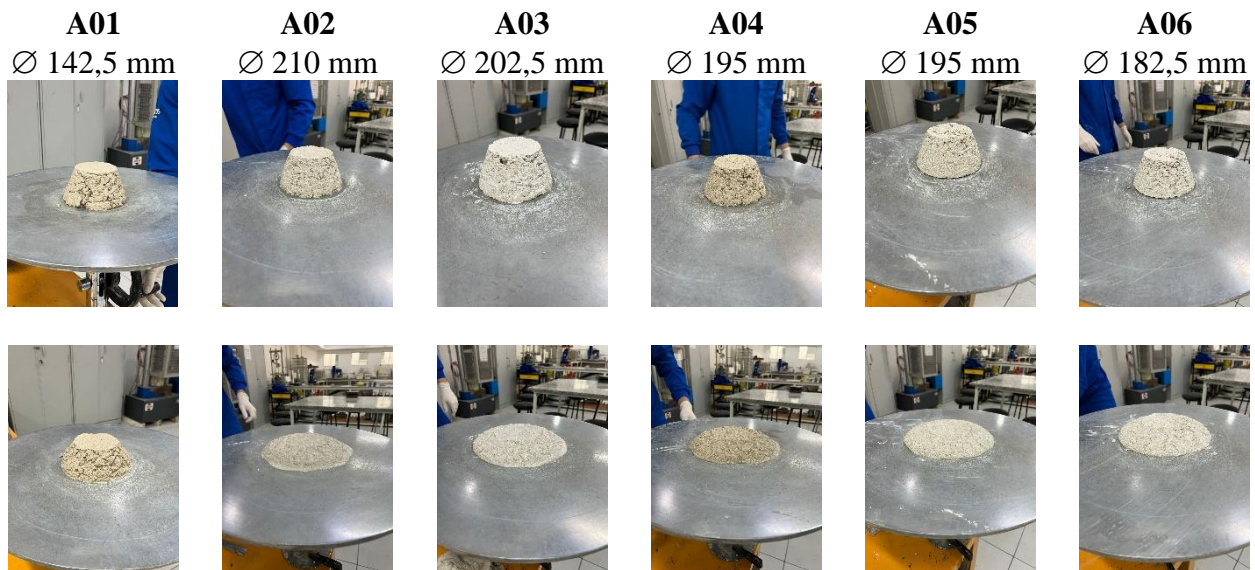


Figure 4. Consistency test.

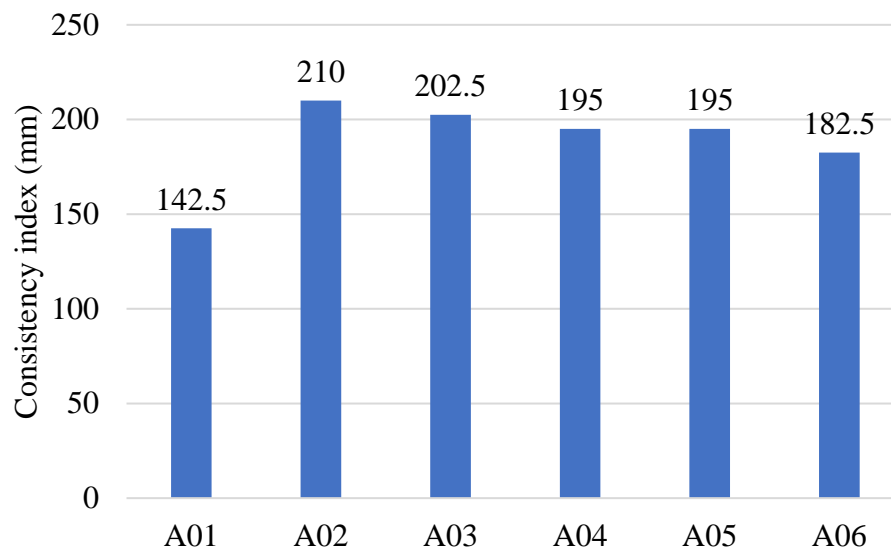


Figure 5. Consistency test results

Overall, little variability was observed in the consistency index among the different mortars. However, because the water-to-binder ratio (w/b) was kept constant, the addition of MK and TiO<sub>2</sub> led to a reduction in this parameter.

### 3.2 Compressive strength

The compressive strength test was carried out at 28 days, and the results are presented in Table 2 and Figure 6. Compressive strength values were, on average, higher for mortars 01, 02, and 03 than for mortars 04, 05, and 06. In mixtures 01, 02, and 03, a greater amount of lime was used than in the other samples. Although metakaolin improves mechanical properties, its pozzolanic effect becomes more evident at later ages, particularly beyond 28 days (Cruz et al., 2019). Future studies should therefore include evaluations at later ages to better understand the combined contribution of metakaolin and TiO<sub>2</sub> to mortar properties. Mattos and Gonçalves (2019), for example, showed that MK contributed to improving the compressive strength of lime mortars only at 180 days.

Table 2. Compressive strength.

Mixture	Mean stress (MPa)	Standard deviation
A01	0,31	0,023
A02	0,32	0,039
A03	0,30	0,013
A04	0,26	0,025
A05	0,24	0,031
A06	0,29	0,021

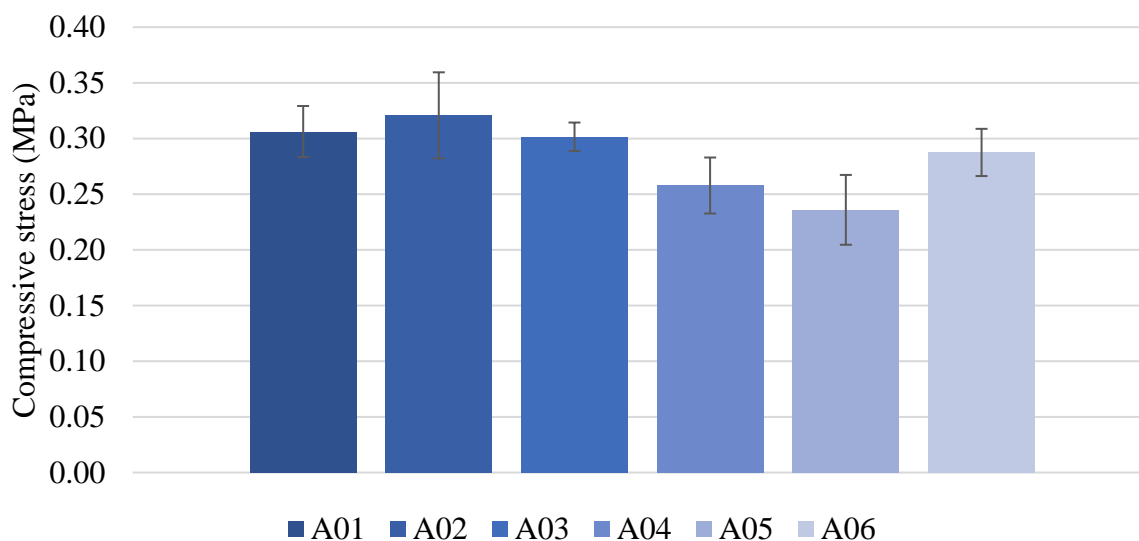


Figure 6. Compressive strength of the samples

The results indicate that the strength values obtained were low, which was expected given the binders used. Lime is a binder with a lower potential for strength development; in the study by Mattos and Gonçalves (2019), the authors obtained similar strengths for lime and MK mortars at 28 days, on the order of 0.4 MPa.

According to Hatori et al. (2023),  $\text{TiO}_2$  also improves the mechanical strength of mortars when incorporated. In the present study, the compressive strength of mortar 02 was higher than that of mortar 01 with only the inclusion of 5%  $\text{TiO}_2$ ; however, no increase in strength was observed for mortars with 10%  $\text{TiO}_2$  (mortar 03). According to Bersch (2021), this behavior may be explained by the high cohesion of the material due to the presence of fines, which may trap voids. A similar result was observed for the samples containing MK.

### 3.3 Self-cleaning evaluation

During the exposure period, weekly photographic monitoring of the mortars was carried out, as shown in Figure 7.



Canoas, day 01



Montauri, day 01



Canoas, day 08



Montauri, day 08



Canoas, day 15



Montauri, day 15



Canoas, day 22



Montauri, day 22



Canoas, day 29



Montauri, day 29



Canoas, day 36



Montauri, day 36



Canoas, day 43



Montauri, day 43



Canoas, day 50



Montauri, day 50

Figure 7. Photographic monitoring of the stained samples.

As shown in Figure 7, all samples exhibited some degree of self-cleaning throughout the exposure period. Although visual inspection may suggest that the reference samples (A01 and A04) exhibited less color change, it is important to consider the color variation data ( $\Delta E$ ) presented in Figure 9. Colorimetric analysis detects differences that are not visually perceptible, and the results indicate that the mortars containing  $TiO_2$  returned to a color closer to the original. This photocatalytic effect tends to become more pronounced with increasing exposure time, suggesting that the self-cleaning benefit may become more evident over the long term. The statistical analysis (ANOVA), presented

in Figure 10, confirms the influence of TiO<sub>2</sub> on color variation. Regarding the staining agents used, a greater reduction in dirtiness was generally observed for red wine and olive oil, whereas more pronounced residues remained for coal ash and rust. Rust staining showed the greatest resistance to self-cleaning, leaving a more visible mark than the other agents applied.

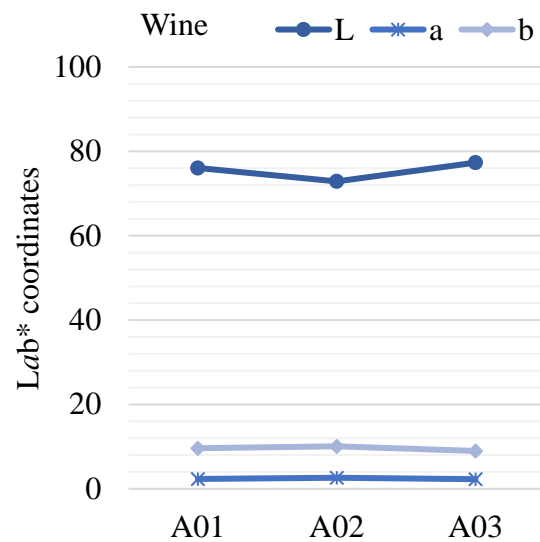
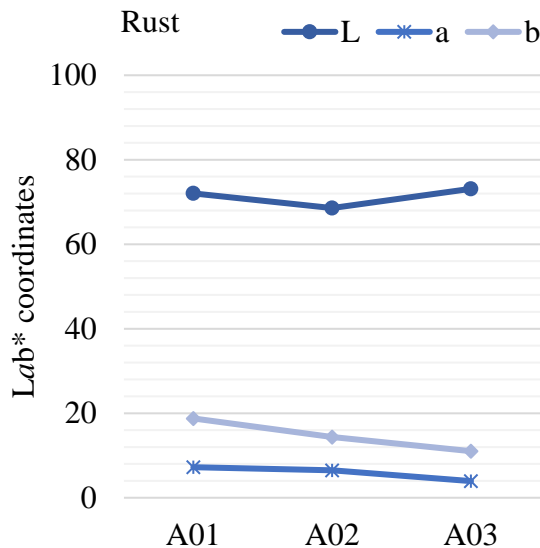
After the exposure period, color variation measurements were performed using the spectrophotometer at the four points where staining had occurred on the specimens. The equipment takes three measurements per point, and the results presented here correspond to the mean of these three measurements (Tables 3 and 4), obtained at the locations identified as most representative of each stain. Figure 8 presents the average results of the Lab\* system, separated into samples A01 to A03 and A04 to A06, so that the evolution of self-cleaning for each mix can be followed as different amounts of TiO<sub>2</sub> are added. Presenting these results graphically provides a clearer visualization of trends and comparisons among the staining agents.

Table 3. Lab\* system coordinates of the specimens exposed in Montauri.

Sample	Rust			Wine			Oil			Coal		
	L	a	b	L	a	b	L	a	b	L	a	b
A01	72,05	7,23	18,77	76,05	2,34	9,62	73,67	2,24	11,44	68,73	1,53	6,34
A02	68,57	6,49	14,37	72,87	2,63	10,08	75,27	3,07	11,64	61,40	1,21	4,46
A03	73,12	3,94	11,02	77,32	2,29	8,98	76,72	2,44	10,03	69,70	1,07	4,61

Table 4. Lab\* system coordinates of the specimens exposed in Caonas.

Sample	Rust			Wine			Oil			Coal		
	L	a	b	L	a	b	L	a	b	L	a	b
A04	73,18	2,88	12,24	75,74	2,72	10,95	76,64	2,82	10,80	73,07	2,44	10,62
A05	70,37	8,11	20,94	65,26	2,10	8,62	77,83	2,01	9,13	80,29	1,68	7,88
A06	74,14	5,29	14,80	77,16	1,49	7,74	74,60	1,51	7,28	79,03	1,75	7,89



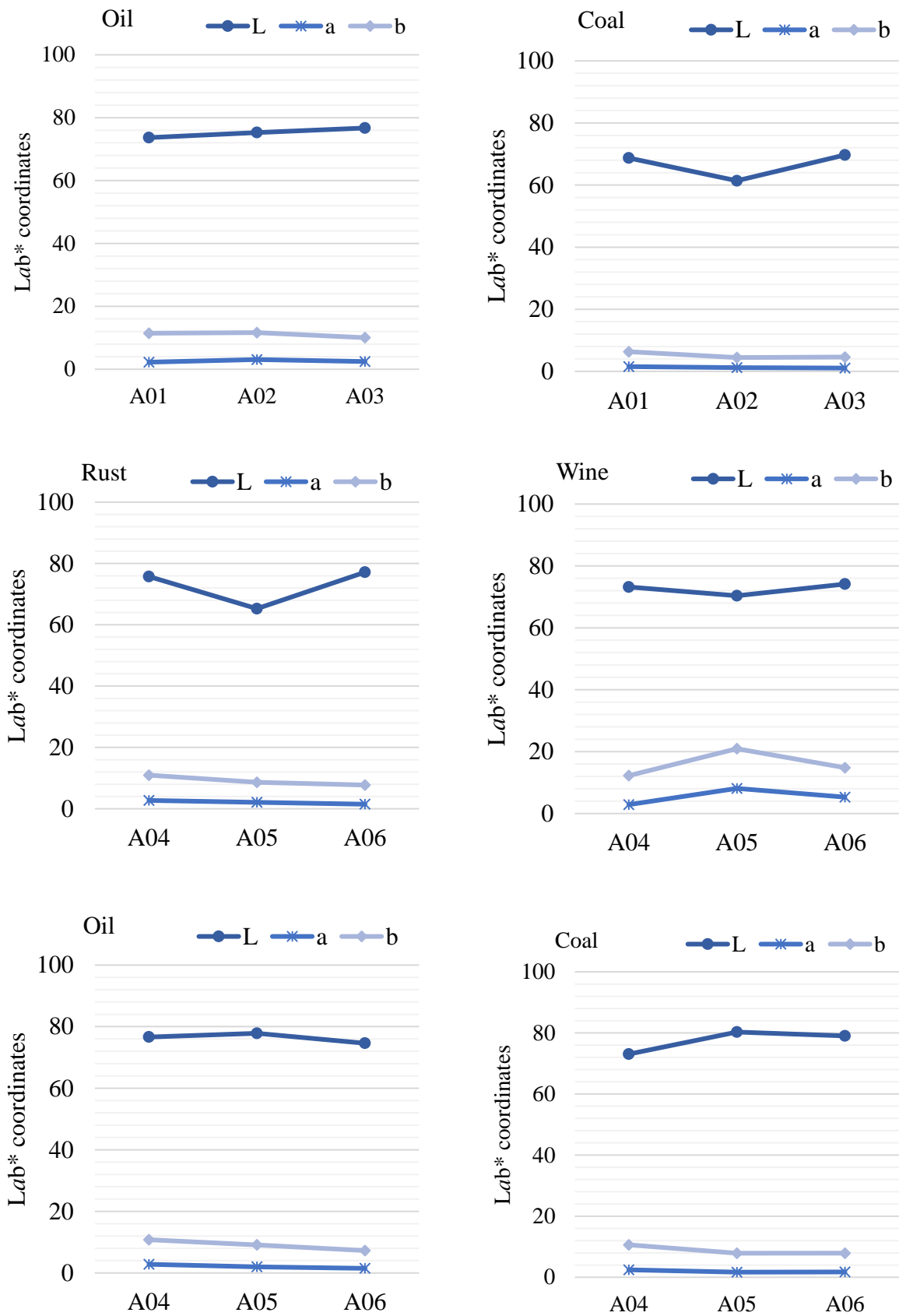


Figure 8. Lab\* system results

Figure 8 shows that, overall, the results were relatively similar. With respect to L\*, for example, that measures the lightness scale from black to white, a trend toward whiteness was observed in the mortars containing TiO<sub>2</sub>.

To assess color differences among the mortars according to TiO<sub>2</sub> content, the mean color variation values ( $\Delta E$ ) were calculated relative to the corresponding reference mortar without TiO<sub>2</sub>. Thus, the values of A02 and A03 were compared with A01, and the same procedure was applied to A05 and A06 relative to A04 (Figure 9).

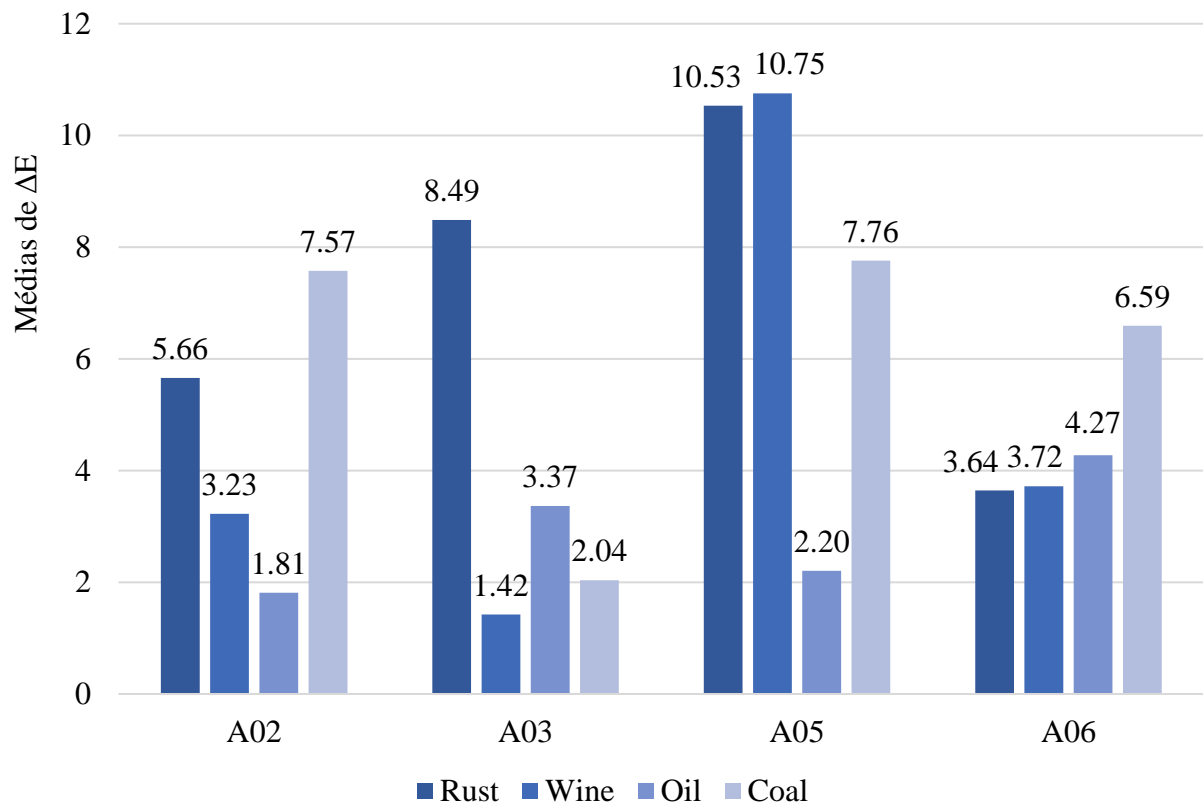


Figure 9. Color variation ( $\Delta E$ ).

Considering the eight scenarios evaluated, a greater tendency toward whiteness can be observed as the TiO<sub>2</sub> content increases; in other words, the mortars became more similar to the reference sample without TiO<sub>2</sub> addition.

Table 5 and Figure 10 present the analysis of variance (ANOVA) for the influence of TiO<sub>2</sub> content and stain type on the samples.

Table 5. Analysis of variance (ANOVA).

Effect	SS	Degrees of freedom	MS	F	p
TiO <sub>2</sub>	102,2103	2	51,1052	6,24599	0,023229
Stain	19,1208	3	6,3736	0,77897	0,538002
TiO <sub>2</sub> * Stain	29,8537	6	4,9756	0,60811	0,719443
Error	65,4566	8	8,1821	-	-

SS=Sum of squares; MS=Mean squares; F=Statistic F; p=Probability.

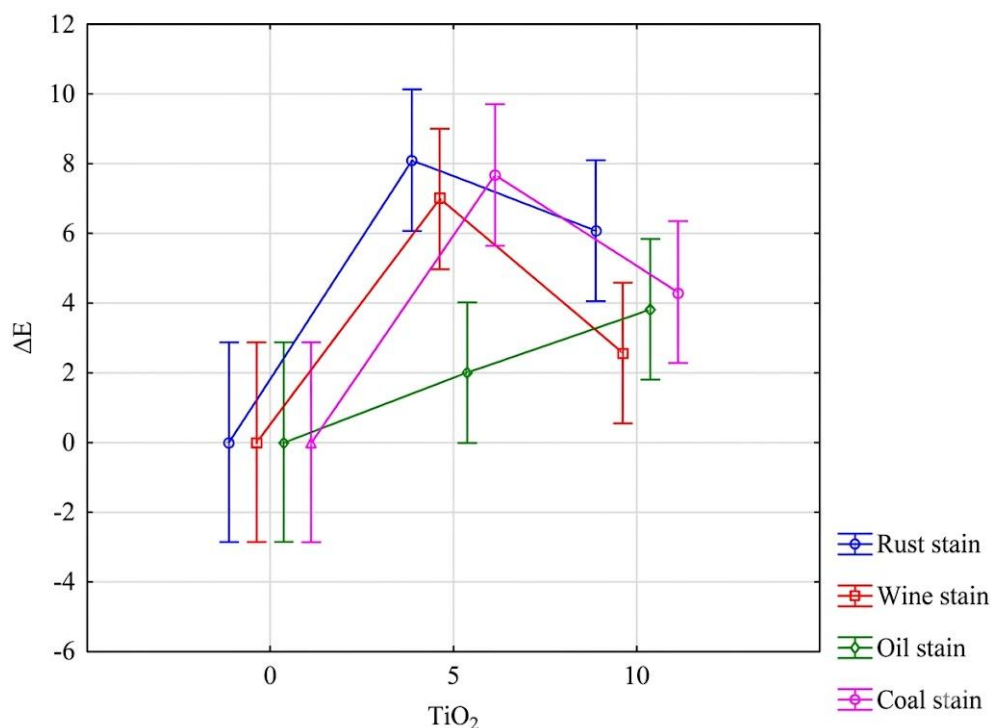


Figure 10. ANOVA for the effect of the interaction between TiO<sub>2</sub> content and stain type.

A factorial analysis of variance (Two-Way ANOVA) was performed to evaluate the effects of TiO<sub>2</sub> and stain type on color variation ( $\Delta E$ ) after exposure to natural weathering. The results showed that only the presence of TiO<sub>2</sub> had a statistically significant effect ( $p = 0.0232$ ), whereas stain type ( $p = 0.5380$ ) and the interaction between TiO<sub>2</sub> and stain ( $p = 0.7194$ ) were not significant. These results indicate that color variation was governed by TiO<sub>2</sub> content, regardless of the staining agent. The interaction plot (Figure 10) supports this interpretation, showing a marked increase in  $\Delta E$  between 0% and 5% TiO<sub>2</sub>, with similar behavior among the different types of stain and no pronounced crossing of the curves.

Regarding the effect of the exposure microclimate, both environments were subtropical and exhibited marked seasonal variation, although with different pollution levels. Canoas was more polluted because it is located in the metropolitan region, whereas Montauri was less polluted because it is rural. This was evident in the visual analysis (Figure 7), which showed faster self-cleaning and fewer residues in Montauri.

In this study, the samples were first exposed at 28 days of age, recognizing that the curing process continues during exposure to the test environments. Therefore, future studies should evaluate specimens at later ages to determine whether the catalytic effect of TiO<sub>2</sub> becomes more efficient.

#### 4. CONCLUSIONS

The results of this study indicate that the compressive strength of the specimens was higher in mortars with a greater amount of lime. Therefore, the effects associated with the presence of metakaolin could not be observed within the strength evaluation period adopted in this research (28 days).

The addition of TiO<sub>2</sub> improved compressive mechanical strength, especially in the samples without MK, and also enhanced self-cleaning performance, as confirmed by the analysis of variance of the color variation ( $\Delta E$ ) results. Stain type, on the other hand, did not significantly affect the results,

indicating that self-cleaning performance is more closely related to material composition than to the staining agent.

Little variation was observed in the consistency index among the mixtures; nevertheless, a decrease in this parameter was identified when metakaolin and  $\text{TiO}_2$  were added.

## 5. ACKNOWLEDGEMENTS

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