Concrete Mixture with use of Iron Powder Waste for Coarse Aggregate Doping

Renan Dias, Sherington Bigotto, Daniel Ukuessunga Benjamim, Denise Luisa Chotolli, Matheus Viana de Souza

***Abstract*—The high consumption of concrete in construction, coupled with the environmental impacts resulting from the extraction of raw material for its manufacture, fosters innovation and ideas for its improvement. In order to optimize its resistance to stresses, as well as durability and dimensional stability, the aggregate doping technique was developed with the purpose of improving low quality aggregates, as well as optimizing those with good mechanical properties. Using the iron residue – iron powder – to perform the process, the doping proposes, besides an improvement in the concrete properties, a microstructural analysis in the interfacial transition zone (ITZ) of the paste and aggregate interface, where the first cracks of the material occur, due to mechanical stress. For this purpose, the focus was on comparing specimens tests at 28 days with and without doped aggregate, comparing results of diametrical compression, and modulus of elasticity. Regarding the compression tests, there was a significant increase in the diametral and axial compression; it was observed compatible values between the modulus of elasticity calculated based on the Brazilian standard and those obtained experimentally.**

***Keywords*—Aggregate Doping, Concrete, Mechanical Testes and Iron Powder Waste**

I. INTRODUCTION

Worldwide, the concrete is one of the most popular materials due to its performance, facility of application, fire resistance, mechanical reliability and adjustable thermal energy properties [1].

This work was carried out during the course “Experimental Analysis of Structural Systems”, held during the second semester of 2019, taught by Prof. José Luiz Pinheiro Melges, pH.D., as part of the PPGEC of FEIS-UNESP.

Dias, R. Author is with the Programa de Pós Graduação em Engenharia Civil – PPGEC, Faculdade de Engenharia de Ilha Solteira, Universidade Estadual Paulista "Júlio de Mesquita Filho"– FEIS/UNESP (corresponding author to provide phone: +55 17 9 9628.7465; e-mail: rdias07@live.com).

Bigotto, S. Author is with the Programa de Pós Graduação em Engenharia Civil – PPGEC, Faculdade de Engenharia de Ilha Solteira, Universidade Estadual Paulista "Júlio de Mesquita Filho"– FEIS/UNESP (e-mail: sherington.bigotto@gmail.com).

Benhamim, D. U. Author is with the Programa de Pós Graduação em Engenharia Civil – PPGEC, Faculdade de Engenharia de Ilha Solteira, Universidade Estadual Paulista "Júlio de Mesquita Filho"– FEIS/UNESP (e-mail: ukuessunga01@gmail.com).

Chotolli, D. L. Author was with the Programa de Pós Graduação em Engenharia Civil – PPGEC, Faculdade de Engenharia de Ilha Solteira, Universidade Estadual Paulista "Júlio de Mesquita Filho"– FEIS/UNESP (e-mail: denise.l.chotolli@hotmail.com).

Souza, M. V. Author is with the Programa de Pós Graduação em Engenharia Civil – PPGEC, Faculdade de Engenharia de Ilha Solteira, Universidade Estadual Paulista "Júlio de Mesquita Filho"– FEIS/UNESP (e-mail: 308164@aluno.unilins.edu.br).

It is known that the cement industry has one of the highest CO_2 emission rates, being approximately 5 to 7% of the world total, generating environmental impacts worldwide [2].

It is the material most widely used in civil construction, based on the data illustrated in Fig. 1, that presents a preliminary forecast of the sale of cement and an estimate between the months of October, 2016 / September, 2017 to October, 2018 / September, 2019.

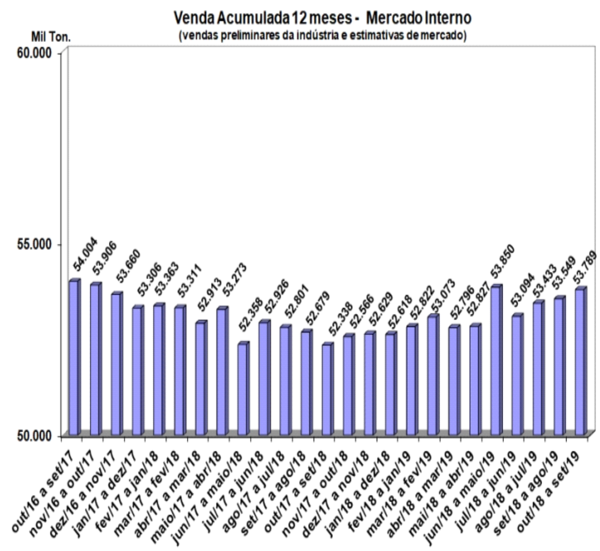

Fig. 1. Sale of cement, preliminary results [3]

To reduce the environmental impact caused by the production of concrete, some solutions have been adopted, such as the use of alternative fuels that improve the carbon capture of cement, optimizing the energy efficiency of the calcination process [4].

Usually, the concrete consists of a mixture of Portland cement, aggregates and water, with or without chemical or mineral additives [5].

According to [6], over the past few years, concrete has been studied in order to improve its mechanical properties and provide economy and speed to buildings. Architectural requirements demand structural elements that are increasingly lighter, slender and even colored.

As a result, new methods are needed to optimize the quality of the concrete, that is the case of aggregate doping.

The term doping is commonly used in the medical, electronic and currently, civil construction areas, mainly in paving, acting to improve the adhesiveness of bitumen in aggregates [7].

Due to its composite nature, concrete can be doped with additives capable of improving its physical properties and providing new characteristics to the material [1].

Many benefits for civil construction can come from the material doping technique, applying them in coarse aggregates that may be used in the production of concrete, whether they are of high performance or even conventional ones [7].

Also according to [7], the doping technique consists of establishing a connection bridge between the binders and the material, or modifying its texture, through the initial impregnation with materials that will react with the binders. The Fig. 2 shows an illustrative scheme of the aggregate doping process, that modifies its texture and, consequently, changes the ITZ.

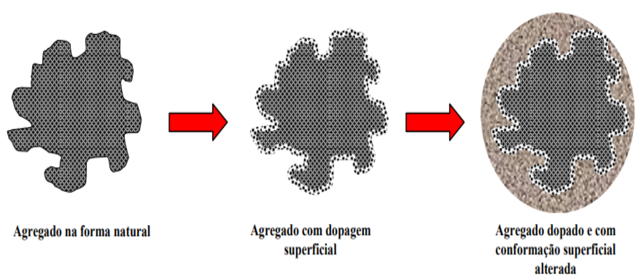

Fig. 2. Alteration of the surface conformation of the aggregate [8]

According to [9], the type and quality of aggregates and the cementitious matrix significantly influence the structural performance of concrete. This is because the interfacial transition zone (ITZ) of these two components is considered the weakest link in the structure.

The use of mineral additions acts on the microstructure of the paste and on the paste/ aggregate interface, refining the pores. In doping, the chemical effect is responsible for the formation of hydrated compounds and for the densification of the ITZ, providing concrete with greater resistance and durability [8].

The utilization of surface treatment in the aggregate brings the possibility of transforming low-quality aggregates into good-quality aggregates, based mainly on the theory of improving the ITZ at the interface between cement paste and coarse aggregate [7].

For [10], the doping technique influences the behavior of the bond between the matrix and the aggregate, the main types being the mechanical bond, the adhesion bond, the chemical bond and the physical bond. In the mechanical bond, according to the author, the crystals of the hydrated components of the cement involve the irregularities of the surface of the agglomerated materials, on a macroscopic scale due to surface roughness. When adhesion bond, part of the water containing part of the dissolved binder is absorbed by the aggregate, that, after the crystallization process, allows a better bond with the paste. The chemical bond is the adhesion between the reaction products of the hydration of the cement and the surface of the aggregate, and finally, the physical bond, by attraction without continuity of the structure between the surface of the aggregate and the cement paste.

According to [7], each type of connection between the aggregate and the cement paste can interfere with the quality of the cement matrix. Among the physical causes of adhesion, the most important is the mechanical connection.

Another factor that significantly influences the strength of the concrete is the aggregate texture: the rougher the aggregate surface, the greater the interlock between it and the cement matrix. So, the aggregate from crushed rock produces superior adhesion compared to that of the pebble [7].

For calcium oxides, aluminum and iron, cement has no restrictions. Known as main oxides, these compounds are able to promote an increase in the strength of the concrete [11].

Civil construction generates large quantities of waste every year, causing a challenge to sustainability and environmental protection. From this point of view, recycling the waste produced becomes an alternative to alleviate this challenge [12].

In the mining sector, large quantities of tailings are generated due to the extraction of iron ore. Thus, to minimize environmental impacts, studies are being carried out aiming at the use of these tailings [13]. As an example, we have the iron powder, which can be used in the doping of coarse aggregate in order to give a rougher texture to the aggregate, improving its connection with the cement matrix.

Therefore, the objective of this work is to compare some mechanical properties of concrete with conventional strength, using coarse aggregates with and without the effect of iron powder doping. Testes of axial compression, modulus of elasticity, traction by diametrical compression and absorption were performed.

II. MATERIALS

The following materials were used in the composition of the concrete mixture: cement, coarse aggregate, doped coarse aggregate, fine aggregate (sand), iron powder, Compound® adhesive and water.

The chosen cement to compose the concrete mix was brazilian CP II E-32, mixed within the parameters of NBR 16697 [14]. Table I presents the main characteristics of the cement supplied by the manufacturer.

For the study of mixture proportion, the Specif Gravity, absolute test was performed by the authors, resulting in a value of 3.0 g/cm³.

As fine aggregate, fine sand was used, coming from the sand port of the municipality of Castilho-SP, Brazil. Gravel #1 was used as a coarse aggregate, according to brazilian Standard NBR 7211 [15], from the Castilho-SP, Brazil, quarry. The iron powder waste used came from a company in the municipality of Jaboticabal-SP, Brazil. As coarse doped aggregate, the same Gravel #1 was used. In Table II, the characteristics of the materials used in the mixture proportion are presented.

Table I. Ordinary Portland Cement (OPC) properties used for concrete mix (Factory CIPLAN)

Característica	Unit	Valor
Blaine fineness (NM 16372/2015)	cm²/g	3,200
Specific Gravity, absolute at 20ºC	g/cm³	2.8 a 3.2
Setting time (NM 16607/2018)	Min	205-320
Compression Strength (f_c)	MPa	10.0 (1 day)
		20.0 (3 days)
		25.0 (7 days)
		32.0 (28 days)

Table II. Materials Characteristics

Característica	Sand	Iron Powder Waste	Gravel #1	Doped Gravel #1
Specific Gravity (g/cm³)	2.630	4.364	2.730	2.902
Absorption (%)	0.481	0.000	2.163	0.620
Fineness Modulus	1.650	1.540	4.840	4.930

In Fig. 3 and Fig. 4, the particle size curve of the fine materials and coarse aggregates used are presented.

Compound® adhesive, manufactured by Vedacit®, was used to fix the iron powder to the Gravel #1, which consists of two components: Epoxy Resin (A) and Polyamine (B). The specific mass, appearance and color are shown in Table III. The recommended dosage by the manufacturer is 1.8 kg / m² / mm.

Table III. Specific Gravity, Aspect and color of Compound® adhesive

Properties	A Product (Epoxy Resin)	B Product (Polyamine)
Specific Gravity (g/cm³)	1.800	1.800
Aspect	Viscous	Liquid
Color	White	Black

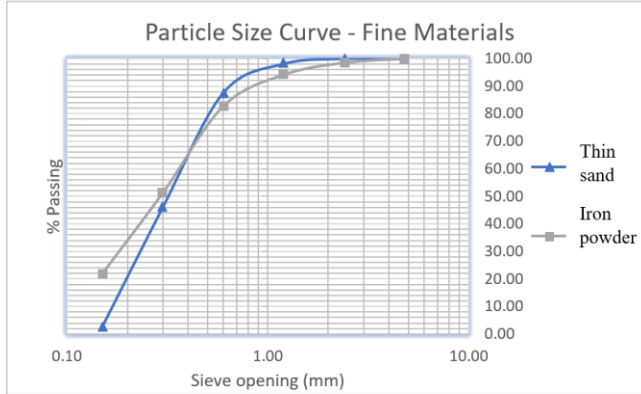

Fig. 3. Particle size curve of Fine Materials

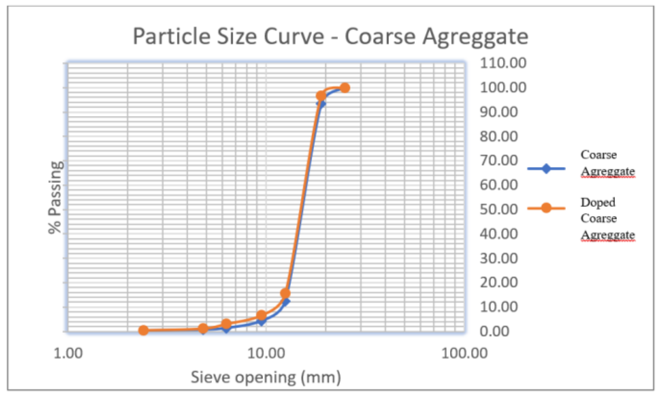

Fig. 4. Particle size curve of Coarse Aggregate

The water used in the concrete mixture was obtained through the public urban supply system in the city of Ilha Solteira-SP, Brazil

III. METHOD

For the mixture proportions and technological control of concrete with doped aggregate used in this work, the mixture proportions method proposed by [16] was used. Two concrete mixture were prepared, maintaining the same characteristics and proportions: in one of them natural gravel was used, without any treatment, and in the other, doped gravel.

Doping of Coarse Aggregate

For the doping of coarse aggregate, the following materials were used: gravel 1, epoxy adhesive and iron powder. All the gravel, already dry and separated, was spread over a plastic canvas placed on the floor, forming a layer with a regular thickness.

Then, the mixture of components A and B of the epoxy adhesive was promoted, in the proportion 1: 1, according to the manufacturer's recommendation, until it became a homogeneous mixture.

Using a manual process, the whole gravel was mixed with the epoxy adhesive, until, visually, the entire surface of the gravel was surrounded by the epoxy adhesive.

Subsequently, the previous process was repeated, adding the iron powder in abundant quantities on the gravel surrounded by the epoxy adhesive. Finally, the period of 7 days was waited for the total cure of the epoxy adhesive as recommended by the manufacturer.

Experimental Mixture Proportions

For the application of the method proposed by [16], in the reference concrete, the following dosing parameters were adopted: slump of 80 ± 20mm, compressive strength corresponding to 25MPa, mortar content of 55% and water / cement ratio initial value equal to 0.5.

Considering the correlation based on the "Law of Molinari", the proportions of the materials for the reference feature are those explained in Eq. (1).

$$C_c = \frac{1000}{\frac{1}{\gamma_c} + \frac{a}{\gamma_a} + \frac{b}{\gamma_b} + \frac{a}{c}} \tag{1}$$

Onde:

γ_c, γ_a e γ_b are the actual Specific Gravity of cement, sand and coarse aggregate, respectively.

A and b are the mass (kg) of sand, coarse aggregate, respectively.

Cc is the consumption of cement in kg / m³.

Having the properties of the materials, and applying the correlation explained in Eq. (1), the cement consumption of the mixture is that obtained in Eq. (2).

$$C_c = \frac{1000}{\frac{1}{3,00} + \frac{2,3}{2,633} + \frac{2,7}{2,689} + 0,5} = 368,87 kg / m^3 \tag{2}$$

To perform the mixture proportions of the reference and doped concrete, the same processes and material quantities were used. Mixing was carried out as follows: all the coarse aggregate was placed in the mixer, followed by sand and half of the water, mixing these materials for 1.5 minutes. Subsequently, the cement and the rest of the water were added, mixing for another 3 minutes. After the mixing procedure was finished, the slump test was carried out. When the slump was lower than desired, more water was included, and the mixer was turned on for another 3 minutes. The Table IV presents the basics characteristics of mixture proportions maked.

Axial Compression and Young Modulus Tests

Axial compression was obtained through tests of specimens with dimensions 10x20cm, according to the guidelines of Brazilians Standard NBR 5738 [17] and NBR 5739 [18], where the compression calculation is given by eq. (3).

$$f_c = \frac{4F}{\pi D^2} \tag{3}$$

Where:

fc is the compression strength, in Megapascal (MPa);

F is the maximum strength resisted, in Newtons (N);

D the diameter of the specimen being cylindrical, in millimeters (mm).

Table IV. Basics Characteristics of Mix Proportions

Items	Definitions (of age 28 days)	
1. Concrete Reference	1 – Refence	2 - Doped
2. Maximal size of coarse aggregate (mm)	9,5	9,5
3. Desirable slump (mm)	7,2 ± 0,2	8,7 ± 0,8
4. Ordinary Portland Cement (OPC)	CPII E-32	CPII E-32
5. Water/cement relation	0,52	0,52
6. Aditives; marca, tipo e porção	Not used	
7. Age of specimens (days)	28	28
8. Mix proportions (1:a:b:a/c) for the experimental mixture, by mass (kg/kg)	1:2,3:2,7:0,54	1:2,3:2,7:0,52

The modulus of elasticity was calculated using Methodology A - σ fixed tension, according to Brazilian Standard NBR 8522 [19], in GPa. The expression used is that obtained in Eq. (4).

$$E_{ci} = \frac{\Delta\sigma}{\Delta\varepsilon} 10^{-3} = \frac{\sigma_b - 0,5}{\varepsilon_b - \varepsilon_a} 10^{-3} \tag{4}$$

Where:

σb is the highest stress, (σb = 0.3 fc), or another stress specified in the project, in megapascals (MPa);

0.5 is the basic stress, also expressed in megapascals (MPa);

ξb is the average specific strain, of greater stress, of the specimens;

ξa is the average specific strain, of the specimens under basic stress.

Diametrical Compression Test

The tensile by diametrical compression was obtained through tests of specimen with dimensions 10x20cm according to the guidelines of Brazilian Standard NBR 7222 [20].

The tensile strength by diametrical compression must be calculated using Eq. (5).

$$f_{c1,sp} = \frac{2F}{\pi d l} \tag{5}$$

Where,

fct, sp is the tensile strength by diametrical compression, in Megapascal (MPa);

F is the maximum force obtained in the test, in newtons

(N);

d is the diameter of the specimen, in millimeters (mm);

l is the length of the test piece, in millimeters (mm).

Absorption Test

The absorptions of the materials used are shown in Table V and were measured according to Brazilian Standard NBR – 9778 [21].

Table V. Concrete Absorption

Concrete Specimen	Absorption (%)
1-Reference	6.61±0.24 (3.57)
2-Doped	5.01±0.19 (3.88)

IV. RESULTS

The results of the slump test are shown in Table VI.

Table VI. Slump test

Referência (cm)	Dopado (cm)
7.2 ± 0,2	8.7 ± 0,8

Analyzing the results of the slump test, it is possible to verify that the doped aggregate improved the workability of the produced concrete, since its aggregates, despite having a rougher surface, also presented a more regular shape.

The results of resistance to diametral compression, axial compression and modulus of elasticity are shown in Table VII.

Table VII. Results

Test	1-Reference (MPa)	2-Doped (MPa)
Diametral compression	3.68±0.32 (8,77)	4.21±0.17 (4,05)
Axial Compression	23.99±1.27 (5,27)	27.31±1.38 (5,04)
Young Modulus	39,639.13±9,387.39 (23.68)	30,214.17±3,987.64 (13.20)

For analysis of diametral compression results, the graphic of Fig. 5 was maked.

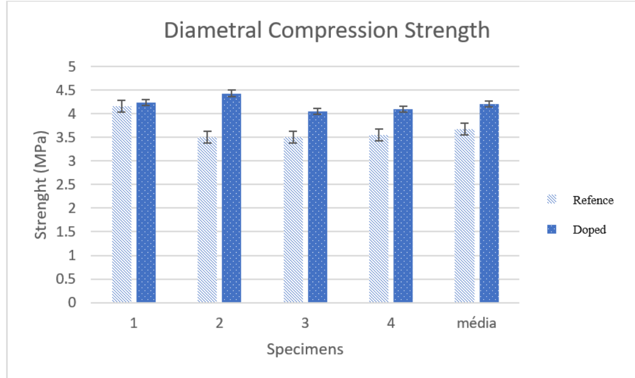

Fig. 5. Influence of mix proportions in Diametral Compression Strength

It's possible to verify that in the entire sample space the results of resistance to diametrical compression were higher for the doped concrete trace. In addition, it appears that the average strength was 14.34% higher and that the standard deviation decreased in the traces of doped concrete.

To analyze the results of diametrical compression, the graph in Fig. 6 was marked.

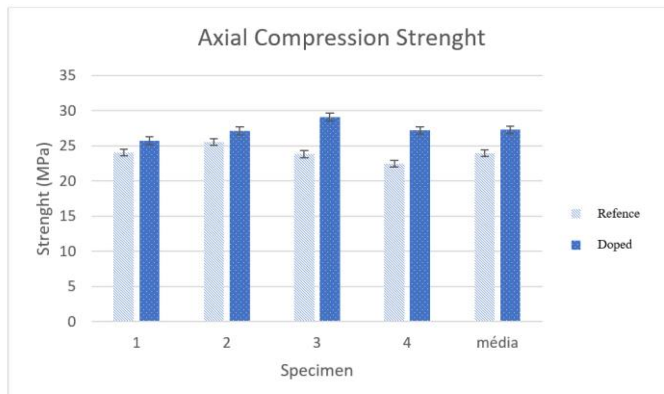

Fig. 6. Influence of mix proportions in Axial Compression Strength

It's possible to verify that in the whole sample space the results of resistance to axial compression were higher for the doped concrete trace. In addition, it appears that the average strength was 13.83% higher and that the standard deviation decreased in the doped concrete lines.

To analyze the results of the modulus of elasticity, the graph in Fig. 7 was maked.

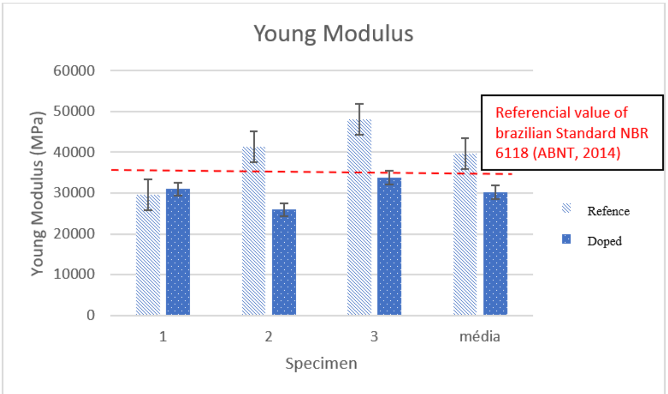

Fig. 7. Influence of mix proportions in Young Modulus of concretes

It's possible to verify that in the entire sample space the results of Young Modulus were lower for the trace of doped concrete. In addition, it appears that the mean of the modulus of elasticity was 23.78% lower and that the standard deviation decreased in the specimens of doped concrete. Comparing with the average value calculated based on Brazilian Standard NBR 6118 [22], it is observed that the values for the concrete reference are much higher than expected, indicating that there may have been some problem during the test.

V. CONCLUSION

After analyzing and comparing the results, it can be seen that the doping technique used contributed to increase in the strength of the concrete, both to axial compression (increase of 13,83%) and to tensile by diametrical compression (increase of 14,34%).

The aggregate doping technique also reduced the absorption (reduction of 24,20%), a fact that can be explained by a possible waterproofing of the crushed gravel with Compound®.

In the fresh state, this possible waterproofing represented a gain in the workability of the concrete, represented by an increase of 20,83% in the reduction of the cone trunk. In the long term (hardened concrete), the reduction in the absorption rate may contribute to an increase in the useful life of the structure and a reduction in the possibility of pathological manifestations in apparent structures, such as leaching and stalactites, for example.

In addition to the improvements in the mentioned properties, the incorporation of the residue of the iron powder for the doping of the aggregate allowed the production of a more sustainable concrete.

It can also be seen that the doping of the aggregate contributes to a decrease in the standard deviation of the tests, showing that a mixture with a more homogeneous behavior was obtained. The use of doped aggregates was found to provide a concrete with rigidity compatible with that calculated according to NBR 6118 [22].

Although the method was effective, a more comprehensive study is recommended for future work, also evaluating other doping techniques and, also, the inclusion of pozzolanic or hydraulic materials, which will contribute to even greater improvements, in addition to being, environmentally friendly.

ACKNOWLEDGMENT

To Prof. José Luiz Pinheiro Melges, pH.D, for his guidance on using the NEPAE Laboratory equipment.

To CNPq and INAGBE, for funding master's scholarships.

To the technicians of the Central Laboratory of Civil Engineering, FEIS-UNESP, for carrying out the experimental tests.

To Prof's Maria da Consolação Fonseca de Albuquerque, pH.D, Mauro Mitsuuchi Tashima, pH.D and César Fabiano Fioriti, pH.D, for authorizing the publication of this work.

REFERENCES

[1] PISELLO, Anna Laura; FABIANI, Claudia; COTANA, Franco. *New experimental technique to investigate the thermal behavior of PCM/doped concrete for enhancing thermal/energy storage capability of building envelope*. 72nd Conference of the Italian Thermal Machines Engineering Association, ATI2017, 6-8 September – Enercy Procedia 126 - ELSEVIER, Lecce, Italy, p. 139-146, 2017.

[2] E. Benhelal, G. Zahedi, E. Shamsaei, A. Bahadori, *Global strategies and potentials to curb CO2 emissions in cement industry*, J. Clean. Prod. 51 (Supplement C) (2013) 142–161.

[3] SNIC. *Resultados Preliminares de Setembro 2019*. Disponível em: http://snic.org.br/numeros-resultados-preliminares-ver.php?id=41¬icia=Resultados%20Preliminares%20de%20Setembro%202019. Acesso em: 10 out. 2019.

[4] M. Schneider, M. Romer, M. Tschudin, H. Bolio, *Sustainable cement production—present and future*, Cement Concr. Res. 41 (7) (2011) 642–650.

[5] MEHTA, P. K.; MONTEIRO, P.J. *Concreto: Microestrutura, Propriedades e Materiais* 4ª edição (inglês) / 2ª edição (português) Departamento de Engenharia Civil e Ambiental da Universidade da Califórnia em Berkeley, Estados Unidos, 2014.

[6] MENESES, I. A. *A Avaliação de concreto com adição de fibras PET submetido a altas temperaturas*. Dissertação (mestrado) - Universidade Federal do Rio Grande do Norte. Centro de Tecnologia. Programa de Pós-graduação em Engenharia Civil. Natal/RN. 2011. 89f.

[7] TRIGO, A P M. *Estudo da dopagem de agregados para a finalidade de uso em concretos estruturais*. Tese (Doutorado em Engenharia Civil) – Escola de Engenharia deSão Carlos e Universidade de São Paulo. São Carlos, p. 9-15. 2012.

[8] LIBORIO, J. B. L.; CASTRO, A.L.; da SILVA, F.G.; SILVA, V.M. *Demystifying the production process of high performance and high strength concretes – design considerations.* IV High Performance Concrete Structures. 2005.

[9] NEŽERKA, V. et al. *Impact of silica fume, fly ash, and metakaolin on the thickness and strength of the ITZ in concrete*. Cement and Concrete Composites: ScienceDirect, Czech Republic, v. 103, n. 1, p. 252-262, Out/2019. Disponível em: https://www.sciencedirect.com/science/article/abs/pii/S0958946519300265?via%3Dihub. Acesso em: 29 out. 2019.

[10] COUTINHO, A. S. (1988) *Fabrico e propriedades do betão*. Laboratório Nacional de Engenharia Civil, Lisboa, vol. I, p. 401.

[11] KIRGIZ, Mehmet S. *Strength gain mechanism for green mortar substituted marble powder and brick powder for*

Portland cement. European Journal of Environmental and Civil Engineering, v. 20, n. S1, p. s38 - s63, 2016.

[12] WANG, Yonggui; HUGHES, Peter; NIU, Haicheng; FAN, Yuhui. *A new method to improve the properties of recycled aggregate concrete: Composite addition of basalt fiber and nano-silica*. Journal of Cleaner Production 236 – ELSEVIER, p. 1- 12, 2019.

[13] GUERRA, André Nillo Lopes Pires. *Caracterização e utilização de rejeito de minério de ferro pellet feed em pavimentos de blocos intertravados de concreto*. Dissertação (mestrado) - Universidade Federal de Minas Gerais. Escola de Engenharia da UFMG. Belo Horizonte, 2014. 124f.

[14] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 16697:Cimento Portland - Requisitos*. Rio de Janeiro, 2018.

[15] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 7211: Agregados para concreto - Especificação*. Rio de Janeiro, 2009.

[16] HELENE, P.; TERZIAN, P. *Manual de dosagem e controle do concreto*. 1. ed. São Paulo, Ed. PINI, 1992.

[17] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 5738: Concreto – Procedimento para moldagem e cura de corpos de prova.* Rio de Janeiro, 2015.

[18] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 5739: Concreto – Ensaios de compressão de corpos de prova cilíndricos.* Rio de Janeiro, 2007.

[19] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 8522: Concreto – Determinação do módulo estático de elasticidade à compressão.* Rio de Janeiro, 2017.

[20] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 7222: Concreto e argamassa — Determinação da resistência à tração por compressão diametral de corpos de prova cilíndricos.* Rio de Janeiro, 2011.

[21] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT. *NBR 9778: Argamassa e concreto endurecidos – Determinação da absorção de água, índice de vazios e massa específica.* Rio de Janeiro, 2005.

[22] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. *NBR 6118: Projeto de estruturas de concreto – Procedimento.* Rio de Janeiro, 2014