

PROPOSAL OF A TEST METHOD FOR DETERMINING WATER STAIN RESISTANCE IN THE BACK OF THE CERAMIC TILE

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1. INTRODUCTION

The water stain is a change of shade due to the presence of moisture in the engobe layer of the ceramic tile. Moisture migrates through the product's back and is lodged in the engobed surface. This color change consists of the darkening of the tile surface noticeable to the human eye, thus affecting the aesthetic performance of the products. This change in surface appearance may be permanent or temporary (after drying, the product's original color returns to normal). Some peculiarities already noted

on this subject are: (a) the spots are only visible in the case of the use of transparent glazes and white engobes, occurring more frequently from the edges (especially if they are cut), but also occasionally appearing in the central regions of the tiles; (b) the spots may simply correspond to changes in gray from the original shade or may have different color patterns (bluish, reddish, greenish, etc.); (c) the occurrence is always associated with the excessive presence of humidity in the environment and the access of water and its accommodation in the engobe layer; therefore, its occurrence in humid environments (outdoor areas, bathrooms, kitchens, etc.) is more frequent and (d) Water stains disappear when parts are simply oven dried or re-burned at 550 °C.

The objective of this work was to develop a technological study to propose a new test method to evaluate the resistance to water stains of ceramic tiles. This study generated a test method proposal that was submitted to the ISO / TC 189 Technical Committee.

2. MATERIALS AND METHODS

In this study the following variables were evaluated: ceramic tiles with different water absorption group (BIa, BIIa, BIIb and BIII), different colors (light and dark), different surface finishes (satin and bright), monocolor and design products, different water stain grades and non-stain products. A total of 24 products were tested using two staining agents (water and methylene blue) and various contact times between staining agent and the tile back. Color change analysis was done visually (not visible, barely visible and very visible) and also by measuring color coordinates using a reflectance spectrophotometer. Figure 1 presents the experimental procedures used for each staining agent.

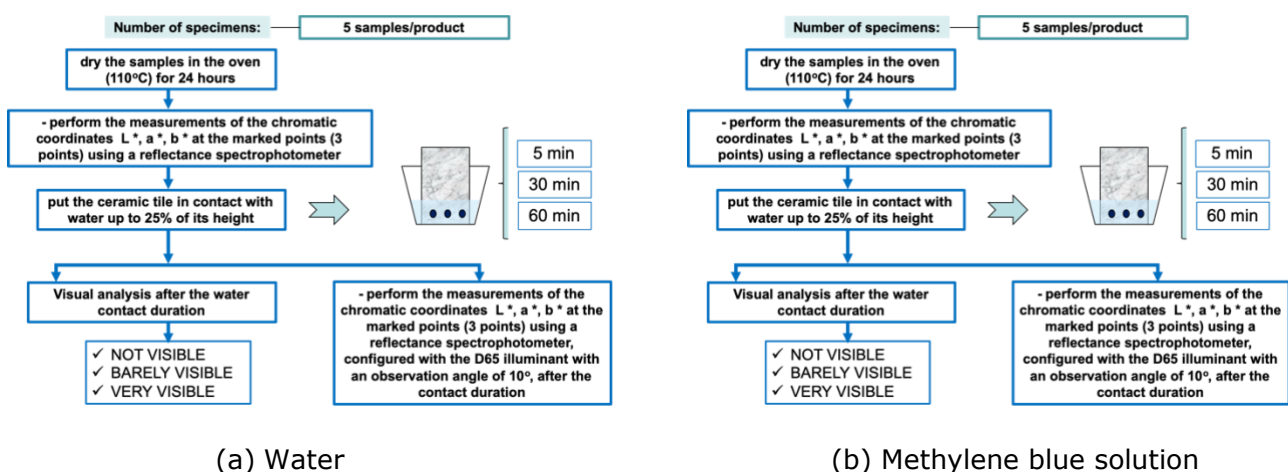


Figure 1: Test procedure for each staining agent: (a) water and (b) methylene blue

3. RESULTS AND DISCUSSION

Table 1 presents the summary of all results obtained.

Absorption group	Colour	Surface finish	Monocolor or decorated	Product	Water			MethYlene Blue			Comparative
					Exposure time - Water			Exposure Time - Methylene Blue			
					5 min	30 min	60 min	5 min	30 min	60 min	
Bla	Dark	Satin	Monocolor	1E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	Ok
	Light	Satin	Monocolor	2E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
	Light	Bright	Decorated	3E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
	Dark	Satin	Decorated	4E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
	Light	Satin	Decorated	5E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
BIIa	Light	Bright	Monocolor	6	Not visible	Not visible	Not visible	Not visible	Not visible	Barely visible (3)	NOT
				6E	Not visible	Not visible	Not visible	Not visible	Barely visible	Barely visible	
	Light	Satin	Monocolor	7	Not visible	Not visible	Not visible	Not visible	Not visible	Barely Visible (1)	NOT
				7E	Not visible	Not visible	Not visible	Not visible	Barely visible	Barely Visible	
	Light	Bright	Decorated	8E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
	Dark	Satin	Decorated	9E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
	Light	Satin	Decorated	10E	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	OK
BIII	Light	Bright	Monocolor	13	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	NOT
				13E	Not visible	Not visible	Not visible	Very visible	Very visible	Very visible	
	Light	Satin	Monocolor	15	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	NOT
				15E	Barely visible	Barely Visible	Barely visible	Very visible	Very visible	Very visible	
	Dark	Bright	Decorated	16	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	NOT
				16E	Not visible	Not visible	Not visible	Very visible	Very visible	Very visible	
	Light	Bright	Decorated	17	Barely visible	Barely Visible	Barely visible	Not visible	Not visible	Not visible	NOT
				17E	Not visible	Not visible	Not visible	Very visible	Very visible	Very visible	
	Light	Satin	Decorated	18	Not visible	Not visible	Not visible	Not visible	Not visible	Not visible	NOT
18E				Not visible	Not visible	Not visible	Very visible	Very visible	Very visible		
BIIB	Light	Satin	Monocolor	14	Very visible	Very visible	-	Very visible (1)	Very visible (1)	Very visible (1)	NOT
	Light	Bright	Monocolor	19	Very visible	Very visible	Very visible	Very visible (3)	Very visible (3)	Very visible (3)	NOT
	Light	Bright	Monocolor	20	Very visible	Very visible	-	Very visible	Very visible	Very visible	OK
	Light	Satin	Monocolor	21	Very visible	Very visible	-	Barely visible	Very visible	Very visible	OK
	Light	Satin	Decorated	22	Very visible	Very visible	Very visible	Barely visible	Very visible	Very visible	OK
	Dark	Bright	Decorated	23	Barely visible	Barely Visible	Barely visible	Not visible	Barely visible (1)	Muito Visível (1)	NOT
	Dark	Bright	Decorated	24	Very visible	Very visible	-	Barely visible (1)	Very visible (1)	Very visible (1)	NOT
	Light	Satin	Monocolor	25	Not visible	Not visible	No visible	Not visible	Not visible	Not visible	OK
	Light	Bright	Monocolor	26	Very visible	Very visible	-	Muito Visível (1)	Very visible (1)	Very visible (1)	NOT

Table 1: Summary of results of water stain resistance tests using water and methylene blue as staining agents.

The partial immersion of the ceramic tile in water is the best way to evaluate the water stain from the back of the piece. The methylene blue solution was not adequate for this type of evaluation. The water stain test should be done only on glazed products. Products that generate complaints are those that very quickly have a water stain (in 5 minutes). The most suitable times to maintain partial immersion in water are 5 and 30 minutes. The 30 minutes represents the time for a long bath. The use of a spectrophotometer to obtain the color coordinates is only valid for monocolor products. Therefore, it is not applicable for decorated and colorful ceramic tiles. It is essential that the submerged part must be cut.

4. CONCLUSION

As a conclusion to this work, a proposal for a test method was elaborated and submitted for evaluation by the ISO / TC 189 Technical Committee. The following is a summary of the proposed procedure:

- a) Dry the test specimens in the oven adjusted to a minimum of $(110 \pm 5)^{\circ}\text{C}$ for a minimum of 24 h.
- b) Put the test specimens in contact with water up to 25% of its height. The cut side should be in contact with water. The specimens should be in contact with water for 5 minutes.
- c) After contact time with water, set the test specimens, with the proper surface under observation, so that it can be viewed perpendicularly to the surface at a distance of $1 \text{ m} \pm 0,10 \text{ m}$. Illuminate them with an even and diffuse light intensity of $275 \text{ lx} \pm 25 \text{ lx}$ at the surface of the tiles. View the tiles with the naked eye or with spectacles if usually worn.
- d) Evaluate if color change has occurred in the area that came into contact with water. If the color change is verified, the test is completed, and it is noted which specimens showed visible color change.
- e) If within 5 minutes no color change is verified, put the same test specimens in contact with water up to 25% of its height. The cut side should be in contact with water. The specimens should be in contact with water for an additional 25 minutes.

7.6 Repeat procedure (c) and (d). Note which specimens showed visible color change.

Acceptance criterion is no presence