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Alteration, evaluation and use of extremaduran granite residues

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Abstract The necessity of eliminating debris from a granite quarry has awakened an interest in applications of by-products, called ‘‘marginal arids’’, in different fields, like construction and foundations for roadways, restoration, material for the manufacture of artificial rocks, and artesian products etc. Conclusions obtained from the results of tests carried out by X-ray diffraction of granite quarry by-products in Extremadura, Spain, submitted to different treatments, are established. Test pieces from two quarries are analyzed and compared generally and specifically, for commercial use. Finally, conclusions relating to essays in test pieces and mineral dynamics of marginal arid granite are exposed.

Keywords Granite · By-products · Mineralogy · Test pieces · X-ray diffraction · Recycling · Construction · Extremadura · Spain

Introduction

Extraction activities in the granite mining industry brings an enormous impact upon the environment (Babiano et al. 1987; Rau and Wooten 1980). This can be combated in Extremadura (Spain) by recycling the by-products to restore the affected spaces (Del Val 1994).

Granite production in Extremadura in 2004 totalled 360,000 tons in 151 mining areas. The current cost for eliminating residues generated in the quarries and the industries that transform the stone can reach more than 70% value of the extracted rock volume. This makes reutilizing these residues attractive economically, given that they could be totally recycled.

Reducing debris from a granite quarry has awakened an interest in the by-products, called ‘‘marginal arids’’ (Gómez Mateo 1994) for applications in different fields, like construction and foundations for roadways, restoration, obtaining material for the manufacture of artificial rocks, and artesian products etc. (Rincon and Romero 1996; Arm 2001). It is well known that some uses of granite by-products or granite chips are composite floor tiles, composite kitchen counters, bathroom and public building walls, landscape stone and blocks, polishing equipment etc. The granite residues as muds have been considered as partial sustitute of feldspar component in porcelainized stoneware tile production due to its homogeneity in composition and granulometry (Hernández-Crespo et al. 2002).

New applications for these sub-products, different finishes, textures and durability are reviewed. The alteration of ornamental artificial rocks and their durability when submitted to different accelerated aging processes (Pisciella et al. 2001; Rodriguez et al. 1997) is studied by applying superficial treatments to the finishes and evaluating the degradation due to environmental pollution. Composites are sometimes as strong as natural stone, but are never as durable-more brittle, more susceptible to environmental damage (Rodriguez et al. 1999). New uses for granite by-products include rehabilitation or incorporation of different treatments using both natural and artificial rocks (García Guinea and Martínez Frías 1992).

Natural rocks have been used in construction throughout much of history, mainly due to their insulating capacity, durability, practicality and aesthetic beauty (Martin 1990).

Today, ornamental stone is used primarily in building and construction for about 80% of its total production. The remaining 20% is dedicated to funeral art and urban decoration (industry, walking paths, gardens, etc.), and in ornamental objects (Mas Serra 1988).

The knowledge of the intrinsic characteristics and the evolution of ornamental stone material, depending upon the surroundings in which they are exposed (Boix et al. 2001; Gómez et al. 2004), is just as important for a reasonable treatment in the ample historic patrimony built in stone, as is to assure the quality in new construction that will make up the patrimony of the future.

In the restoration or rehabilitation of natural stone monuments and buildings problems arise with the introduction of new materials that may differ, due to the difference in age or contamination (Mingarro Martín et al. 1996). The way in which a stone will evolve when exposed to the extrinsic factors of material (water, contamination, rapid changes in temperature, etc.) depends upon its intrinsic characteristics and environmental conditions where it is sited (Pelino 2000; Arm 2001). It is necessary to know the nature of the material to forecast its evolution: The natural chemistry and mineralogy, homogeneity, texture, structure, etc., of a stone conditions, its behaviour (Pisciella et al. 2001).

The objective of this investigation is the characterization of slabs manufactured with arid marginals from granite by-products. These by-products are crushed and ground in grinding plants located near the quarries. A present no specific uses exist for these materials after being ground up (Gómez Mateo 1994). There is a great interest about possible uses for these arid marginals because of the ecological impact of the immense quantities of residues and leftovers produced by quarries. The magnitude of these residues is truly inconceivable without visiting these sites and contemplating this man-made debris left behind (Solaz and Calvo 1991a).

One of the uses for arid marginals is the manufacture of slabs and constructive elements used in buildings and civil works, which have similar finishes to that of natural stone but with a greatly lower cost (Solaz and Calvo 1991b).

To summarize, the primary objectives are to investigate ideal conditions to obtain marginal arids from the initial granite subproducts, to apply accelerated aging processes via surface treatments to characterize mineralogically the aged granites, and to minimize environmental impacts produced by these residues in the immediate surroundings of granite quarries.

Materials and methods

Sources and preparation of the natural granite test pieces

The initial test-pieces originate from two different quarries: The La Lagunilla meadow quarry, located to the south of Quintana de la Serena, and the Boyal meadow quarry, located northeast of Quintana (Fig. 1).

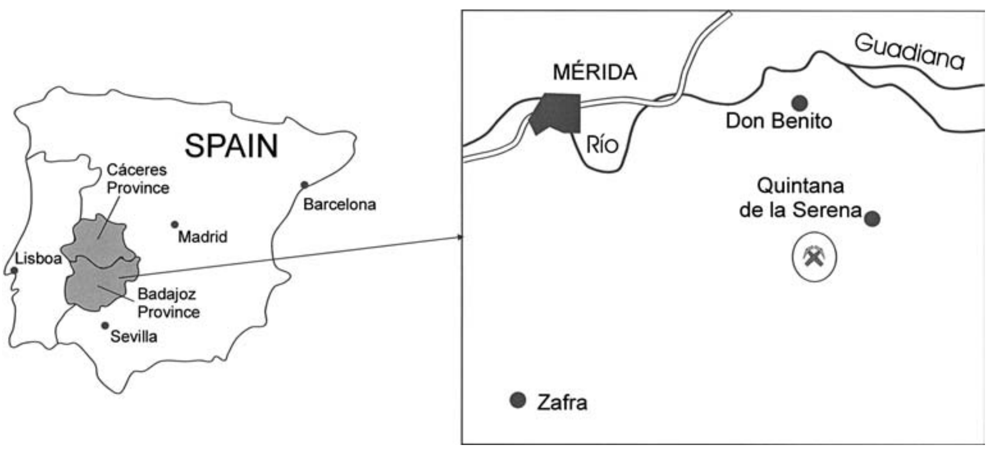

Fig. 1 Geographical location of the study area

Both zones are in the Quintana de la Serena municipality, Badajoz. From these zones, the granite variety *Gris Quintana* is extracted (Solaz and Calvo 1991a).

Test-pieces possessing several different finishes were obtained from each of the quarries. Experiments were conducted to explore the distinct aging forms associated with surface treatments.

La Lagunilla

There are seven slabs with different finishes (Table 1), each having approximate dimensions of 3×6×6 cm^3. The slabs have only one face polished, with the exception of No. 5 (bush hammered or carved stone 9 third phase) that has a 5 L finish on its top edge and an 8 L finish (sawed) on the opposite face. A total of 63 test-pieces were used (nine test-pieces × seven slabs), with the same results for test-pieces 5 and 8 L. As these finishes on 5 and 8 L to the same test-piece, a total of eight real test pieces were used.

Test pieces having bush hammered finishes are made with bush hammers that depending upon the depth and size of the crater, are formed in a series of the numbers 2, 4, 5, 7, 9, etc., realized with the help of a punch that is, respectively, 2×2, 4×4, etc. The depth and size increases, as the number of punches decrease.

Boyal

There are eight slabs from the Boyal meadow with different finishes (Table 2), each having approximate dimensions of 3×6×6 cm^3, the same as for La Lagunilla. Nine test-pieces with dimensions of 3×3×3 cm^3, were obtained from each slab. Each has only one edge finished. For La Lagunilla, there are 72 test-pieces (nine test-pieces × eight slabs). To summarize, a total of 135 test-pieces were examined, 63 from the La Lagunilla meadow quarry and 72 from the Boyal meadow quarry.

Once the final test-pieces were identified, characterization began (AENOR 1997; Calvo et al. 1991), by determining their weight, real and apparent densities, porosity etc.

Table 1 La Lagunilla quarry finishes

Test-piece No.	Finish	Legend
1	Natural cut	1L
2	Hand cut	2L
3	Bush hammered or carved stone 5 first phase	3L
4	Bush hammered or carved stone 7 second phase	4L
5	Bush hammered or carved stone 9 third phase	5L
6	Thermal finish	6L
7	Polished	7L
8	Sawed	8L

Soluble salt immersion resistance, RILEM test, modified

The soluble salt immersion test consists of introducing test-pieces into a soluble salt bath. The soluble salt test is also called the RILEM. The salt utilized is Na_2SO_4, 10 H_2O, which dissolves in distilled water; and once dissolved, it is introduced into the pores of the test-pieces by submerging them in the aforementioned dissolution. Once the pores contain the dissolution, the test-pieces are dried to eliminate the water contained within. The dehydrated salt settles and with this, its volume changes. These volume changes degrade the pores in the test-piece and the test-piece itself as well. During the immersion of the test-pieces in the saline solution the bath is subjected to ultrasonic waves. This variation was introduced to the original RILEM test (Liso et al. 2001). So that the salt does not settle in the bath bottom, and that there is not early sedimentation upon the test-piece surfaces.

Resistance immersion in acids

One of the ways stones exposed in the environment is reduction by acids. These reagents have an atmospheric origin and are formed by environmental pollution. These agents produce chemical changes in the rock, altering its form and composition (Liso et al. 1999, 2000a). These alterations include oxidation, carbonation, sulphatation, nitrification, etc. The tests were accelerated by using concentrations much higher that can be found naturally. The procedure used is norm UNE 22-198-55 (AENOR 1985). The reagents used were H_2SO_4, HCl, HNO_3, FH (the norm only specifies H_2SO_4). For confirmation of no alteration, a test was made with NH_3, a base.

Mineralogy of the samples

X-ray diffraction (DRX) was performed with either the dust or the Debye-Scherrer technique. The difractogram

Table 2 Boyal quarry finishes

Test-piece No.	Finish	Legend
1	Natural cut	1B
2	Bush hammered con bush hammer 7×7	2B
3	Puntero	3B
4	Arenization	4B
5	Polished	5B
6	Thermal finish	6B
7	Apomazado	7B
8	Cleaned or sawed	8B

is collected with an electronic device that produces a diffraction spectrum where peaks originated by families of planes of the different minerals in the sample appear, proportional to the percentages of the minerals of the sample total, but also dependent upon the "reflecting power" of the X-ray of each mineral type (Martín Pozas 1968; Aylon 1974). The reflective power utilized with the diffraction equipment possessing automatic slots is presented in Table 3 (Liso et al. 2000a, 2001b).

Results and discussion

The results of the semi-quantitive analysis of the X-ray difractograms are explained next. The data presented make reference to the percentages of the most abundant minerals present in the tested granites. The data will be expressed by quarry source, the test performed upon them, and by the kind of finish each possesses.

It can be seen that unlike the La Lagunilla quarry, the mica does not disappear in the FH tests, nor is it as reduced as in the HNO_3 tests. The percentage of mica in the Boyal quarry test-pieces is superior than the percentages obtained in the La Lagunilla quarry, and because of this, that although the mica is attacked equally in both quarries in one it does not disappear (Vázquez and Jimenez-Millán 2001). This is due its greater content in the Boyal quarry, while in the La Lagunilla quarry, it either disappears entirely or its percentage greatly reduces.

It can be seen, like in this case, the micas have completely disappeared because the sample analyzed belongs to the weathered residues during the HNO_3 immersion test in both quarries. The alteration has been very high, indicating that in longer immersions, the mica tested from the Boyal quarry would also have disappeared. In general terms, the plagioclase is more alterable than alkaline feldspars. Black mica, or biotite, is more alterable than white mica, or muscovite. Granites with calcium plagioclases (anorthites) are more alterable than those containing sodium plagioclases (albites).

Table 3 Spacing and reflective powers

Phase	Spacing (Å)	Reflective power
Phyllosilicate	4.47	0.10
Phyllosilicate	9.91	1.00
Quartz	4.26	0.50
Feldspars	3.20	1.03
Chlorites	7.05	2.75
Illite	10.00	1.00

Mineralogical description of the samples

La Lagunilla quarry

The mineralogy of the altered samples from each test are discussed next. These are compared with the unaltered sample (Table 4). The test pieces from this quarry with the thermal finish were not submitted to X ray diffraction, while the Boyal quarry thermal finish test pieces were submitted to this type of analysis. The pre-alteration process is the same for both test-pieces, and the changes produced are the same as well. Only one of the two test-pieces was submitted and will serve in the description.

HCl immersion

The principal minerals appearing in the difractograms are micas (biotite), quartz, Na-Feldspar, K-Feldspar, and chlorite. In the HCl immersion, the decrease produced in the biotite is clear. Chlorite is present in spacing d7.04, at 224 counts/s. The Bertierines in d3.54, also experienced a very minor decrease in the HCl, 150 counts/s.

H_2SO_4 immersion

The principal minerals appearing in the difractograms are Micas (Biotite), Quartz, Na-Feldspar, K-Feldspar, and Chlorite. The decrease produced in the biotite in the H_2SO_4 immersion is evident. In spacing d3.18, the Na-Feldspar peak intensity changed from 536–1790 counts/s. Chlorite is also present in spacing d7.04, but at 73 counts/s it is much less than the HCl. The Bierterines in d3.54, at 15 counts/s, was also much less than the

Table 4 Mineral dynamics of different test-piece immersions from the La Lagunilla meadow quarry

Mineral phases (%)	Natural (1L)	Immersion HCl (3L)	Immersion H_2SO_4 (1L)	Immersion FH (1l)	HNO_3 (1L)	RILEM (2L)
Mica	69	22	17	–	11	27
Quartz	18	25	32	78	33	23
Na-feldspar	8	18	12	12	36	28
K-feldspar	3	34	38	9	19	21

HCl. A decrease in the layer silicates (micas) was observed while the quartz and feldspars increased.

FH immersion

The principal minerals appearing in the difractograms are Micas (Biotite), Quartz, and Na-Feldspar. In the FH immersion a decrease produced in the biotite was seen. The quartz increase was due the disappearance of the remaining minerals, and this made the quartz stand out more. The silicate decomposition in the FH produced a peak intensity increase in the spacings where quartz was present, these were d3.34 and d4.24. In this case, neither chlorite nor bierterines were found.

HNO_3 immersion

The primary minerals appearing in the difractograms are micas (biotite), quartz, Na-Feldspar, K-Feldspar, and chlorite. In the HNO_3 immersion, a decrease produced in the biotite can be seen. The attack primarily affected the micas, drastically descending their intensities. The Bierterines in spacing d3.54, 120 counts/s, was somewhat inferior to the intensity obtained in the HCl.

Soluble salt immersion, RILEM (1978) test

The primary minerals appearing in the difractograms are Micas (biotite), Quartz, Na-Feldspar, K-Feldspar, and Chlorite. A decrease produced in the biotite in the soluble salt immersion is clearly seen. The attack primarily affected the micas, drastically decreasing their intensity, while the quartz and feldspars greatly increased. The Chlorite found in spacing d7.04, at 60 counts/s, was much lower than normal. However, the Bierterines in d3.54, at 150 counts/s, was close to normal.

Boyal quarry

Thermal finish

From the difractograms, the unaltered granite difractogram (Table 5) is compared with that having a thermal finish (Table 6). This is done because the temperatures in the thermal finish process reach 2,800°C, and these can provoke changes in the minerals comprising granite. It is because of this granite is studied, due to the existing pre-alteration in such a finish before being subjected to any other alteration process. The principal minerals appearing in the difractograms are Micas (biotite), Quartz, Na-Feldspar, K-Feldspar, and Chlorite. In the thermal finish, the decrease in the biotite is clear. It can be deduced that quartz is the mineral least altered in the thermal finish, because this is the most predominant mineral remaining after this finish is applied. Feldspars are next, and in last position fall the biotites. Chlorite appeared in spacing d3.54, and in the thermal finish this decreased as well.

HCl immersion

The primary minerals appearing in the difractograms are micas (biotite), quartz, Na-Feldspar, K-Feldspar, and chlorite. The HCl immersion produced a clear decrease in the biotite. The decrease in the thermal finish was not as drastic. It can be deduced that the mineral least altered by the HCl is the quartz, followed by the feldspars, and lastly the biotites. The chlorites found in spacing d3.54 also decreased in the HCl, but in a more moderate way than they did in the thermal finish. With alteration other minerals appear, like fluorite and silvite in d3.14, and chlorapatite in d2.77.

H_2SO_4 immersion

The primary minerals appearing in the difractograms are micas (biotite), quartz, Na-Feldspar, K-Feldspar, and chlorite. In the H_2SO_4 Immersion, a decrease produced in the biotite was visible. The quartz slowed the intensity decrease, also remaining above the value obtained from the thermal finish nearest the HCl. The mineral least altered in the H_2SO_4 is quartz, followed by the feldspars and lastly biotites. The biotite values here were similar to those obtained in the HCl immersion. Chlorite was also found in spacing d3.54. Here, these values diminished in the H_2SO_4, but more moderately than they did in the thermal finish. There were also other minerals present, like mirabilite in spacings d3.28, d3.20, d3.14, d2.80, and d2.51.

Table 5 Mineral dynamics of different test-piece immersions from the Boyal meadow quarry

Mineral Phases (%)	Natural (1B)	Immersion HCl (3B)	Immersion H_2SO_4 (1B)	Immersion FH (1B)	HNO_3 (1B)	RILEM (2B)
Mica	56	34	26	13	19	28
Quartz	3	23	22	40	30	26
Na-feldspar	28	34	31	38	35	36
K-feldspar	12	8	20	8	15	9

Table 6 Mineral dynamics of treated test-pieces following HNO_3 immersions from the Boyal meadow quarry

Mineral Phases (%)	Bush hammered (2B)	Puntero (3B)	Arenization (4B)	Polished (5B)	Thermal finish (6B)	Apomazado (7B)	Sawed (8B)
Mica	13	21	19	26	37	15	23
Quartz	20	35	23	18	28	49	31
Na-feldspar	48	20	42	47	24	28	30
K-feldspar	18	23	15	8	10	7	15

FH immersion

The primary minerals appearing in the difractograms are micas (biotite), quartz, Na-feldspar, K-feldspar, and chlorite. The FH immersion produced a decrease clearly visible in the biotite. There was an increase in quartz due the attack carried out by the FH upon the silicates in all the granite components. In this case, there were no Feldspars noticeable.

HNO_3 immersion

In this test, as mentioned in the introduction, all finishes from this quarry are studied by comparing them with those natural and unaltered. The HNO_3 attack produces an important reduction in micas, chlorites, bierterines and K-Feldspars. In contrast, this acid produces an increase in quartz (d4.24) and in general, augments FNa (plagioclases), and as such the granite alteration index is noticeable. The bush hammered (2B), arenization (4B) and polished (5B) samples are those containing greater plagioclase quantities, and this indicates a greater decomposition of the silicate minerals, forming other SiO_{4-} (FNa), and if the attack contains these, they would then decompose into silica, and the rock would crumble. The bush hammered (2B), arenization (4B), and polished (5B) rock finishes were those most decomposed by the HNO_3.

Soluble salt immersion, RILEM test

The primary minerals appearing in the difractograms are micas (biotite) quartz, Na-feldspar, K-feldspar, and chlorite. In the soluble salt immersion ($Na_2SO_4 \cdot 10\ H_2O$) the decrease produced in the biotite is noticeable. This drop is similar to the one produced in H_2SO_4. Chlorite and bierterines are also found in spacing d3.54. Because of the $Na_2SO_4 \cdot 10\ H_2O$ deposition on the surface of the test-pieces, minerals such as mirabilite appeared ($Na_2SO_4 \cdot 10\ H_2O$). The alteration process is important because of a high FNa concentration. This is unlike other ageing tests, because here the micas (in small measurements) and quartz remain, and salts such as mirabilite appear in small quantities. This is what is used in the actual RILEM test.

Weathered HNO_3 residues

All of the test-pieces from both La Lagunilla and those from Boyal were submerged in the same bath. The results of the weathered granite residues difractogram caused by the HNO_3 were observed as being similar to those obtained from the samples taken having the attacked surfaces (Table 7).

In the first place the decrease in the micas (d9.91), moving from 1,650 counts/s in the La Lagunilla quarry, and 6,500 counts/s in the Boyal quarry, to 20 counts/s in the residues, almost disappeared. This value is seen as greatly inferior to that obtained in the Boyal quarry and nearly the one obtained in the La Lagunilla quarry (see the HNO_3 immersion difractograms from both quarries). This indicates that the decrease was produced in the Boyal quarry and because of this the great majority of residues come from this quarry.

The quartz in (d3.24) decreased, the residues remaining with a peak intensity of 1,340 counts/s above the value obtained in the HNO_3 immersion from the La Lagunilla quarry, and inferior to the value from the Boyal quarry (~2,500 counts/s). This indicates the peak intensity decrease is proportional to the decrease the Boyal quarry experienced, being even more probable that the residues are from this quarry.

In spacing d4.24 the peak intensity takes a value of 280 counts/s inferior to that taken at the La Lagunilla quarry in both the unaltered test-pieces (~400 counts/s), as well as in the HNO_3 altered variety (~320 counts/s), and superior to the peak intensity value of the natural unaltered Boyal quarry test-piece (210 counts/s). The value obtained in the altered test-piece from this quarry was 490 counts/s.

The increase achieved by this test-piece and the increase achieved by the residues with respect to the peak

Table 7 Semi-quantitative data analysis corresponding to weathered residues immersed in HNO_3 (L.B.)

Mineral phase	(%)
Mica	–
Quartz	41
Na-feldspar	49
K-feldspar	9

value of the natural unaltered Boyal quarry test-piece are related, but neither one is related to the decrease achieved by the altered Lagunilla quarry test-piece with respect to the natural and unaltered test-piece. This is another clear indication that the majority of the residues are from the Boyal quarry.

The Na-Feldspars in spacing d3.20 decrease their peak intensity (520 counts/s), just the same as does the altered Boyal quarry test-piece (1,635 counts/s). Notwithstanding, the value of the altered La Lagunilla quarry test-piece spacing increases, again a clear example that the majority of the residues are from the Boyal quarry. The chlorites decreased just like the bierterines, occurring with both the altered test-pieces from the Lagunilla quarry as well as with the altered test-pieces of the Boyal quarry.

Conclusions

By X-ray diffraction, the Boyal quarry granites are shown to have higher mica content (biotites) than do the La Lagunilla quarry granites. The thermal finish process reaches temperatures of 2,800°C before provoking changes in the minerals in the granite, primarily the elimination of the mica-biotites and the vitrification of the surface, which leaves greater resistance to alteration. However, natural granite stones with high content in mica, without thermal finish treatment, are more susceptible to weathering. With respect to the quarry with more mica content, some complementary experimentation is being carried out in order to determine the influence of mica grains on the weathering, though in principle only it would only affect the humidity expansion, which after being determined, must be taken into account by preventing the joint separation into the applications of these materials. Test-pieces tested with HCl and H_2SO_4 decolor the granite without attacking the minerals, this is to say, without weathering them. Both acids are good devices to age the stone without modifying its integrity. But HCl is the more yellowing of the two and so can be used to age new stone in restoration.

HF and HNO_3 acids excessively attack granite and vary its mineralogical homogeneity. Because of this, they are not useful in restoration or ageing. HF is the most aggressive. This completely changes the gray color in granite to white. In addition to highly weathering the stone, it also dissolves the quartz, and quartz is the hardest mineral comprising granite. HNO_3 alters granite less than does HF, but its effects are not negligible. While the chromatic variation is not very high, the weathering is substantial.

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