ABSTRACT
This paper had the objective of statistically analyzing the effects of the substitution of high levels of fly ash in concretes. It was produced reference concretes and concretes containing fly ash with substitutions of 30, 50, and 70% of the Portland cement mass. The water/cement material relations in these concretes ranged between 0.45, 0.50, and 0.55. With these parameters, it was conducted a factorial project 3² to analyze the results concerning the mechanical properties of compressive strength and consistency index of the concretes. It was elaborated nine concrete mixtures and their replicates. With the results obtained, it was performed a statistical analysis using ANOVA, Pareto Diagram and Tukey Test. The regression models obtained were adequate, with acceptable values of R². The validation of the model occurred through two concretes with water/cement material ratios of 0.475 and 60% of substitution of fly ash in relation to the Portland cement. This mixture presented an average compressive strength of 33.38 MPa at 115 days. The study showed that the ground fly ash was the most significant factor to the mechanical resistance of the concretes, significantly contributing to the increase of the compressive strength in advanced ages.

KEYWORDS: Fly ash concrete. High-volume fly ash. Mechanical properties. Statistical analysis.

ANÁLISE ESTATÍSTICA DAS PROPRIEDADES DE CONCRETOS COM ALTOS VOLUMES DE CINZA VOLANTE EM SUBSTITUIÇÃO AO CIMENTO

RESUMO
Este artigo tem como objetivo analisar estatisticamente os efeitos da substituição de elevados teores de cinza volante em concretos. Foram produzidos concretos de referência e concretos contendo cinza volante em substituição de 30, 50 e 70% da massa do cimento Portland. A relação água/materiais cimentícios destes concretos variou entre 0,45, 0,50 e 0,55. Com esses parâmetros, foi realizado um projeto fatorial 3² para analisar os resultados referentes à propriedade mecânica de resistência à compressão e abatimento de tronco de cone dos concretos. Foram elaboradas nove misturas de concreto e suas réplicas. Com os resultados obtidos, foi desenvolvida uma análise estatística incluindo ANOVA, diagrama de Pareto e teste de Tukey. Os modelos de regressão obtidos se mostraram adequados, com valores de aceitáveis de R². A validação do modelo foi desenvolvida através de dois concretos com relação água/materiais cimentícios de 0,475 e 60% de substituição de cinza volante em relação à massa do cimento Portland. Esta mistura apresentou resistência à compressão média de 33,38 MPa aos 115 dias. O estudo mostrou ainda que a cinza volante moída foi o fator que apresentou maior significância na resistência mecânica dos concretos, contribuindo significativamente para o aumento da resistência à compressão em idades avançadas.

PALAVRAS-CHAVE: Concreto com cinza volante, Elevado volume de cinza volante, Propriedades mecânicas, Análise estatística.
1 INTRODUCTION

Concrete systems using a high content of fly ash are an excellent option technically, environmentally, and economically for conventional concrete since their use results in more durable structures and cause less environmental impact. The content of fly ash should be superior to 50% in mass of the total cement material to meet the definition of high fly ash concrete (Malhotra & Mehta, 2012).

In the past, concrete with a high fly ash content did not show good mechanical resistance performance since the waste produced by the old thermoelectric plants presented a larger average particle diameter than the current ones and normally presented high content of carbon. However, recent experiments have shown that the fly ash obtained from modern powerplants is characterized by low carbon content and fine particles (Mehta & Monteiro, 2014).

The use of mineral additives as a substitute for Portland cement reduces the consumption of cement in the production of concrete, thus reducing the emission of carbon dioxide into the atmosphere. Therefore, it is possible to say that Brazilian CP III cement, with 75% blast furnace slag, and CP IV cement, with 50% fly ash, maximum limits established by NBR 16697 (ABNT, 2018), present the best environmental performance among all cement produced in Brazil.

Research conducted by Yildirim and Sumer (2013), Supit, Shaikh and Saker (2014), and Yu et al. (2017) showed that fly ash is one of the most commonly used mineral additives to replace Portland cement. In addition to economic and environmental advantages, concretes made with fly ash present good performance and higher durability, lower hydration heat, and less tendency to retraction during the curing when compared to conventional concretes since the amount of cement available for hydration is reduced (Moghaddam et al., 2019). Thus, it is highly recommended for works where temperature rise must be controlled, such as dams and large foundation blocks (Ignjatovic et al., 2017).

Large quantities of fly ash incorporated to the concrete change the microstructure of the cementitious paste, directly affecting the mechanical behavior of the mixture on a macroscale. The pozzolanic reaction between the fly ash and the CH producing additional secondary C-S-H gels, present mainly in the transition zone, makes the matrix more compact and impermeable resulting in denser microstructure (Sun et al., 2019).

Concretes with a high content of fly ash tend to present higher elasticity modulus, lower permeability, and a lower tendency to cracking and creep than conventional concretes with the same compressive strength (Hemalatha & Ramaswamy, 2017).

This study aims at statistically analyzing the mechanical properties of concretes made of high fly ash replacing the CPV Portland cement using the experimental planning technique.

The Design of Experiments (DOE) is a scientific process with a wide range of applications in various fields of knowledge. The method consists of planning, outlining, and analysis of experiments, contributing to the achievement of objective and effective results (Antony, 2014).
The full factorial planning is the most advisable technique to study the effect of two or more influencing variables in a given experiment. This technique causes all factors to be varied together, that is, in each attempt or replicate, all possible combinations of the levels of each variable are investigated. With this approach, it is possible to estimate all the effects of a factor, leading to conclusions that are valid within the analyzed conditions, remaining the only form to discover interactions among factors (Montgomery, 2009).

The usual nomenclature of a factorial design is represented by $b^k$, where $k$ is the number of factors adopted and $b$ is the number of levels chosen. The factors express each variable of the system. The levels represent the operating conditions of the control factors investigated in the experiments (Montgomery, 2009). Once the experiment is outlined and the data obtained, the model was adjusted, which must always be evaluated through Analysis of Variance (ANOVA). This technique assumes the hypothesis that the terms related to the errors are independent and normally distributed with mean zero and constant variance (Montgomery, 2009).

According to Devore (2006), the analysis of variance, or ANOVA, consists of a statistical procedure suitable for evaluating the quantitative responses in planned experiments. Linear regression analysis is also very useful in planned experiments that include factors at continuous levels. In this case, the analysis of variance is used to identify significant factors followed by the regression analysis used to construct a model that incorporates these factors.

Complementing the analysis of variance, it is common to perform a multiple comparison test such as the Tukey test. This tool allows us to identify the magnitude of the differences between all pairs of means obtained with each of the levels and factors. The Pareto graph also allows the visualization of the magnitude and importance of each of the analyzed effects, helping to determine the conditions of the factors that lead an optimal value for the response variable (Antony, 2014).

In this study, it was assessed concretes with water/cement material ratio ranging between 0.45, 0.50, and 0.55, using different contents (30, 50, and 70%) of fly ash in substitution of the Portland cement. The proposed regression models were validated for high content of fly ash, 60% replacing Portland cement, and presented 33.38 MPa of compressive strength at 115 days, which confirms its use as structural concrete.

2 MATERIALS AND METHODS

The materials used to make the concrete mixtures were: CPV ARI Portland cement, according to specifications of NBR 16697 (ABNT, 2018); fine aggregate (river sand) from northeastern Santa Catarina with a fineness modulus of 2.45, maximum diameter of 2.36 mm and a specific mass of 2.60 g/cm$^3$; coarse aggregate (gravel) with a maximum grain size of 19 mm and a specific mass of 2.76 g/cm$^3$; fly ash from the thermoelectric power plant of the Jorge Lacerda Thermoelectric Complex; water supplied by the local concessionaire; and plasticizer additive in the dosage of 0.4% for the mass of cementitious materials.
The CPV ARI Portland cement was selected for containing no mineral addition, which could influence the results of the research. Furthermore, this cement presents the smallest particle size and, therefore, greater surface area, ensuring superior resistance in the early ages. It is the cement with the highest content of clinker found in the Brazilian market (95% clinker) and contains a maximum addition of 5% of limestone filler. The use of ARV CPV compensates the low compressive strength of fly ash concretes at early ages.

The chemical composition of the fly ash was analyzed using X-ray fluorescence spectroscopy (Table 1). It was verified that the concentrations of silica (SiO₂), alumina (Al₂O₃), and iron oxide (Fe₂O₃) represent 88.52% of the mass of the material, being in accordance with ASTM C618-17 (ASTM, 2017), which establishes a minimum value of 70% for the sum of these three elements. According to Cho, Jung, and Choi (2019), these three components significantly affect the pozzolanic activity of a material.

| Element | Mass content (%) | Element | Composition (%) |
|---------|------------------|---------|-----------------|
| SiO₂    | 48.785           | V₂O₅   | 0.092           |
| Al₂O₃   | 30.271           | MnO     | 0.057           |
| Fe₂O₃   | 9.460            | ZnO     | 0.053           |
| K₂O     | 4.194            | SrO     | 0.040           |
| CaO     | 2.299            | Cr₂O₃   | 0.031           |
| TiO₂    | 1.764            | Rb₂O    | 0.029           |
| SO₃     | 1.176            | Y₂O₃    | 0.027           |
| P₂O₅    | 0.502            | Loss of ignition | 1.220 |

The physical characteristics of the in-nature material to complement the fly ash characterization tests were determined, after having subjected the material to a 10-minute milling process in a ball mill. It was observed that the in-nature ash presents a specific mass of 2.17 g/cm³, the particle size of 45 μm, and performance index with Portland cement equal to 101.88%. The ground ash, with a specific mass of 2.25 g/cm³, presented a Portland cement performance index of 114.92%, in compliance with the minimum criterion of 90% defined by NBR 12653 (ABNT, 2015). The increase of the pozzolanic activity of the material after the milling process occurs due to the decrease in the average particle size and the consequent increase of the specific surface, allowing more contact between the reactive elements of the mixture.

Despite this, a sieving test showed that, without the milling process, the fly ash cannot be used as pozzolanic material, given that the percentage of mass retained in the 45 μm sieve was of 43%, considerably higher than the limit of 20% established in the ASTM C618-17 (ASTM, 2017). However, after 10 minutes of milling, 100% of the material passed through the 45 μm sieve, confirming the pozzolanicity of the ground ash. Therefore, it was defined that the fly ash should be ground for 10 minutes for its use in concretes with high fly ash content. Figure 1 shows the particle diameter size distribution for ground fly ash: 100% of the particles have a diameter smaller than
41 μm, 90% are smaller than 17.78 μm, 50% have a diameter smaller than 8.32 μm, and 10% of the particles, less than 1.45 μm.

After analyzing the properties of the fly ash, it was conducted preliminary tests comparing concretes with a partial replacement of the Portland cement mass with fresh and ground fly ash. Three mixtures were prepared: A - with 100% Portland cement, B - using 25% of fly ash replacing the Portland cement, and C - using 25% of ground fly ash to replace the Portland cement. The produced concretes were evaluated according to their compressive strength at 28 days and 115 days, void index, specific mass, and water absorption.

Subsequently, it was developed an experimental design based on a 3² factorial design, that is, two factors and three levels of treatment for each factor. Factor 1 is the water/cement material ratio, with the following treatment levels: 0.45, 0.50, and 0.55. Factor 2 corresponds to the fly ash content replacing the Portland cement: 30, 50, and 70% of replacement. The ash used was previously milled for 10 minutes in a ball mill. Nine concrete mixtures were fabricated (10 specimens for each mixture) and their respective replicates (Table 2). In addition to the fly ash mixtures, a reference concrete without ash was elaborated. The experimental design was randomly outlined using the Statistica 13.0 software (Startsoft). For the factorial design, ground ash was used as it performs better in preliminary tests.
Table 2: Mixtures of the factorial experiment for the volume of 1 cubic meter.

| Mixture | w/cm | CV Content (%) | Cement (kg) | CV (kg) | Sand (kg) | Gravel (kg) | Water (kg) |
|---------|------|----------------|-------------|---------|-----------|-------------|------------|
| 1       | 0.45 | 30             | 328.13      | 140.63  | 660.63    | 1043.13     | 211.25     |
| 2       | 0.45 | 50             | 234.38      | 234.38  | 660.63    | 1043.13     | 211.25     |
| 3       | 0.45 | 70             | 140.63      | 328.13  | 660.63    | 1043.13     | 211.25     |
| 4       | 0.50 | 30             | 295.63      | 126.88  | 706.25    | 1040.63     | 211.25     |
| 5       | 0.50 | 50             | 211.25      | 211.25  | 706.25    | 1040.63     | 211.25     |
| 6       | 0.50 | 70             | 126.88      | 295.63  | 706.25    | 1040.63     | 211.25     |
| 7       | 0.55 | 30             | 268.75      | 115.00  | 743.13    | 1038.75     | 211.25     |
| 8       | 0.55 | 50             | 191.88      | 191.88  | 743.13    | 1038.75     | 211.25     |
| 9       | 0.55 | 70             | 115.00      | 268.75  | 743.13    | 1038.75     | 211.25     |

To estimate the water/cement material ratio of the reference mixture, it was adopted a \( f_{cj} \) of 35 MPa and w/cm ratio of 0.50. Thus, the unit reference mixture (in mass) can be represented in the proportion of 1:1.68:2.47:0.50. That is, for every 1 kg of cement, 1.68 kg of fine aggregate, 2.47 kg of coarse aggregate, and 0.50 kg of water is required.

From the reference unit mixture (Table 3), two other mixtures were calculated, denominated mixture X (with water/cement material ratio of 0.45) and mixture Y (with water/cement material ratio of 0.55), considering the limits of the concrete workability. To prepare these reference mixtures (X and Y), it was kept the cohesion (\( \alpha \)) and the water-dry materials ratio (H) constant, varying only the water/cement material ratio.

Table 3: Mixtures of control concrete (in mass).

| Mixture (in mass) | Cement | Sand | Gravel | Water |
|-------------------|--------|------|--------|-------|
| X                 | 1      | 1.41 | 2.22   | 0.45  |
| Reference         | 1      | 1.68 | 2.47   | 0.50  |
| Y                 | 1      | 1.94 | 2.72   | 0.55  |

The concrete was produced in an inclined-axis mixer, following the recommendations of NBR 12821 (ABNT, 2009). A total of 210 cylindrical specimens were molded with 10 cm in diameter and 20 cm of height. The specimens were manually packed in two equally thick layers with 12 strokes each and then cured in a tank filled with calcium hydroxide saturated water at 23ºC.

With the concretes in the fresh state, it was analyzed the workability by consistency index, according to NBR NM 67 (ABNT, 1998).
In the hardened state, it was conducted the compressive strength test at 28 and 115 days, using a hydraulic press with nominal capacity of 200 tons. The specimens were previously rectified and maintained in the saturated condition. The loading was applied at a constant rate of 0.45 MPa/s until verifying the rupture of each sample.

The results obtained with the factorial design were submitted to the analysis of variance (ANOVA) to verify the influence of fly ash content and water/cement material ratio in the measured properties (Montgomery, 2009).

A level of significance of 7% was adopted for the statistical analyzes of all tests performed.

The regression model proposed through the factorial design was valued through a new mixture (denominated V1) with factors within the range studied. For this, it was defined a fly ash content of 60% and water/cement material ratio of 0.475. The proportion used for V1 was: 1:1.54:2.35:0.475. Its physical and mechanical properties were studied and later compared with an identical mixture without the addition of fly ash, denominated VP. Sixteen (16) cylindrical specimens were molded for each mixture to determine the compressive strength at the ages of 28 and 115 days.

3 RESULTS AND DISCUSSION

3.1 Influence of the grinding process of the fly ash on the concrete

The results of the consistency index for the concretes without fly ash and with in nature and ground ash are presented in Table 4.

| Time of reading | Reference concrete | Concrete with in nature ash | Concrete with ground ash |
|-----------------|--------------------|-----------------------------|-------------------------|
| Initial reading | 160                | 190                         | 80                      |
| After 15 min    | 70                 | 150                         | 40                      |
| After 30 min    | 30                 | 120                         | 30                      |
| Consistency loss| 81%                | 37%                         | 62%                     |

The use of fly ash improves the rheological properties of the concrete since its spherical and regular shape reduces the internal friction between the aggregates and the paste, increasing the concrete slump from 160 mm (standard mix) to 190 mm (mixing with in nature ash). In the case of the ground fly ash, it was verified a lower fluidity of 80 mm since the milling process increases the specific surface area of the material, resulting in an increase in water adsorption. This reduces the amount of free water in the mixture, impairing the fluidity of the concrete.

Even so, the use of in nature and ground fly ash decreased the loss of concrete slump by 37% and 62%, respectively, when compared to the standard mixture, where the consistency loss
as of 81%. The lower the consistency loss, the greater the workability of the concrete (Neville, 2016). This is an important property in machined concretes, where the work site (discharge) is often far from the Concrete Company.

Figure 2 presents the results of the mechanical strength test for concrete specimen compression at the ages of 7 and 28 days. It can be observed that the two mixtures with fly ash presented lower compressive strength than the reference concrete at both ages. The reduction is very significant at 7 days: 18.4% and 32.2% for concretes with the ground and in nature ash, respectively, when compared to the compressive strength of the reference concrete. This can be explained by the high quantities of fly ash contributing little to the compressive strength of the mixture in the early ages since the degree of reaction of the mineral addition is lower than that of the cement. Thus, pozzolanic reactions tend to occur late (Wang & Park, 2015).

![Figure 2: Compressive strength tests.](image)

Nevertheless, at 28 days, it can be noticed that the concrete without fly ash and ground ash presents very similar values of compressive strength, with a difference of only 14.3% between them. This is because the increase in compressive strength at advanced ages is the tendency in concretes with pozzolanic materials, given that the pozzolanic reaction continues after the hydration of the cement. The pozzolanic reaction is slow and requires cement hydration products to occur (Bae et al., 2014). Therefore, it is natural that the resistance gain is only observed at ages after the hydration of a large portion of the cement in the mixture.

It is also worth mentioning the great difference between the mechanical resistance to compression of the concrete with in nature and ground fly ash. This confirms what has already been shown in the material characterization, on the pozolanicity and reaction capacity of the
ground ash when compared to in nature fly ash, justifying the choice of the material after the grinding process.

Table 5 presents the values of the voids index, specific mass, and water absorption tests for the concrete without ash, with ground ash, and with in nature ash. There is a significant reduction in the void index and water absorption of the fly ash concretes. This reduction is even more striking when the residue is ground.

| Tests               | Reference concrete | Concrete with ground ash | Concrete with in nature fly ash |
|---------------------|--------------------|--------------------------|--------------------------------|
| Voids index (%)     | 10.34              | 9.16                     | 9.52                           |
| Specific mass (g/cm³) | 2.27               | 2.13                     | 2.12                           |
| Water absorption (%) | 4.55               | 4.04                     | 4.25                           |

This occurs because the fly ash physically acts on the microstructure of the concrete, improving the packing conditions of the cement matrix and decreasing the voids present in the paste (Xu et al., 2017). The material also contributes by reducing the water absorption and permeability, ensuring greater durability to the concrete since it makes difficult the entrance of aggressive agents. Chemically, the pozzolanic reaction between fly ash and calcium hydroxide (CH) present in the of the Portland cement hydration, produces hydrated calcium silicate (CSH) and hydrated calcium aluminosilicate (Bae et al., 2014), which makes the matrix even more compact and impermeable (Neville, 2016).

It can also be reported that the concrete became lighter, which was expected since the specific mass of the fly ash is smaller than the specific mass of the CPV ARI cement.

3.2 Statistical Analysis of the Factorial Design

Table 6 shows the values of the Slump test (consistency index) and the mean compressive strength results at 28 and 115 days of curing of the 9 factorial mixtures and their replicates.

The obtained data were submitted to the analysis of variance, considering only the statistically significant values, assuming a significance level α of 7% for the hypothesis test, where, in the case of p ≤ α, the null hypothesis should be rejected.

| Mixture | Factor w/cm | Ground fly ash (%) | Slump (mm) | RC28 (MPa) | RC115 (MPa) |
|---------|-------------|--------------------|------------|------------|-------------|
| 1       | 0.45        | 30                 | 120        | 40.75 ± 5.24 | 47.08 ± 5.39 |
| 2       | 0.45        | 50                 | 75         | 28.93 ± 4.43 | 35.70 ± 4.00 |
| 3       | 0.45        | 70                 | 60         | 20.68 ± 1.51 | 29.42 ± 1.90 |
| 4       | 0.50        | 30                 | 175        | 30.58 ± 1.09 | 40.41 ± 3.53 |
| 5       | 0.50        | 50                 | 110        | 29.14 ± 2.40 | 37.84 ± 2.36 |

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3.3 Compressive strength at 28 and 115 days

Equations 1 and 2 present the regression models used to represent the effect of the studied factors on the compressive resistance of the concretes at 28 and 115 days.

\[
Y_{28} = 112.959 - 0.804x_1 - 0.005x_1^2 - 160.317x_2 + 1.855x_1x_2 \\ (Eq. 1)
\]

\[
Y_{115} = 107.939 - 0.433x_1 - 0.009x_1^2 - 143.771x_2 + 1.799x_1x_2 \\ (Eq. 2)
\]

\(Y_{28}\) and \(Y_{115}\) correspond to the compressive strengths at 28 and 115 days, respectively, in MPa; \(X_1\) is the percentage of fly ash replacing the Portland cement, and \(X_2\) is the water/cement material ratio (w/cm) of the mixture.

The proportion of data variability explained by the studied factors (water/cement material and fly ash) was of 0.934 for the 28 days compressive strength and 0.926 for the 115 days compressive strength. Moreover, there were no problems of mismatch between the replicates (\(p = 0.1726\) for RC28 and \(p = 0.0419\) for RC115). This means that the measured values for the compressive strength of the replicates at both ages are acceptable.

The analysis of the residues (the difference between the experimental values and those predicted by the variance analysis model equation) indicated that both models are adequate since the residuals present normal distribution and are randomly distributed.

The validation of the models was done using the V1 mixture, containing 60% of fly ash and w/cm factor of 0.475. The results presented in Table 7 show that both equations satisfactorily represent the experimental results of compressive strength since the difference between the experimental mean values and the value predicted by the model is only of 2.8% at 28 days and 2.6% at 115 days.
Table 7: Test for validation and predicted values for RC28 and RC115.

| Factor | Compressive strength at 28 days | Compressive strength at 115 days |
|--------|--------------------------------|---------------------------------|
|        | Coefficient of Regression | Coefficient value | Coefficient of Regression | Coefficient value |
| Constant | 112.959 | 112.96 | 107.939 | 107.94 |
| %Fly ash (L*) | -0.804 | -48.24 | -0.433 | -25.98 |
| %Fly ash (Q*) | -0.005 | -18.00 | -0.009 | -32.40 |
| Factor w/cm (L) | -160.317 | -76.15 | -143.771 | -68.29 |
| 1L by 2L | 1.855 | 52.87 | 1.799 | 51.27 |
| Predicted value (MPa) | 23.44 | | 32.54 | |
| Experimental value (MPa) | 22.78 ± 1.09 | | 33.38 ± 3.99 | |

*L = Linear term: 1L (x1); Q= Quadratic term: 1Q (x2)

Analyzing the Pareto diagrams of compressive strength at 28 days (Figure 3a) and at 115 days (Figure 3b), it was observed that, in both, the fly ash percentage is the most influential factor for the compressive strength.

![Figure 3: Pareto Diagram for the compressive strength: (a) 28 days; (b) 115 days.](image)

The Tukey test for the compressive strength at 28 days (Table 8) subdivided the mixtures considered statistically equal into 7 groups. Group A1, containing concrete with water/cement material ratio of 0.55 and 0.50 and fly ash content of 70%, presented the lowest mean resistance at this age. Group G1, which includes only the mixture with lower water/cement material and lower fly ash content, presented the greatest compressive strength at 28 days.
Table 8: Results of the Tukey test for the compressive strength at 28 days.

| Tukey Test | Treatments | Factor w/cm | Content of fly ash (%) | Means (MPa) |
|------------|------------|-------------|------------------------|-------------|
|            | A1         | 0.50        | 70                     | 14.94       |
|            | A1 B1      | 0.55        | 70                     | 16.64       |
|            | B1 C1      | 0.45        | 70                     | 20.22       |
|            | C1 D1      | 0.55        | 50                     | 22.90       |
|            | E1         | 0.55        | 30                     | 26.78       |
|            | E1 F1      | 0.50        | 50                     | 28.46       |
|            | E1 F1      | 0.45        | 50                     | 28.58       |
|            | F1         | 0.50        | 30                     | 32.02       |
|            | G1         | 0.45        | 30                     | 37.60       |

At 115 days, the concretes were distributed into 4 categories (Table 9). Group A2, which includes the same mixtures of group A1, again showed the lowest values of compressive strength. Group D2, which presented the highest values of compressive strength, also included the mixture with w/cm of 0.50 and the mixture with w/cm of 0.45 of group G1.

Table 9: Results of the Tukey Test for the compressive strength at 115 days.

| Tukey Test | Treatments | Factor w/cm | Content of fly ash (%) | Means (MPa) |
|------------|------------|-------------|------------------------|-------------|
|            | A2         | 0.50        | 70                     | 21.65       |
|            | A2 B2      | 0.55        | 70                     | 23.52       |
|            | B2         | 0.45        | 70                     | 27.38       |
|            | C2         | 0.55        | 50                     | 35.49       |
|            | C2         | 0.55        | 30                     | 35.58       |
|            | C2         | 0.45        | 50                     | 36.72       |
|            | C2         | 0.50        | 50                     | 37.77       |
|            | D2         | 0.50        | 30                     | 43.39       |
|            | D2         | 0.45        | 30                     | 46.63       |

For better visualization of the factor effects (% CV and w/cm), it was elaborated the outlining graphs for the compressive strengths at both ages (Figure 4). It can be observed that the increase of the fly ash content contributes to obtaining concretes with resistance to reduced compression, both at 28 days and at 115 days.
3.4 Slump test

The regression model proposed to represent the effects of the factors studied in the slump test is indicated in Equation 3.

\[ Y_{slump} = -243.819 - 1.979 x_1 + 933.333 x_2 \]  

(Eq. 3)

\( Y_{slump} \) corresponds to the value of the slump measured in mm; \( X_1 \) is the percentage of fly ash replacing the Portland cement, and \( X_2 \) is the water/cement material (w/cm) ratio of the mixture.

The proportion of variability obtained in the analysis of variance (\( R^2 = 0.895 \)) is high, which means that more than 89% of the variability of the results depends on the studied factors, that is, there is no other external factor that could have significantly affected the presented results. It was not identified large variability between the replicates or problems associated with the lack of adjustment, whereas \( p (0.4493) > \alpha (0.07) \).

The Pareto diagram for slump (Figure 5) indicates that both factors considered in the experimental design, water/cement material ratio and fly ash percentage, significantly affect the workability of the concretes, in almost the same proportion. However, the variables have opposite signs, that is, the increase in the relation water/cement material ratio contributes to the increase of the workability of the mixture. On the other hand, the increase in the percentage of fly ash causes the workability of the concretes to reduce.
Figure 5: Pareto Diagram for slump.

Table 10 presents the results of the validation test of the slump model for concrete V1. The difference between the experimentally measured value and the predicted value is of 11%, indicating that the selected regression model is adequate to represent the concrete consistency. The residues are also consistent with normality and randomness.

| Factor                  | Coefficient of Regression | Coefficient Value |
|-------------------------|---------------------------|-------------------|
| Constant                | -243.819                  | -243.819          |
| % Fly ash (L)           | -1.979                    | -118.740          |
| Factor w/cm (L)         | 933.333                   | 443.333           |

| Predicted value (mm)    | 81                        |
| Value measured for V1 (mm) | 90                        |

The outline graph presented in Figure 6 allows us to predict the behavior of concretes with fly ash and water/cement material ratio for the limits studied. Within these ranges, of w/cm ratio between 0.45 and 0.55 and percentage of fly ash between 30% and 70%, the slump of the concretes can vary between 60 mm and 210 mm.
3.5 Physical and mechanical properties of concretes V1 and VP

The slump test for mixtures V1 and VP was of 90 and 130 mm, respectively. The use of 60% of fly ash resulted in less fluidity to the concrete, a result also obtained by Ignjatovic et al. (2017).

In the compressive strength test (Figure 7), the concrete with a high content of fly ash, V1, presented lower compressive strength when compared to the VP concrete in both curing ages, 28 and 115 days. At 28 days, the VP mixture presented a resistance 32.66% superior to the results of the V1 mixture, a difference which decreases to 26.12% at 115 days. This occurs because the V1 mixture presented an increase of 46.53% in resistance from 28 to 115 days, whereas, for the VP mixture, this increase was of 39.31%. This evolution is a tendency in concretes with mineral additions since the pozzolanic reaction is relatively slow and its contribution to the resistance of the mixture occurs mainly in advanced ages (Siddique, 2004).
Table 11 presents the values of the voids index, specific mass, and water absorption tests for concretes VP and V1.

| Tests                  | VP    | V1    |
|------------------------|-------|-------|
| Voids index (%)        | 10.46 | 8.95  |
| Specific mass (g/cm³)  | 2.63  | 2.50  |
| Water absorption (%)   | 4.44  | 3.93  |

The use of fly ash replacing the Portland cement improves the packaging conditions in the concrete due to the filler effect, thus decreasing the void indices within the paste, which also reduces the water absorption of the concrete. A better packaging condition contributes to lower permeability and, consequently, higher durability of the concrete since it hinders the entry of aggressive agents. Furthermore, the pozzolanic reaction between fly ash and CH, present mainly in the transition zone, makes the matrix even more compact and impermeable (Neville, 2016).

4 CONCLUSIONS

- The concrete made with ground fly ash presented lower void index, lower water absorption, and higher refinement of the matrix, and is, therefore, more resistant to the migration of aggressive agents. It was also verified a mechanical compressive strength 31.3% higher in the concrete with ground ash when compared to concrete with fly ash.
- Both the percentage of fly ash and the w/cm ratio affected the average compressive strength of the concretes for 28 and 115 days of curing as well as the slump, although the percentage of fly ash is more determining.

- The factorial design method enabled the calculation of regression models, describing, the compressive strength of the concrete with high contents of fly ash for 28 and 115 days of curing and the slump in function of the Fly ash percentage and w/cm ratio. The outline graph allows us to determine the minimum value of mechanical compressive strength from predefined factors, which can be defined as the project demands.

- The V1 concrete with 60% of fly ash content can be considered as a structural concrete since it presented a mechanical compressive strength of 33.4 MPa at 115 days.

- Concrete V1 presented better physical properties than concrete VP. Concrete V1 also has a lower void index and water absorption, therefore, it is more resistant to the migration of aggressive agents.

- In general, it can be stated that the use of high contents of fly ash in the elaboration of structural concretes is technically feasible and remains an alternative in several types of constructions to reduce the environmental impacts generated by the cement industry.

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