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ACTION OF URINE ON STONE-BUILT HERITAGE

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Abstract

Due to its resistance and durability, stone is the material most used in construction by humanity. The aim of this study was to investigate the action of urine on granites. Accelerated alteration tests were performed on Grey Itaquera, Pink Itupeva, and Black Piracaia granites – stones from the São Paulo State, Brazil, which were widely used in the historical built heritage of the São Paulo state. The test was performed with 30 cycles of partial immersion of nine samples in artificial urine solution and the sanitising of the specimens, totalling 60 consecutive days of testing. In the first 24 hours, the samples were partially immersed in urine; in the following 24 hours, the samples were sanitised in three different groups: A) without washing; B) washing with natural water; C) washing with a 5:1 solution of natural water with bleach. The alterations induced in the specimens were evaluated by spectrophotometry, P-wave velocity, and scanning electron microscopy. The main results obtained were chromatic alterations and crystallisation of salts on the three types of granite. The study concluded that urine is a substance that can cause moderate alterations in the aesthetics and structure of granitic stones.

***Keywords**: Urine; Heritage; Granite; Accelerated alteration; Colour*

Introduction

Since the beginnings of humanity, stone has been the most used construction material due to its characteristics of durability and resistance. With the advance of technology, it has accompanied the development on construction techniques for the formation, growth and accelerated urban development of cities. Besides its use in the building of cities, this material also has also found wide application in the construction of monuments and decorative elements.

In order to preserve and conserve stone-built heritage, it is necessary to investigate the intrinsic and extrinsic characteristics of the stones, since they can contribute to the appearance of forms of degradation found in historical monuments.

The various processes of alteration and degradation of stone-built materials are closely related to the intrinsic properties of the materials themselves, such as the presence of carbonates, sulphates, swelling clay minerals, as well as the degree of fracturing, faulting and porosity in the stones [1-10].

The façades of buildings and historical monuments are subjected to the constant action of exogenous agents, such as temperature variation, the actions of biocolonisers and pollution, the presence of salts and humidity, as well as the action of urine [11]. The evaluation of the impact of this solution on stones is still lacking in academic circles.

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Due to the severe economic crisis in Brazil, which has generated mass unemployment, a major socio-economic problem has been created in the urban centres of the major state capitals, mainly due to the inefficient public policies of aid for the unemployed, which have led to them becoming homeless street dwellers. As such, there is a significant number of people sleeping outdoors or in encampments in urban centres, and a serious problem is the cleanliness and hygiene of this population under non-existent sanitary conditions, which force these residents to meet their physiological needs in the streets.

The aim of this research was to investigate forms of degradation in granitic rocks that constitute monuments and clad façades in the old centre of São Paulo City due to the action of urine. To achieve this goal, a laboratory study was carried out in which samples were partially immersed in artificial urine.

Experimental part

Materials

The three granitic rocks analyzed—Itaquera Granite, Itupeva Granite, and Piracaia Granite—were selected for their chromatic and petrographic characteristics and due to the widespread presence of these granites in the façades of buildings and monuments built at the beginning of the 20th century in the central region of São Paulo City, Brazil (Fig. 1).

Itaquera Granite (ITA)

According to the petrographic characterization, this rock was classified as biotite granite, grey in colour, with a fine grain, a slightly deformed structure, and an orientation of micaceous minerals. The mineralogical composition obtained was: microcline (35%), quartz (30%), plagioclase (20%), hornblende (8%), biotite (5%), muscovite (2%), and accessory minerals (titanite, apatite, fluorite, opaques).

This granite was widely used in the buildings in the old centre of São Paulo City, including the Obelisk of the Memory, the oldest monument in the city, dating from 1814, and the foundations or façades of the Shopping Center Light, the São Bento Monastery, the Department of Justice, the Municipal Market, House No. 1, and the Float Valve House, among others [12]. It was also used in the monuments Nostalgia, Aretuza, The Girl and the Calf, Monument to Ramos de Azevedo, The Reaper, After the Bath, and Faun, among others [13]. The old source quarry is now shut down and filled in.

Itupeva Granite (ITU)

This rock has a reddish pink colour due to the presence of potassium feldspar, an equigranular structure, and a fine-to-medium texture. The microscopic analysis provided the following mineralogical composition: microcline (36%), quartz (31%), plagioclase (21%), biotite (10%), muscovite (2%), and chlorite as a secondary mineral.

Nowadays, Itupeva Granite is used for the production of crushed stone, as is the case with the Viracopos quarry in the city of Itupeva. Some examples of foundations or façades of buildings made of this granite are: the Court of Justice of the State of São Paulo, the Palace of Justice, the Banco do Brasil Cultural Centre (CCBB), among others, as well as the Monument to Lebanese-Syrian Friendship, the Monument to the Founders of São Paulo, and Immortal Glory to the Founders of São Paulo, among others.

Piracaia Granite (PI)

The microscopic analysis of this rock provided the following composition: plagioclase (30%), microcline (28%), biotite (25%), hornblende (7%), quartz (5%), and titanite (3%). Due to the small concentration or even absence of mineral quartz in its constitution, this rock is not granite but monzonite, according to the nomenclature of igneous rocks. However, since the commercial name of this rock is Piracaia Granite, in this work we chose to keep the commercial name. This rock is made up of a high concentration of mafic minerals, which give it its dark colour. When unpolished, it has a dark grey colour; when polished, its colour is characteristically black.

Fig. 1. Examples of sound rocks and their respective uses in stone-built heritage A) Block of Itaquera Granite B) Façade of the São Bento Monastery, made of Itaquera Granite C) Block of Itupeva Granite D) Lower façade and foundation of the Banco do Brasil Cultural Centre (CCBB) with unpolished and polished Itupeva Granite, respectively. E) Block of Piracaia Granite F) A tomb consisting of Piracaia granite located in the Consolação Cemetery

Macroscopically, the rock presents a massive structure of fine granulation with strips of plagioclase that can reach up to 2cm, scattered without orientation in the matrix.

Piracaia Granite is very similar to Black Bragança Granite, both of which belong to the same suite as the Socorro Complex [14]. Black Bragança Granite is characterised by the presence of plagioclase crystals in the form of scattered white spots in the matrix, while in Piracaia they occur as pinkish-white strips, also randomly dispersed in the matrix. For restoration projects, both granites present the same behaviour, so it has been assumed in this work that the black granites found in the monuments of São Paulo coming from this granitic suite are denominated Piracaia Granite.

Some examples of buildings clad with Piracaia Granite can be observed in the Caixa Econômica Cultural Centre, Counselor Crispiniano Street, the Sampaio Moreira building, and tombs in the Consolação Cemetery, among others.

Methods

Samples of the granites were collected from active and inactive quarries, the latter in the case of Itaquera Granite.

The tests performed were petrographic characterization, ultrasound, spectrophotometry, scanning electron microscopy, and accelerated alteration.

Three cubic samples of each granite with 7cm edge dimensions were used for the accelerated-alteration test, making nine samples in total.

Petrographic characterization

The petrographic analysis of the granites aimed to classify them and describe their mineralogy. The Olympus BX-40 microscope at the Laboratory of Optical Microscopy of the Institute of Geosciences of the University of São Paulo (IGc-USP) was used.

Ultrasound

The P-wave velocity was measured to evaluate possible changes in the internal structural characteristics of the rocks. The readings were performed before and after the accelerated-alteration test at the Institute of Technological Research (IPT-SP), using the Pundit MPS device with 150kHz transducers.

Spectrophotometry

The spectrophotometer was used for colour measurement prior to testing and evaluation of possible chromatic changes in materials after the alteration test. The Konica Minolta spectrophotometer model CM-2500D of the Cultural Heritage Studies Laboratory of IGc-USP was used.

Scanning electron microscopy (SEM)

SEM was used to determine the composition of the salts formed on the surfaces of the specimens during the accelerated-alteration test. The analyses were performed at the Laboratory of High-Resolution Geochronology of the IGc-USP.

Accelerated alteration

This test was performed by partial immersion of samples in artificial urine. This solution was chosen for health reasons and for its constant composition, since natural urine contains variable biological components.

Artificial urine was manufactured at the Chemistry/ICP-MS Laboratory of the IGc-USP in accordance with the proposal of *S. Chutipongtanate and V. Thongboonkerd* [15]. The test was performed for 60 consecutive days, giving 30 cycles of 48 hours each. Each cycle was divided into two stages.

In the first 24 hours, the nine specimens were partially immersed in artificial urine. Each sample remained isolated in an individual container to avoid possible cross-contamination. After the first day of each cycle, the solutions were removed from the containers and discarded. Each sample of granite was assigned to a cleaning group: In the first set, the samples were not sanitised (ITA-A, ITU-A, PI-A); in the second, the samples were washed with natural tap water only (ITA-B, ITU-B, PI-B); in the third group, the specimens (ITA-C, ITU-C, PI-C) were sanitised with a mixture of tap water and bleach (sodium hypochlorite, NaOCl) in a 5:1 ratio.

In the other 24 hours of the cycle, the samples were set out to dry on a bench exposed to sunlight for much of the day, thus completing the cycle. The samples were always immersed in the same position.

Results

The results obtained from these tests—accelerated alteration, measurement of the P-wave velocity, determination of the chromatic parameters by spectrometry, and identification of the salts formed using SEM—are presented below.

Accelerated alteration

The partial-immersion alteration test in artificial urine solution was performed to investigate the action of this solution on three granitic rocks since this type of investigation is very seldom carried out and urine may make a significant contribution to the alteration of the stones.

Alterations in the tested samples were observed during the 60-day accelerated-alteration test with partial immersion in artificial urine. The first alterations (Fig. 2) observed during the alteration test were bleaching in Itupeva and Itaquera granites and localised yellowing in one specimen of Itaquera Granite.

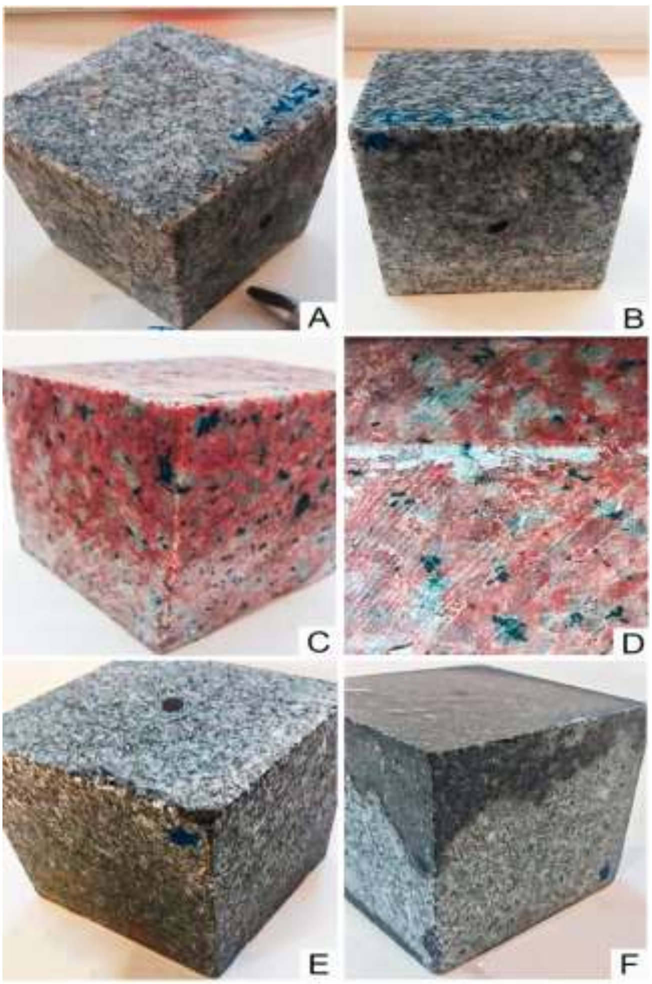

Fig. 2. Changes observed during and after the experiment. A) Yellowing at the vertices of the non-immersed faces of the Itaquera Granite specimen. B) Whitening on the immersed portion of the Itaquera Granite sample. C) Bleaching on the immersed portion of the Itupeva Granite sample. D) Salts crystallised on the lateral face of the Itupeva Granite sample, following the level of the urine solution. E) Fragmentation of the vertex of the Piracaia Granite sample. F) Intense dampness of the immersed portion of the Piracaia Granite sample

As the cycles progressed, bleaching gradually became more intense, and there was the appearance of crystallised salts and increased yellowing of the three granites. Darkening and salt formation were also observed at the contact between the immersed and non-immersed portions of the three lithotypes.

The Piracaia Granite specimens presented moisture even after the 24-hour drying period.

Ten days after the end of the experiment, new alterations were observed in the samples: Itaquera Granite: appearance of salts at the levels of urine solution, reduction of bleaching, and appearance of yellowish stains; Itupeva Granite: increased bleaching, appearance of salts at the solution line, and decreased staining; Piracaia Granite: darkening and salt formation at the level of the urine solution, staining, and absence of moisture.

After the accelerated-alteration test had been performed, the nine samples were left on the laboratory bench for one year. During this time, visual analysis of the specimens indicated new alterations: Itaquera Granite presented an increase in yellowing, no salt formation was noted, and there was attenuation of the mark of the urine solution line. Itupeva Granite presented new marks from the urine solution; there was a more abundant appearance of salts at the level of the urine solution and bleaching of the immersed portion of ITU-B. Piracaia Granite presented the appearance of salts, darkening of the part that was not immersed, and the non-disappearance of the mark of the urine solution line.

Ultrasound

The test for determining the P-wave velocity was performed in three distinct phases: one before the accelerated-alteration test, a second after the end of the experiment, and a third one year after performing the test. Measurements were performed in directions parallel and perpendicular to the urine-solution mark (Table 1).

Table 1. P-wave velocities obtained in the specimens in parallel and perpendicular directions

Specimen	Before test (m/s)	After test (m/s)	Percentage change in velocity (%)	One year after the end of the test (m/s)	Percentage change in velocity (%)
Parallel direction					
ITA-A	4,090	4,323	**5.7**	5,000	**15.7**
ITA-B	3,990	4,180	**4.8**	4,800	**14.8**
ITA-C	3,940	4,011	**1.8**	4,900	**22.2**
ITU-A	4,900	5,148	**5.1**	5,000	**-2.9**
ITU-B	4,770	5,068	**6.2**	4,800	**-5.3**
ITU-C	4,770	4,607	**-3.4**	4,700	**2.0**
PI-A	5,100	5,175	**1.5**	5,100	**-1.4**
PI-B	5,240	5,201	**-0.7**	5,100	**-1.9**
PI-C	5,430	5,520	**1.7**	5,400	**-2.2**
Perpendicular direction					
ITA-A	-	4,750	-	5,300	**12.0**
ITA-B	-	4,851	-	5,400	**11.0**
ITA-C	-	4,689	-	5,200	**11.0**
ITU-A	-	4,936	-	4,800	**-3.0**
ITU-B	-	5,179	-	4,400	**-15.0**
ITU-C	-	5,256	-	4,700	**-11.0**
PI-A	-	5,454	-	5,400	**-1.0**
PI-B	-	5,181	-	5,500	**6.0**
PI-C	-	5,191	-	5,100	**-2.0**

In the measurements taken in the direction parallel to the urine-solution mark, the granites indicated different behaviours:

– Itaquera Granite revealed an increase in velocity after the alteration test and an even greater increase one year after the end of the test, reaching an increase of up to 22% (ITA-C);

– Itupeva Granite revealed an increase in the velocity for ITU-A and ITU-B after the test and a decrease in the velocity one year after the experiment. The ITU-C sample had the opposite behaviour;

– Piracaia Granite revealed practically no change in velocity in the various sanitization groups.

In the direction perpendicular to the urine solution mark, the following behaviours were observed:

– Itaquera Granite revealed an increase in velocity for the three sanitization groups;

– Itupeva Granite presented a velocity reduction;

– Piracaia Granite presented stable velocity values for the non-sanitised and bleach-sanitised samples, and there was an increase in the velocity (6%) in the sample sanitised with tap water.

Spectrophotometry

The chromatic parameters [16–20] of the rocks before and after the accelerated alteration test are shown in Table 2.

Table 2. Spectrophotometric readings (mean values) obtained for the three granites studied.

Location of readings	CIElab						ΔE
	L*	a*	b*	L*	a*	b*	
	BEFORE EXPERIMENT			AFTER EXPERIMENT			
Solution level	67.2	-0.4	-0.4	60.9	-0.3	-0.3	6.3
Not immersed	66.2	-0.4	-0.4	62.3	-0.4	-0.2	3.9
Immersed	67.5	-0.4	-0.9	64.9	-0.5	-1	2.6
Solution level	67.0	-0.4	-1.5	63.9	-0.5	-0.2	3.4
Not immersed	67.4	-0.5	-2.2	61.7	-0.5	-1.1	5.8
Immersed	68.2	-0.5	-1.9	65.9	-0.6	-1.2	2.4
Solution level	69.3	-0.6	-2.4	63	-0.6	-0.9	6.5
Not immersed	69.0	-0.6	-2.2	64.6	-0.6	-1.3	4.5
Immersed	70.0	-0.6	-2.4	68.6	-0.6	-1.7	1.6
Solution level	62.7	4.3	3.9	55.9	5.4	5.8	7.1
Not immersed	61.9	4.6	3.9	54.6	6.1	6.8	8.0
Immersed	61.9	4.3	3.4	58.3	5.5	5.9	4.5
Solution level	62.2	4.8	4	63.3	3.4	3.4	1.9
Not immersed	61.6	4.6	3.7	56.7	5.7	6	5.5
Immersed	61.4	4.4	3.6	58.8	4.8	4.7	2.8
Solution level	59.7	5.2	4.2	54.1	6.4	6.8	6.3
Not immersed	61.5	4.7	3.7	54.4	6.4	7	8.0
Immersed	60.1	4.7	3.7	57.8	5	5	2.7
Solution level	49.2	-1.3	-0.4	47	-1.4	-1.6	2.5
Not immersed	49.4	-1.4	-0.8	44.5	-1.2	-0.3	4.9
Immersed	50.0	-1.5	-0.9	48.1	-1.4	-0.9	1.9
Solution level	54.4	-1.5	-0.5	48	-1.3	-0.6	6.4
Not immersed	53.6	-1.5	-0.7	46.7	-1.4	-0.3	6.9
Immersed	51.3	-1.4	-0.5	49.3	-1.4	-0.9	2.0
Solution level	53.3	-1.5	-1.2	46.9	-1.3	-0.6	6.4
Not immersed	50.8	-1.5	-0.7	46.9	-1.3	-0.3	3.9
Immersed	49.6	-1.5	-1.2	47.2	-1.5	-1	2.4

The granites presented the following chromatic variations:

– Itaquera Granite had a darkening of all samples, yellowing of ITA-B and ITA-C samples, and a greater total colour difference (ΔE*) at the level of the urine solution and in the non-immersed portion;

– Itupeva Granite had a darkening, reddening, and yellowing of samples, a greater total colour difference (ΔE*) in the non-immersed portion and at the solution level in ITU-A and ITU-C samples, and only in the non-immersed portion of ITU-B. The ITU-B sample had anomalous behaviour;

– Piracaia Granite exhibited darkening and stability of the a* parameter. The b* parameter varied slightly, sometimes increasing and sometimes decreasing at the solution level and in the non-immersed part. There was a greater total difference in colour (ΔE*) at the solution level in PI-B and PI-C.

SEM

The salts were collected from the water-sanitised samples due to the greater amount of these materials on the ITA-B, ITU-B, and PI-B specimens and analysed by SEM. The three samples presented salts with a powdery appearance, with the presence of crystals scattered in the powdery matrix. Peaks were detected for phosphorus (P), chlorine (Cl), sulphur (S), calcium (Ca), magnesium (Mg), sodium (Na), and potassium (K).

Discussions

The alterations observed in the three lithotypes studied are discussed below.

Itaquera Granite

During the test, it was observed that this lithology presented an intensification of the yellowing of the samples as the cycles progressed, from localised alterations to alterations that were disseminated to the other faces of the samples. This chromatic change is ratified by the increased value of the b* parameter. A.M.S. Gimenez [21] reported that, in his study, spectrophotometric data revealed changes in the colours of granitic rocks, among them Itaquera Granite, caused by hydrochloric acid, sulphuric acid, and nitric acid. The samples tested with hydrochloric acid showed more intense yellowing, while those with nitric acid had less yellowing. The yellowing observed in this study is related to the exposure to urine as well as to the bleach used to sanitise the samples.

Itaquera Granite showed an increase in the P-wave velocity. The highest percentage increase occurred in the direction parallel to the urine-solution line in a sample from the group sanitised with the water solution with bleach, from 4,011 m/s before the experiment to 4,900 m/s one year after the end of the accelerated-alteration test. This same specimen had practically no change at the end of the experiment (only a 1.8% increase). This increase was evident only one year after the end of the experiment. The considerable increase in velocity in both directions indicates a probable crystallisation of salts in the pre-existing voids in the samples.

The velocity of Itaquera Granite was previously determined by some authors. D. Grossi and E.A. Del Lama [22] obtained a velocity of 4.3km/s in the Monument to Ramos de Azevedo. In partial-immersion tests with sulphuric, hydrochloric, and nitric acids and sodium hydroxide, A.M.S. Gimenez [21] obtained velocities between 4.09 and 4.31km/s. E.A. Del Lama et al. [23] obtained velocities between 4.1 and 5.0km/s for sound rock. In comparison with the data reported in the literature, the P-wave velocity obtained in the partial-immersion test with urine solution performed in this work indicated that it was at the average of the velocities obtained from sound rock.

Itupeva Granite

There were chromatic alterations in the samples tested, mainly in the non-immersed portions, in which strong whitening was observed. The presence of salts at the solution line emerged after the end of the test and remained after one year of the experiment, with a significant increase in the salts and whitening of this region.

Itupeva Granite indicated yellowing of the samples seen in the increase in the values of the b* parameter, which varied from an average of 3.8 (before) to 5.9 (after the test), and a tendency to reddening seen in the increase in the measurements of the a* parameter, which varied between 4.8 (before) and 5.6 (after the test). A.T. Suzuki [24] performed spectrophotometric readings at the Palace of Justice of São Paulo and obtained means of 7.1 and 11.8 for the a* and b* parameters, respectively. Thus, it was observed that the author's readings were higher than those obtained during the present study, indicating different petrographic facies of the granite, as discussed in *F.P. Araujo et al.* [25].

In the parallel direction, there was an increase of up to 6.2% in the P-wave velocity when compared to measurements obtained before (4,770m/s) and after the test (5,068m/s), and a reduction of up to 5.3%, from readings taken at the end of the test (5,068m/s) to those taken after one year (4,800m/s). In the perpendicular direction, there was a reduction of up to 15% in the P-wave velocity according to the readings obtained after the experiment (5,179m/s) and in the following year (4,400m/s). The data indicates that there was crystallisation of salts in the pre-existing voids in the samples, but that the salts ended up solubilising after 1 year of testing, returning the velocity to the initial values.

The values of ΔE^* were on average more than 3.0 for the Itaquera and Itupeva granites, except for the immersed part, where the values are below 3.0. *R.S. Berns* [26] points out that $\Delta E^* > 3$ is noticeable to the human eye, corroborating our observations of chromatic alterations clearly visible on the surfaces of the samples.

Piracaia Granite

No chromatic alterations were observed at the beginning of the test. As the cycles progressed, salts appeared on the samples, with an increased presence mainly at the urine-solution lines. One year after the trial, the darkening of the non-immersed portions was still evident, in addition to the continued presence of salts on all samples.

No significant changes were observed in the readings taken with the spectrophotometer for the a* parameter. For the b* parameter, there was an increase in the values for PI-C at the urine-solution level and in the non-immersed portion of PI-A. A.M.S. *Gimenez* [21] did not present changes in the a* parameter readings, with an increase in the yellow band of the sample exposed to nitric acid and on the sides of the sample immersed in sulphuric acid. *A.P. Meyer et al.* [27] found discoloration and corrosion due to attack by hydrochloric acid, a slight loss of colour with citric acid, and little susceptibility to the action of ammonium chloride. In contrast, Piracaia Granite changed less in colour, indicating that the aforementioned acids are more effective in causing chromatic change.

On some faces of the black granite samples, ΔE^* values obtained were below 3.0, a limit non-noticeable to the human eye, so no chromatic changes were obvious to the naked eye.

Piracaia Granite indicated a maximum increase of 6% of the P-wave velocity in only one sample, in the perpendicular direction, in which measurements of 5,181 m/s (after the test) and 5,500m/s (after one year) were obtained. It should be noted that the other samples remained practically unchanged in parallel and perpendicular directions. *A.M. Godoy and J.C.P. Arrais* [14] measured the P-wave velocity in the three types of Piracaia Granite: fine granulation (5,455m/s), fine-to-medium granulation (5,318m/s), and medium granulation (5,183m/s)—readings consistent with the values found here.

The elements P, Cl, S, Ca, Mg, Na, and K detected in the samples of the three granites by SEM indicated their probable origin in the urine solution. The presence of magnesium phosphate (a material removed from ITU-B) was also observed. This is not usual in granite and therefore comes from urine.

Conclusions

This study was carried out with the objective of evaluating the action of artificial human urine in three granitic lithotypes that were widely used in the building of the historical heritage of the old centre of São Paulo City.

The granitic rocks studied indicated moderate resistance to the action of urine, but it has been proven that this substance causes changes in rock, both in aesthetics by means of chromatic alterations and the formation of efflorescence, as well as in the presence of structural damage caused by salts that can alter the resistance and integrity of the stone in the medium and long term.

After the end of the test, two representative points were observed: Itupeva Granite was the lithotype that presented the highest chromatic change. Among the sanitising groups, tap water indicated greater efficacy for urine removal.

It should be noted that biological components are present in human urine that can modify its colour, thus directly affecting the action on the stone, which may cause staining and/or (sub)efflorescence formation.

Attention should be paid to the improper use of cleaning products for the removal of urine from pavements and buildings, which can also directly affect the physical and aesthetic integrity of the stones.

Thus, it can be concluded that, among some degradation features observed in the façades of buildings and monuments of the old centre of São Paulo City, such as chromatic alteration, the presence of saline efflorescences, and detachment, some are possibly due to or contributed to by human urine, especially when located in their foundations.

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