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Simulated Acidic Weathering of "Black Granites": an Assessment using Regression Analysis

Simulação de Atmosfera Ácida em "Granitos Negros": uma Avaliação por Análise de Regressão

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Abstract

A number of "black granites" were tested using simulated acidic weathering (H_2SO_4, HNO_3, H_2SO_4/HNO_3 and HCl, at pH = 3,00), freeze resistance and relative loss of brightness. The same plates of these granites remained immersed in each acidic solution over eight weekly cycles. The pH data from residual acid solutions were plotted versus timing of the experimental cycles in XY diagrams for interpretation as log-linear regressions. The global set of data, transcribed in a ternary diagram, shows a distinctive area highlighting the best commercial granites. In addition to these granite coatings, the proposed methodology allows easy and accurate identification of their best performances.

Keywords: Geochemistry; Alterability; Ornamental rock

Resumo

Alguns "granitos negros" foram testados por meio de meteorização ácida simulada (H_2SO_4, HNO_3, H_2SO_4 / HNO_3 e HCl, a pH = 3,00), resistência ao congelamento e perda relativa de brilho. As mesmas placas destes granitos permaneceram imersas em cada solução ácida durante oito ciclos semanais. Os dados de pH de soluções ácidas residuais foram traçados em função do tempo dos ciclos de experimentação em diagramas XY para interpretação como regressões log-lineares. O conjunto global de dados, transcritos em diagrama ternário, mostra uma área distinta destacando os melhores granitos comerciais. Fora destes revestimentos de granitos, a metodologia proposta permite uma identificação fácil e precisa dos seus melhores desempenhos

Palavras-chave: Geoquímica; Alterabilidade; Rocha ornamental

1 Introduction

The so-called "black granites" include several lithologies that have been widely commercialized for many centuries. The most common are gabbros, diabases, basalts, anorthosites, diorites, and metamorphic rocks. *Nero assoluto* is a lithic product whose homogeneous black color is due to a predominance of mafic minerals and very fine texture. A very extensive range of black granites, contains different mineralogical compositions and textures, which

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can influence the performance of external cladding products, leading to unexpected and undesirable results.

In order to predict the behavior of lytic coating plates, particularly those exposed to natural weathering, several authors have investigated the individual behavior of major minerals contained in ornamental rocks (Shen et al. 2019; White & Brantley 2003). Other authors have applied accelerated weathering to rock plates in the laboratory using various acidic solutions (Simão, Leal & Galhano 2020). These processes face an as yet unsurmounted problem: the transposition of each experimental cycle in the laboratory to its corresponding real-time in nature. The difficulty lies in the limited number of variables that can be controlled together in the laboratory, as opposed to a greater number that interfere in nature, producing rock weathering under considerably different environments. More importantly, geological time is not available in the laboratory to produce results.

From a marketing standpoint, in order to overcome the difficulty of selecting products for external lytic coatings, it is assumed that relatively homogeneous black granites behave predictably and similarly against natural weathering. However, from a scientific perspective, it is known that these products may exhibit sufficiently different magmatic evolution to produce markedly different performance under meteoric conditions. This paper studies and compares the performance of some traditional Brazilian black granites marketed as being of "equal quality". This type of experimental study can also contribute to safe and correct application of the rock, avoiding esthetic, structural and negative economic impacts.

2 Samples and Methods

Accelerated acidic weathering of four Brazilian "black granites" (BG1, BG2, BG3 and BG4) was performed using fresh cut plates. These simulated weathering experiments were carried out using standard acidic solutions at pH = 3,00: H_2SO_4, HNO_3, H_2SO_4/HNO_3 (50/50%) and HCl, reacting on two polished plates (5 x 5 x 2 cm) of the "granite" variety. The experiments involved eight weekly cycles with samples immersed in acidic reagent (Ribeiro 2011). A Digimed pHmeter measured the pH of each residual solution. Plates were removed from the acid solutions once a week, washed in deionized water and dried at room temperature. They were then re-submerged in a new acid solution, the same as in the previous immersion. At the beginning (t = 0), middle (t = 28 days) and end (t = 56 days), of the experiments, brightness was measured on the polished surface of the dry plates (average of 27 measurements), using a Sanwa Kenma IG-330-Gloss Checker, expressed as percentage reflectance.

The pH of the residual acidic solutions (variable *y*) and the cumulative time of the reactive steps (variable X) were plotted as linear regressions in order to define the "critical time" throughout the reactive experiments. The "critical time" is reached when the standard acidic reagent displays apparent ineffectiveness over a certain time step, i.e. when the pH of the residual acidic solution is the same as the acidic reagent selected. This condition indicates that hydrolytic corrosion dissolved the most weatherable surface minerals, producing a discernible loss of brightness on the polished plate. This "critical time" is graphically determined in XY diagrams, and indicates possible "granite" performance when submitted to natural weathering. The best products are those whose linear regressions reach the X axis later when Y = 3.

Petrographic analyses were performed to determine the mineralogical composition and classification of the "black granites" studied and select only fresh samples, where textures are considered negligible for an evaluation of commercialized "black granites".

3 Results and Discussion

3.1 Petrographic Data

The modal composition indicates that the selected "granites" are basic rocks (Table 1) from presumably well-preserved quarries. Thus, the influence of textural characteristics (e.g. Navarro 2002; Passchier & Trouw 2005), is not considered in this study.

Table 1 Summary on the modal composition of the studied "black granites".

Black granite	Petrographic classification	Plagioclases	Pyroxenes	Biotite	Hornblende	Accessories (opaques)	Accessories (others)
BG1	Gabbro-norite	63,1	8,9	17,3	6,3	4,4	tr
BG2	Biotite diorite	59,8	14,3	20,4	3,9	1,6	tr
BG3	Diorite-norite	41,8	24,0	17,7	8,0	7,2	1,3
BG4	Ortho-Amphibolite	44,5	---	2,9	48,4	3,5	1,7

Among these rocks, pyroxene participation in whole rock (WR) is always greater than that of amphibole (hornblende), while biotite is relatively constant (17.3 to 20.4% WR), except in the BG4 “granite”. The most striking difference involves the number of feldspars (plagioclases), more important in the BG1 and BG2 varieties than in BG3 (41.8% WR). By contrast, the BG4 variety includes hornblende and plagioclase as dominant minerals, while pyroxene is absent and biotite an accessory mineral.

3.2 Relative Loss of Brightness

The relative loss of brightness (Table 2) reveals discrepant results because the tested plates did not necessarily originate in the same commercialized block.

This reinforces the hypothesis that even so-called homogeneous mafic rocks usually show different facies across their outcrops (e.g. Hyndman 1985; McBirney 2007). This suggests that the technical characterization

Table 2 Summary of medium surface brightness of “black granites” (27 measurements/plate) along different times of the weathering cycles.

Black Granite	∑ Time of Weathering Experimentation / Brightness			Acidic solution pH = 3,00	% Final Relative Loss of Brightness
	T = 0	T = 28 days	T = 56 days		
BG1	75,92	51,92	46,48	H_2SO_4	39
	72,22	65,48	56,14		22
	71,55	56,81	53,81	HNO_3	25
	78,29	59,00	47,51		39
	81,88	59,37	57,59	H_2SO_4/HNO_3	30
	82,92	69,59	63,59		23
	71,18	61,55	49,48	HCl	30
	77,03	60,70	44,70		42
BG2	73,14	53,29	46,22	H_2SO_4	27
	42,74	24,03	23,40		45
	79,92	67,14	57,11	HNO_3	29
	83,24	76,55	66,92		20
	78,85	71,66	55,03	H_2SO_4/HNO_3	30
	87,59	62,70	55,59		37
	80,66	58,41	46,29	HCl	43
	79,62	58,88	50,55		37
BG3	64,33	57,66	52,59	H_2SO_4	18
	64,74	61,92	50,18		22
	48,37	44,03	42,37	HNO_3	12
	50,70	43,96	41,29		19
	54,92	48,70	46,70	H_2SO_4/HNO_3	11
	64,11	54,37	50,55		21
	53,11	47,25	46,03	HCl	13
	79,62	46,77	46,03		42
BG4	63,70	48,81	48,07	H_2SO_4	25
	64,70	54,48	48,18		26
	70,14	55,44	51,18	HNO_3	27
	45,40	34,22	31,11		31
	74,33	62,59	55,18	H_2SO_4/HNO_3	26
	58,74	41,14	38,62		34
	52,55	39,77	35,25	HCl	33
	62,96	43,74	38,81		38

of ornamental rocks is significantly dependent on the representativeness of their tested samples, and often links their characterization to particular facies of a lithic body.

Considering the overall relative loss of brightness (n = 8/tested variety), whose data come from different acid weathering simulations, the performance of most "black granites" would be similar, reaching around 30% loss over their original brightness after 56 days of experimentation (Table 3). By contrast, the "BG3 variety" would be a noteworthy exception and its lower vulnerability to weathering suggests better quality in the tested rocks. This statement applies to mafic rock with greater pyroxene WR participation, i.e., with a corresponding lower proportion (67.5%) of aluminosilicate minerals. Moreover, overall relative loss of brightness shows different responses from different rocks commercialized as "black granite". The stability of minerals against weathering differs between pyroxenes, amphiboles, and biotites, following old concepts established in Bowen's reaction series, later ratified by Goldich (1938). However, regardless of the mineral association, when crystalline structures are physically affected, acidic reactivity will be more effective and faster in these cases. This produces micrometric scraping on the plate surface, causing dispersion in the reflection of incident light that results in a faster decline in brightness.

In agreement, the results showed that the relative loss of brightness is more intense during the first weathering steps (28 days), tending almost invariably to subsequent asymptotic behavior (Figures 1A, B, C and D). This behavior is generally adjustable as first-order linear regression. However, original well-polished plates (higher brightness values) show relative loss of brightness as a logarithmic regression design.

3.3 Simulated Acidic Weathering

Several studies on simulated acidic weathering and the weathering of silicate minerals in nature have been conducted for several decades.

Since silicate minerals are the most abundant in the Earth's crust, their reactive kinetics have strong repercussions on the hydrochemistry of surface waters, as shown by Gibbs (1970), Meybeck (1987), Velbel (1993), Viers et al. (1997) and White and Brantley (2003), among many others. Several factors have influenced the reactive kinetics of major rock-forming silicates (Ollier 1984), mainly their crystallographic nature (Velbel 1999; Brantley 2008), regional rainfall hydrochemistry, climate (White & Blum 1995; Viers et al. 1997), and even the geological time scale (White & Brantley 2003).

Research involving acid attacks on gabbroic natural stones (commercialized as "black granite" because of their lytic nature) can be found in Simão and Silva (1997), Silva and Simão (1997, 2003) and Simão and Carvalho (2005), where the rocks were attacked by acidic solutions in order to observe their alterations as a response to weathering in polluted environments. These changes in mineralogical composition were reported by Silva and Simão (2004), in chemical composition by Galembeck et al. (2009) and Simão and Silva (2003) and in chromatic changes by Galembeck et al. (2009).

In simulated acidic weathering, only a reduced set of variables are considered and jointly controlled. It is deemed impossible to reproduce all possible variables acting in nature in the laboratory, because these variables work over a geological time scale. Nevertheless, the results tend to corroborate the mineral vulnerability predicted by

Table 3 Statistical data about the original brightness and relative loss of brightness of some Brazilian "black granites", under simulated acidic weathering (56 days).

"Black Granite"	Petrographic classification	Some mineral components (%)	Original brightness	Final relative loss of brightness
			AA SD	**AA SD**
BG1	Gabbro-norite	Pyrox. = 8,9 Anf. = 6,3	76,4 4,6	31,3 7,9
BG2	Biotite diorite	Pyrox. = 14,3 Anf. = 3,9	75,7 13,9	33,5 8,5
BG3	Diorite-norite	Pyrox. = 24,0 Anf. = 8,0	60,0 10,3	19,8 9,9
BG4	Ortho-amphibolite	Pyrox. = none Anf. = 48,4	61,6 9,3	30,0 4,7

AA = arithmetic average
SD = standard deviation

Goldich (1938). The best performance of "granite" BG-3, with greater participation of pyroxenes WR, may be due to silica precipitation from these minerals, as demonstrated by Schott et al. (1981) at low pH solutions. This mechanism would produce a protective film on the polished plates, delaying its loss of brightness.

The pH values from the simulated acidic weathering steps (Table 4) are presented in multiple scatterplot diagrams, showing their average values (two tested polished plates through accelerated alterability) versus the cumulative time in the corresponding experiment (Figures 2A, B, C and D).

A first approach to the pH values in scatterplot diagrams confirms that their best adaptable configuration is by log-linear regression. The evidence of higher initial reactivity provoked by the acidic solutions follows that observed during the experiments on relative loss of brightness, that is, regardless of the mineral composition of the rocks, procedures for cutting and polishing the plates may cause deformations in crystallographic structures, generating major reactive surface vulnerability to hydrolysis. The tendency to asymptotic behavior during the subsequent reactive cycles represents the time when an affected crystalline surface is being removed by acid etching.

During the first cycles of reactive kinetics, the number of minerals showing major vulnerability to weathering diminishes on the plate surface. Thus, the consumption of the reagent acid solution declines, considering the short duration of each cycle. The log-linear regression can reach the X axis for pH = 3,00 (Y variable), featuring the same pH values between standard acid solutions and their corresponding residual acidic solutions. From a methodological standpoint, this represents a "critical time" in the weathering experiment, graphically determinable or calculated using the statistical equation of the log-linear regression. The earlier the critical time is reached, the more vulnerable the black granite tested. Pragmatically, at this time point in the simulated experiment, the weathering consequences on the polished plate are well known, affecting its luster and creating an undesirable esthetic aspect, considered unacceptable. As such, this statistical treatment provides values to assess and compare the vulnerability of ornamental rocks under meteoric weathering.

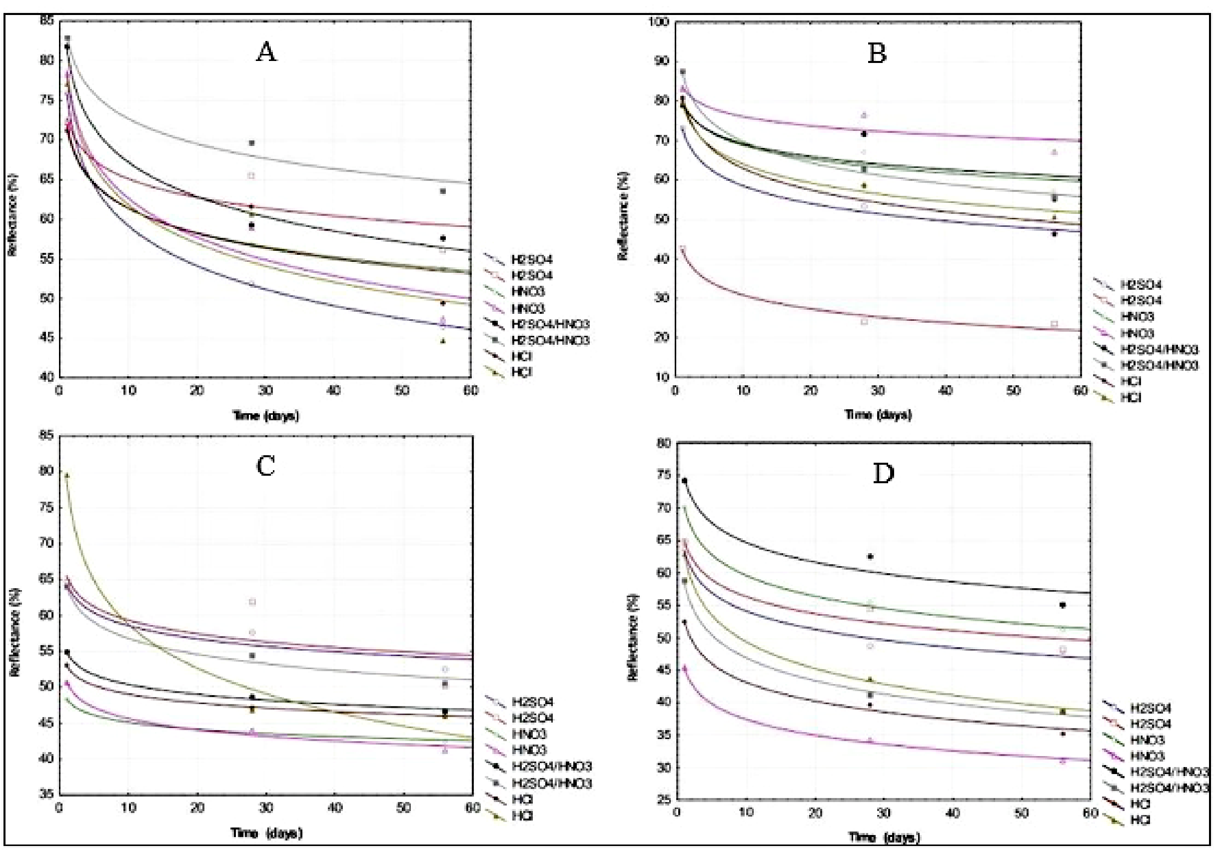

Figure 1 Relative loss of brightness measured in polished plates of granites: A. BG1; B. BG2; C. BG3; D. BG4; during simulated weathering experiments, using different acidic solutions at pH = 3.00.

Table 4 Summary of pH values from different residual acidic solutions along simulated eathering of some Black granites.

	BG1								BG2							
Weeks	**H_2SO_4**		**HNO_3**		**H_2SO_4/HNO_3**		**HCl**		**H_2SO_4**		**HNO_3**		**H_2SO_4/HNO_3**		**HCl**	
	P1	P2	P3	P4	P5	P6	P7	P8	P1	P2	P3	P4	P5	P6	P7	P8
1	4,8	4,6	4,3	4,5	4,5	4,0	4,6	4,2	4,2	4,2	4,3	4,2	4,0	4,0	4,2	4,3
2	4,4	4,2	4,0	4,0	4,1	3,7	4,2	3,8	3,8	3,8	3,8	3,8	3,6	3,6	3,7	3,4
3	4,2	4,0	3,6	3,7	4,1	3,8	4,1	3,9	3,6	3,7	3,6	3,6	3,7	3,7	3,8	3,8
4	4,0	3,8	3,6	3,6	3,8	3,6	3,9	3,7	3,7	3,6	3,6	3,5	3,5	3,5	3,6	3,7
5	3,9	3,7	3,8	3,8	3,8	3,8	3,9	3,7	3,5	3,5	3,6	3,5	3,6	3,5	3,6	3,6
6	3,7	3,6	3,4	3,4	3,6	3,4	3,6	3,4	3,4	3,5	3,3	3,4	3,3	3,4	3,4	3,5
7	3,6	3,5	3,3	3,3	3,5	3,4	3,6	3,5	3,4	3,4	3,3	3,3	3,3	3,3	3,4	3,4
8	3,6	3,5	3,4	3,4	3,4	3,4	3,6	3,5	3,4	3,4	3,3	3,3	3,3	3,3	3,3	3,3

	BG3								BG4							
Weeks	**H_2SO_4**		**HNO_3**		**H_2SO_4/HNO_3**		**HCl**		**H_2SO_4**		**HNO_3**		**H_2SO_4/HNO_3**		**HCl**	
	P1	P2	P3	P4	P5	P6	P7	P8	P1	P2	P3	P4	P5	P6	P7	P8
1	4,2	4,1	4,1	3,8	3,8	3,7	4,0	4,1	3,7	3,6	3,5	3,6	3,5	3,5	3,6	3,5
2	3,8	3,7	3,7	3,6	3,6	3,6	3,6	3,7	3,5	3,5	3,4	3,4	3,2	3,2	3,3	3,4
3	3,6	3,6	3,5	3,6	3,6	3,6	3,6	3,7	3,3	3,3	3,3	3,2	3,4	3,4	3,4	3,4
4	3,4	3,5	3,5	3,5	3,5	3,5	3,5	3,5	3,2	3,2	3,2	3,2	3,2	3,2	3,3	3,3
5	3,6	3,4	3,5	3,5	3,5	3,5	3,5	3,6	3,2	3,2	3,2	3,3	3,2	3,2	3,3	3,3
6	3,4	3,4	3,4	3,3	3,3	3,3	3,4	3,5	3,2	3,2	3,1	3,1	3,2	3,2	3,2	3,2
7	3,4	3,4	3,3	3,3	3,3	3,3	3,4	3,3	3,1	3,1	3,1	3,1	3,1	3,1	3,2	3,2
8	3,4	3,3	3,3	3,3	3,3	3,3	3,3	3,3	3,2	3,1	3,1	3,1	3,1	3,1	3,1	3,1

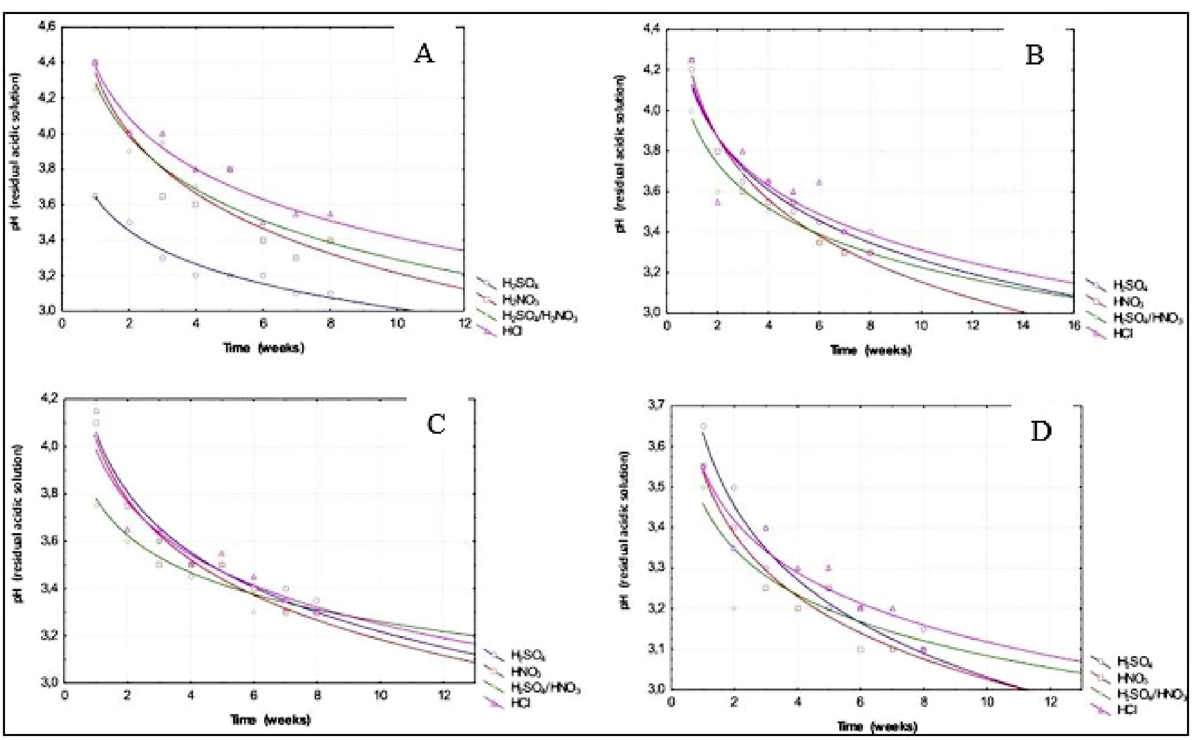

Figure 2 pH variations of residual acidic solutions from simulated weathering of black granites: A. BG1; B. BG2; C. BG3; D. BG4; using differents acidic solutions.

The general behavior of the rocks studied under simulated weathering is similar despite different acidic solution performance and mineralogical composition. In this context, two results can be expected: the linear regression reaches the X axis for pH = 3,00 (variable Y) before the eighth week, or it would need a longer experiment for this to occur, followed by mathematical calculations to determine the "critical time". Pragmatically, these timing values indicate rock performance when exposed to natural conditions. Accordingly, "BG3 granite" performed best, while "BG1 and BG4 granites" exhibited impairments. These conclusions corroborate freeze resistance tests (*in* Cavalcanti 2013) and the relative loss of brightness experiments (Table 5).

Given the widespread availability of "black granites" in a globalized market, practical graphical visualization can be designed through a ternary diagram containing the variables under study here (Figure 3). This diagram displays the domains that characterize the predictability of the best performances of these granites in terms of their exposure in natural settings.

Table 5 Summary of different experiments on the qualitative evaluation of "black granites" under simulated weathering during steps totaling 56 days.

"Black granite"	Final relative loss of brightness (%)	Timing to the critical time (weeks) (*)	Freeze resistance (% medium relative loss of weight)
BG1	31,3	10,5	0,31
BG2	33,5	20,0 (**)	0,22
BG3	19,8	17,5 (**)	0,11
BG4	30,0	11,0	0,35
Reference	This work	This work	Cavalcanti, 2013

(*) chosen acidic solution: H_2SO_4 pH = 3,00
(**) calculated from a log-linear equation mathematics

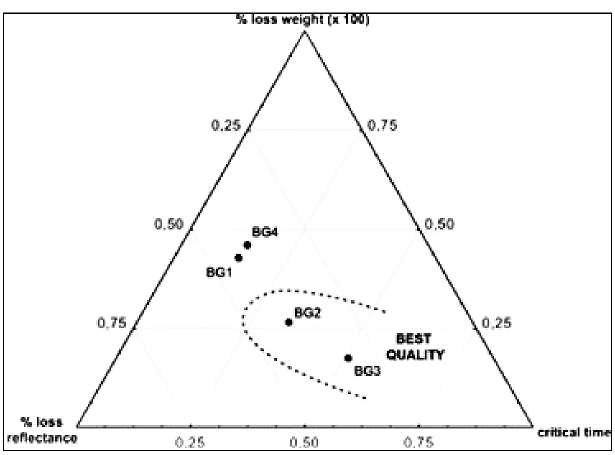

Figure 3 Diagram for predictability of the behavior of "black granites" under meteoric weathering.

4 Conclusion

"Black granites" exhibit a wide lithological variation in peridotites, basalts, gabbros, gabbro-norites, and diorites, since the visual pattern is dominated by dark tones. These rocks display remarkable differences in their mineralogical composition, and different performances under acidic weathering. However, the relative behavior of their polished plates can be evaluated by comparing their performance under simulated weathering, freeze-resistance experiments and loss of brightness, using standard laboratory procedures. The association of this set of parameters was selected because they produce visible damage to the polished plates.

The data produced in the present study were plotted into a triangular diagram that defines specific domains, indicating with better reliability a predictive assessment for "black granites" when exposed to acidic weathering. In terms of esthetic conservation, this methodology allows easy comparison between "black granites", leading to a more reliable assessment of these ornamental rocks. In other words, the method proposed here enables the rejection of low-performance products when selecting "black granites" available on the market, particularly where the application is for external cladding. Considering comparison criteria proposed here for "black granites", the remaining challenge is to estimate as accurately as possible the time it takes for the surface of "black granite" to exhibit unacceptable loss of esthetic quality.

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Ely Brasil de Arruda Luna Cavalcanti: writing; experimental analysis. **José de Araújo Nogueira Neto:** review and editing. **Eldemar de Albuquerque Menor:** review and editing. **Adejardo Francisco da Silva Filho:** review and editing. **Marcelo Reis rodrigues da Silva:** technical support. **Lucas Fontenele Amorim:** formatting.

Conflict of interest

The authors declare no potential conflict of interest.

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