Stone Construction Products – Physical and Mechanical Characterisation

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Abstract: The scope of this work was to study the physical and mechanical properties of natural stone in order to develop an optimised characterisation procedure useful for companies to implement quality controlling systems employed in the transformation processes of construction products. The Portuguese natural stone sector is mainly comprised of family-based small-medium companies showing basically a strong empiric knowledge. This situation is presently originating great deficits and problems related not only with the legal obligations of the sector but also to the lack of knowledge when a new product / application is being developed. With the ratification of the stone construction products directive (Directive no. 89/106/CE) all companies that sell their stone construction products in the EU are obliged to have the CE marking. The CE marking is mainly set up in two phases: (i) initial-type tests and (ii) factory production control. The certification conformity system for the natural stone products is the lowest in demand (4), and establishes an exclusive responsibility of the producer in terms of performing tests and characterising their stones and in terms of establishing factory production control.

Pursuant to the above mentioned legal obligation, Assimagra – Portuguese Marble and Granites Association and FrontWave – Engineering and Consultancy, S.A., developed, in January 2005, a CE marking implementation service in compliance with the Portuguese natural stone sector and legislation.

This implementation was initially preceded by a large study on characterisation procedures for determining the physical and mechanical properties of natural stone. This work, summarised in this abstract, has revealed the possibility to investigate alternative procedures (more adequate for industrial application), new correlations between properties and the use of a statistical tool (Weibull statistical analysis) allowing to evaluate no only the properties of the stones but also how these properties vary in a certain company by taking into account the effects caused by the natural variation of each stone and the impact caused by the transformation process.

The service developed includes the CE marking implementation at the companies and the relevant maintenance. The implementation stage comprises: (i) the necessary physical-mechanical tests for the characterization of the product; (ii) the conformity declarations and; (iii) a file were all the technical information is kept and archived. During the maintenance stage the service provided makes available a factory production control support with production records evaluation and periodic samplings.

The two presented case studies refer to a small company based in the centre of Lisbon and to a medium company based in Viseu. The implementation process is very similar in both cases: i) stone and products acknowledgment; ii) first meeting at the company for sampling and training session,

initial-type tests implementation, conformity declaration and the emission of technical documents; and iii) second meeting to deliver the technical file and training session.
The experimental results obtained in the physical-mechanical tests for the same stone types allowed the following conclusion: stone is a natural product and inside a single stone denomination (arbitrarily given by each producer), the properties may suffer alterations depending on the quarry levels of extraction and/or on the exact location of each quarry. A stone named “Moleanos” or “Semi-Rijo” may not correspond to the “real” lithological type. Hence, this work has contributed to face the serious need of correcting stone denomination by implementing a characterization procedure useful to improve the selection of natural stone construction products according to each application.

Introduction

The European internal market offers the stone producers the possibility of enlarging their market but it is also a cause of new rules and obligations. This is mainly due to the fact that technical barriers to the trade can be removed, provided that a harmonization of different technical regulations and specifications in use in different European countries is reached.
It is therefore essential for the stone producers to know the general rules set forth by the European Community as a basis for this harmonization.
In the building sector the reference is the Directive 89/106/EEC on "Construction products" (“CPD”). According to the CPD, construction products may be placed on the marked only if they are suitable for the intended use. Hence, the products which are to be incorporated, assembled, applied or installed are in compliance with the legal requirements if properly designed and built and if they satisfy the following essential requirements:
- Mechanical resistance and stability;
- Safety in case of fire;
- Hygiene, health and the environments;
- Safety in use;
- Protection against noise;
- Energy economy and heat retention.

In Portugal, the legal obligation is achieved by the DL 113/93 from April 10. However, despite the existence of regulation for CE marking of natural stone construction products since 1993, only in 2005 the needs of fulfilling this regulation has been achieved. Such delay is due, among others factors, to the fact that there was a delay in the harmonized test standards publication - essential for all the technical part of the process. [1, 2, 3]
Natural stone products are very appreciated in the worldwide, recognized for their beauty and unquestionable opinion that a correct stone application brings an added value in any work, both at the aesthetic level and durability. The applications using stone products are diverse, being possible to transform a great block of stone into a work of art of recognized aesthetic value.

This happens because the aesthetic criteria frequently prevails on the technical criteria, many times because these are unknown for who applies the stone. Situations of inadequately placed stone lead to problems of defective visual aspect or even accidents, which can be serious and put in risk a human life. Physical-Mechanical characteristics, shape, dimension and mainly the application of a stone must be taken to account in any work.

In this work, the main methods studied for the characterization of natural stone construction products are explained with reference to the harmonized product standards. All the complementary methods of characterization that had assisted in the estimative correlation of some of the properties (for example: determination of the abrasion resistance), are equally referred. The most important results also are briefly presented and discussed for different classes of stones.

Physical – Mechanical Characterization

These tests were studied, implemented and carried through the support of the IST – Instituto Superior Técnico. Among others less used, we may refer to:

Determination of Flexural Strength – Concentrated Load and Constant Moment

This is one of the most important tests on natural stone characterization. It may be carried out by two different methods:

- Under Concentrated Load (EN 12372)
- Under Constant Moment (EN 13161)

Both methods advised the use of a minimum number of specimens, 10 specimens shall be selected from a homogeneous batch; the surface finish of the faces of the specimens shall be sawn, honed or polished. It may also be required to do some tests on specimens with other surface finishes, e.g. flamed, sandblasted, when required for application. [4, 8]

The result shall be expressed in Megapascal (MPa) to the nearest 0,1 MPa and it's calculated using the following equation:

$$\frac{3F\left(L_0 - L_i\right)}{2bh^2} \qquad \text{Equation (1)}$$

$$\frac{3F\left(L_0 - L_i\right)}{bh^2} \qquad \text{Equation (2)}$$

Where, *F* is the breaking load (N), L_0 e L_i represent the distance between the supporting rollers (mm), *b* represent the width of the specimen (mm) and *h* the thickness. [4, 8].

The aim of the constant moment flexural tests, using a high number of specimens (between 25 and 30), is related with the application of a statistical tool – Weibull Statistic's.

On similar natural stones, a great number of distinct defects are observed, such as fissures, pores, inclusions, secondary phases, or even machining defects or defects cause by environmental exposure that may contribute to fracture. In order to study flexural resistance variation (on similar stone populations) the results were treated by the Weibull statistics that describes the strength distribution of brittle materials.

Weibull analysis takes into account the weakest link model approach, for witch the strength of a body involves the "products" of the survival probabilities for the individual volume elements. [5]

Using the results of bending strength of a large number of specimens, 25/30, and applying the graphic method (fitting the bending strength values to a three-parameter Weibull distribution), we estimated the Weibull parameters σ_0= scale parameter and m= shape parameter.

The shape parameter, *m*, represents the way like the fracture occurs, that is, the fracture dispersion in a certain stone population.

The experimental results obtained from specimens with similar Weibull parameters may be considered, like specimens with the same mechanical properties, at least in terms of flexural resistance. This analysis will be discussed furtherer.

Water Absorption at Atmospheric Pressure

Water absorption at atmospheric pressure and water absorption by capillarity should be determined for a proper materials selection (despite the fact that this last method should only be applied when the stone specimens have an open porosity larger than 1 or 2 %) [4]

Water rolls an important part on building deterioration. As consequence, it's extremely important the property determination that involves water transport trough out the stone pores (water absorption at atmospheric pressure and water absorption by capillarity). The water effects can be bad not only on stone construction products, but also on concrete where the water can be conducted if a high porous stone has been applied. This situation is particularly dangerous when the stone is used in cladding. If, for example, the stone (tiles with reduced thickness) is fixed on the concrete only with cement and this started to corrode by the water that reaches the concrete trough the stone pores, probably the tile will collapse and fall due to a lack of fixing.

The determination of Water absorption at atmospheric pressure is made by the procedure described on the standard EN 13755:2005 and the water percentage can be estimated knowing the dry mass and the water satured mass of the specimen, from equation 3 [6]:

$$\frac{(M_{Sat} - M_S)}{M_S} * 100\% \qquad \text{Equation (3)}$$

M_S – Mass of the dry specimen

M_{Sat} – Mass of the satured specimen

Apparent Density and Open Porosity

The stone apparent density is the quotient between the mass of the dry specimen and its apparent volume (limited volume by the external surface of the specimen, including the vacant). The open porosity (or apparent) is the quotient, in percentage, between the open pore volume and the apparent volume of the specimen.

Considering that the apparent density is the parameter that relates the open porosity of a certain stone with its water absorption, these three characteristics may be determined at the same time. However, the procedure described on the standard EN 1936:1999 establishes that the density and apparent porosity should be determined in vacuum. In this work this was not possible, because we didn't have the necessary equipment for the vacuum.

Apparent density (Kg/m^3) may be determined by the equation 4 [4, 7]:

$$\frac{M_S}{(M_{Sat} - M_I)} * d_{H2O} \quad (\text{kg/m}^3) \qquad \text{Equation (4)}$$

d_{H20} = water density at 20ºC (998 Kg/m^3);

M_S = mass of the dry specimen (Kg);

M_{Sat} = mass of the saturated specimen (Kg);

M_I = mass of the immerged specimen (Kg).

The open porosity (%) may be estimated by the equation 5 [4,7]:

$$\frac{M_{Sat} - M_S}{M_{Sat} - M_I} * 100(\%) \qquad \text{Equation (5)}$$

Experimental Results

Limestones

		Physical - Mechanical Properties											
Lithological type	Name of the Stone	Stress (MPa)	SD (MPa)	m (Weibull)	σ0 (MPa)	Stress (MPa) - concentrated load	SD (MPa)	Apparent Density (kg/m3)	SD (MPa)	Open Porosity (%)	SD (MPa)	Water Abs. Atm Pressure (%)	SD (MPa)
Limestone	Moleanos C - 1	8,2	0,8	11,8	8,5	9,9	0,5	2339	30	10,9	0,2	4,7	0,1
	Moleanos C - 15	13,9	0,6	**27,5**	**14,1**	12,6	2,0	2357	7	6,9	0,2	2,9	0,1
	Moleanos C - 20	13,0	2,1	**7,3**	**13,9**	9,7	4,6	2555	7	2,6	0,1	1,0	0,05
	Moleanos C - 29	9,4	1,0	11,3	9,9	10,0	1,3	2395	23	5,7	0,4	2,4	0,2
	Lioz C - 2	14,8	1,6	**10,9**	**15,5**	19,4	1,4	2693	8	0,34	0,05	0,13	0,02
	Lioz C - 22	11,8	1,3	10,7	12,3	12,7	1,8	2686	3	0,32	0,05	0,12	0,02
	Lioz C - 24	13,2	2,2	**6,55**	**14,2**	17,1	1,7	2698	1	0,15	0,02	0,06	0,01
	Lioz C - 28	12,0	1,4	10,3	12,6	13,6	1,3	2680	2	0,4	0,1	0,13	0,06
	Azul Valverde C -42	11,4	3,7	**5,6**	**13,4**	19,3	1,6	2690	1	0,43	0,04	0,16	0,01
	Azul Valverde C - 39	15,9	2,1	8,8	16,8	13,0	1,5	2632	4	1,0	0,1	0,36	0,02
	Azul Valverde C - 32	12,5	2,7	**9,3**	**13,7**	12,7	1,3	2612	8	1,3	0,1	0,5	0,05
	Azul Valverde C - 13	14,9	2,0	8,7	15,7	13,4	1,0	2609	1	1,51	0,04	0,58	0,02

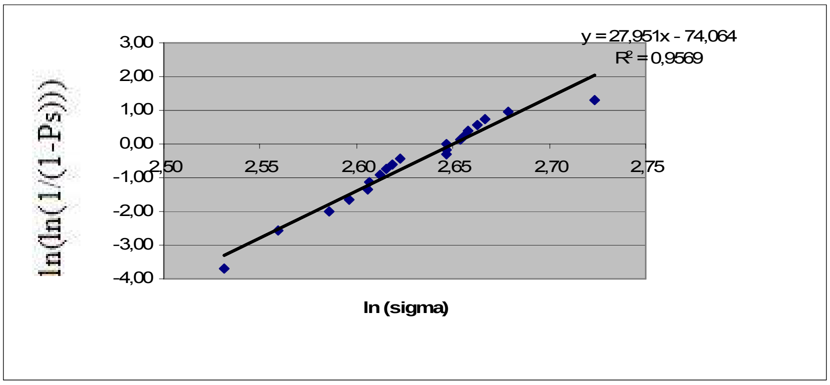
Table 1 – Limestone´s Characterization results

Comparisons between the three limestone populations, with the same finishing treatment and geometrical control, allow us to verify that: on the four samples of Moleanos, we observe very distinct values for the Weibull modulus 27,5 and 7,3. The fitting of the bending strength values (constant moment) to a three-parameter Weibull distribution, for the samples C-15 and C-20, trough the graphical method can me observed on figures bellow.

This difference is reflected on the flexural resistance under concentrated load mean value – 12,6 and 9,7 (MPa).

The stone Moleanos shows very significant variations, when compared with the other two Limestone's in terms of porosity – 2,6 to 10,9 (%), water absorption – 1,0 to 4,7 (%) and flexural resistance under constant moment – 8,2 to 13,9 (MPa).

The four Lioz samples shows, Weibull modulus variations between 6,5 and 10,9. Lioz is a Limestone that presents low variations than Moleanos in terms of open porosity – 0,15 to 0,4 (%), water absorption – 0,06 to 0,13 (%) and flexural resistance under constant moment – 11,8 to 14,8 (MPa).



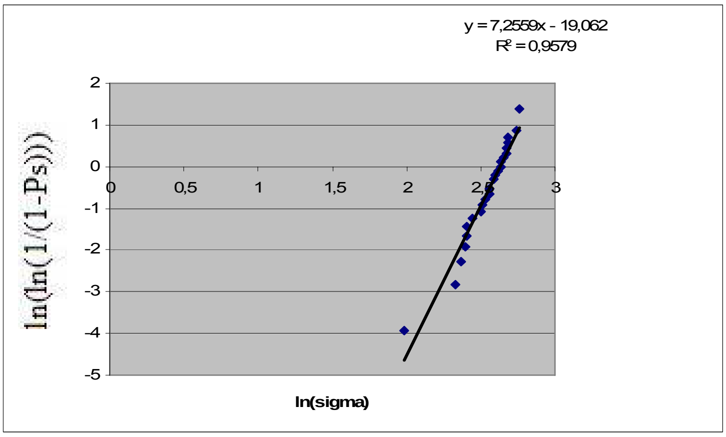


Fig. 1 and 2 - Fitting of the bending strength values (constant moment) to a three-parameter Weibull distribution, for the samples C-15 and C-20, trough the graphical method.

The four samples of Azul Valverde show Weibull modulus between 5,6 and 9,3. The limestone Azul Valverde reveals lower variations than Moleanos in terms of (i) open porosity – 0,43 to 1,51 (%) and (ii) flexural resistance under concentrated load – 11,4 to 15,9 (MPa).

When we compare the three limestone populations, with the same finishing treatment and geometrical control, there's a very interesting curiosity: to the lower Weibull modulus (m) always matched the lower water absorption value (%).

Marbles

		Physical - Mechanical Properties											
Lithological type	Name of the Stone	Stress (MPa)	SD (MPa)	m (Weibull)	σ0 (MPa)	Stress (MPa) - concentrated load	SD (MPa)	Apparent Density (kg/m3)	SD (MPa)	Open Porosity (%)	SD (MPa)	Water Abs. Atm Pressure (%)	SD (MPa)
Marble	Estremoz M-2	13,8	0,7	23,7	14,1	16,8	0,8	2687	2	0,32	0,08	0,12	0,03
	Estremoz M-4	12,4	1,6	8,8	13,1	15,8	1,8	2711	3	0,15	0,01	0,060	0,005
	Estremoz M- 11	19,3	0,9	25,3	19,7	21,5	0,7	2703	1	0,2	0,02	0,072	0,004
	Estremoz M-13	14,8	2,1	8,3	15,7	16,2	1,2	2705	1	0,18	0,02	0,07	0,01
	Ruivina M-1	10,5	0,8	14,9	11,0	13,5	1,6	2673	12	0,19	0,01	0,07	0,01
	Ruivina M- 5	9,6	1,2	9,4	10,1	10,4	1,5	2699	3	0,34	0,05	0,13	0,02
	Ruivina M- 7	14,8	1,6	10,4	15,6	16,5	1,0	2702	2	0,27	0,04	0,10	0,01
	Ruivina M-12	22,7	2,2	12,1	23,7	20,6	1,7	2709	2	0,16	0,01	0,06	0,005

Table 2 – Marble´s Characterization results

Comparisons between the two different marble populations, with the same finishing treatment and geometrical control, allow us to verify that all the four Estremoz samples are very homogeny among each other in terms of open porosity and water absorption.

The Weibull modulus however, revealed notorious differences – 25,3 to 8,3. Especially between the samples M – 13 and M – 11/M – 2. This difference is reflected on the flexural strength under concentrated load value - 21,5 and 16,2 (MPa).

The four samples of Ruivina show low porosity (%) and absorption (%) values very similar with the ones observed on Estremoz. The Weibull modulus varies between – 9,4 and 10,9 on M – 5 and M – 1, respectively.

Granites

		Physical - Mechanical Properties											
Lithological type	Name of the Stone	Stress (MPa)	SD (MPa)	m (Weibull)	σ0 (MPa)	Stress (MPa) - concentrated load	SD (MPa)	Apparent Density (kg/m3)	SD (MPa)	Open Porosity (%)	SD (MPa)	Water Abs. Atm Pressure (%)	SD (MPa)
Granite	Pedras Salgadas G-1	13,9	0,7	25,2	14,2	17,5	0,3	2606	3	0,51	0,03	0,19	0,01
	Pedras Salgadas G-7	13,6	0,7	24,0	13,9	16,1	0,9	2603	3	0,61	0,08	0,24	0,02
	Pedras Salgadas G-11	13,0	0,6	26,8	13,3	16,3	0,6	2620	2	0,46	0,05	0,18	0,02
	Amarelo Vila Real G-3	7,4	0,7	11,8	7,7	12	1,4	2596	15	1,2	0,1	0,45	0,04
	Amarelo Vila Real G-13	4,9	1,5	3,1	5,5	6,7	0,3	2570	7	1,74	0,02	0,68	0,01
	Amarelo Vila Real G-18	4,5	0,7	6,9	4,9	5,9	0,5	2518	25	2,5	1,1	0,70	0,46

Table 3 – Granite´s Characterization results

Comparisons between the two different granite populations, allow us to verify that:

In the three samples of Pedras Salgadas we observe lower porosity values – 0,46 to 0,61 (%) than in the three samples of Amarelo Vila Real – 1,2 to 2,5 (%).

The Pedras Salgadas granite is more homogeneous, in concern with flexural resistance under constant moment, when compared with Amarelo Vila Real.

Pedras Salgadas granite shows a Weibull modulus that varies between 24,0 and 26,8 for the samples G – 7 and G – 11, respectively. Amarelo Vila Real granite shows a Weibull modulus that varies between 3,1 and 11,8, for the samples g – 13 and G-3, respectively.

Discussion

Limestones

The difference that occurs between the flexural resistance under concentrated load for the samples C-15 and C-20 (Moleanos), 12,6 and 9,7 (MPa) respectively, does not allow us to be attentive on the flexural mechanical performance of the C - 20 limestone . C – 20 limestone presents a Weibull modulus, m, of 7,3, a very low value when compared with the one observed by the limestone C-15 (m=27,5).

The same happens with the difference between the flexural resistance under concentrated load for the samples C-2 and C – 24 (Lioz), 19,4 and 14,2 (MPa) respectively. Again, these values do not allow us to be careful on the flexural mechanical performance of these samples. Limestone C-24 presents a Weibull modulus of 6,5, on the other hand limestone C-2 shows m=10,9.

Moreover, we can see the same occurs with the limestone Azul Valverde. In the samples C-42 and C-32 the difference between the flexural resistances under concentrated load values is too small, hence not allow an identification of the flexural performance of the specimens. The results shows Weibull modulus of 5,6 and 9,3 for the samples C-42 and C-32 respectively.

The problem here is connected with the test type that we used. Flexural resistance determination under concentrated load, tests only one plan of the specimen, leading to scale effect problems. We would have to use a larger number of specimens in order to observe the different flexural behaviour of all the three limestones.

Weibull statistic's turns up here as a powerful tool to compare stone populations and to predict performances. With these tools we can extrapolate stress values of small specimens, to bigger specimens, see equation (6):

$$\left(\frac{\sigma_1}{\sigma_2}\right) = \left(\frac{V_2}{V_1}\right)^{1/m}$$

Knowing the flexural stress of specimen 1 - σ_1, the volume of specimen 1 - V_1 and the Weibull modulus – m_1 it is possible to extrapolate the value of the flexural resistance (under constant moment) for a sample with a bigger volume , V_2.

Another important observation deals with the variations (between the same lithological types) on the porosity and water absorption at atmospheric pressure. These differences, easy to verify on Moleanos, are related with the quarry zones. The different quarry levels will always correspond different mechanical properties. The quarry level variation is not taken into account by some industrials who sell their stone always with the same denomination.

Marbles

The difference which occurs between the flexural resistance under concentrated load for the samples M – 13 and M – 11 (Estremoz), 16,2 and 21,5 respectively, does not allow us to be attentive on the flexural mechanical performance of the M- 13 Marble. M – 13 Marble presents a very low Weibull modulus of 8,3, this value is very small when compared with the one's presented by M – 11 marble (m=25,3).

The same happens with the difference between the flexural resistance of M-5 and M-1 (Ruivina), 10,4 and 13,5 (MPa) respectively, one more time these values does not show the mechanical performance of the specimens. M – 5 marble presents a Weibull modulus of m=9,4 and M – 1 marble the m value is 14,9.

Hence, we face the same problem that was registered in limestones. If the specimen number, to make flexural resistance tests under concentrated load, is not enough, several errors can be made on the selection of a stone for a specific application.

All Estremoz four samples are very homogeneous in terms of open porosity (%) and water absorption (%). This lithological type of stone doesn't show, normally, great property variations. However, when a quarry level alteration occurs there are some important changes on the flexural resistance under constant moment mostly due to the presence, or not, of carbonaceous materials – "veins" (very common in some variations of Estremoz) [9].

Normally, producer's well knower's of all the Estremoz variations and the more cleanly the stone is more expensive it becomes.

Ruivina is very similar to Estremoz, in terms of physical-mechanical properties. The microscopy a shoe that the main difference between these two marble types is the presence of some carbonaceous material's that gives Ruivina a dark grey colour. [9]

Granites

The difference's that occur on the flexural resistance values under concentrated load for the specimens G – 7 and G – 11 (Pedras Salgadas), 16,1 and 16,3 (MPa) respectively, are small and even in this case there's a Weibull modulus difference. For G – 11 granite we have m = 26,8 and for G – 7 m = 24,0.

In Amarelo Vila Real granite the difference's between the flexural resistance values under concentrated load for the samples G – 3 and G – 13, 12,0 and 6,7 (MPa), doesn't allow us to predict the enormous discrepancy on the Weibull modulus and thus the distinct mechanical behaviour of the to specimens. For G – 3 m = 11,8 and for G – 13 m = 3,1.

Pedras Salgadas granite is an extremely uniform stone especially when compared with Amarelo Vila Real granite. So, it is highly recommended that we use a larger number of specimens to characterize Amarelo Vila Real granite. This stone type is used with frequency in churches and houses in the north of Portugal because, when placed, it gives an antique look to all the constructions.

Concluding Remarks

The physical and mechanical characterization of natural stone construction products (included on CE marking obligation) intends to be a platform in witch companies may grow and state themselves as large quality stone producers.

The implementation of the technical service by studying the characterisation methodologies in detail and technical analysis of results for different classes of stones, has allowed the companies to benefit of a inexistent technical support very competitive in terms of cost.

The service competitiveness is justified by the use of some correlations and statistical methods studied during the experimental work, that help to reduce the test time without jeopardise the technical validity of the results.

In what concerns results, the experimental values demonstrated the impossibility to control or modify that the characteristics of such natural construction product. Stone properties may change significantly and therefore we have to use statistical analysis to improve our knowledge of tools.

Notorious changes occur especially on high porous stones like limestones and inside this group changes are, for example, more evident in Moleanos than in Lioz. This difference is justified because of the fossil presence in Lioz that gives to this stone a less porous structure.

Generally, the characterization methods revealed suitability for the stone construction products CE marking system (level 4). In technical terms, some characteristics obliged by the CE marking were found to be of complex application. For example, flexural resistance under concentrated load shows insufficient to predict the mechanical behaviour of a stone, which is not desirable when designing or selecting the stone for a certain application. In fact, the work has showed that Weibull statistics is a good analysis tool, simple to be used, allowing to identify identical stone populations and even contributing for dimensioning stone products with a given volume (equation 6).

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