ULTRA-HIGH-PERFORMANCE CONCRETES WITH PHENOLIC FOUNDRY SAND: MECHANICAL AND MICROSTRUCTURAL EVALUATION

CONCRETOS DE ULTRA ALTO DESEMPENHO COM AREIA DE FUNDIÇÃO FENÓLICA: AVALIAÇÃO MECÂNICA E MICROESTRUTURAL

HORMIGONES DE ULTRA ALTO DESENPEÑO CON ARENA DE FUNDICIÓN FENÓLICA: EVALUACIÓN MECÁNICA Y MICROESTRUCTURAL.

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RESUMO

O uso de concretos de ultra alto desempenho (CUAD) tem se tornado cada vez mais comum em diversas soluções de Engenharia civil, principalmente pela sua composição de materiais que conferem propriedades de resistência e durabilidade que ultrapassam significativamente as classes dos concretos convencionais. Neste estudo buscou-se produzir CUAD usando areia fenólica de descarte de fundição (PFS). Foram ensaiadas cinco misturas de concreto. Uma CUAD de referência, chamada de REF, com 85% de cimento Portland branco (WPC) e 15% de sílica ativa (SF), como ligantes, e agregados em uma proporção de 36% de pó de quartzo e 84% de areia industrial. E quatro misturas eco amigáveis com adições minerais: RHA15, com 85% de WPC e 15% de RHA; FA15, com 85% de WPC e 15% de FA; RHA10-FA5, com 85% de WPC, 10% de RHA e 5% de FA; e RHA10-LF5, com 85% de WPC, 10% de RHA e 5% de LF. Com exceção da mistura REF, todas as demais misturas tiveram a adição de 15% de cal, tipo CH I, juntamente com agregados compostos por 36% de pó de quartzo e 84% de areia fenólica (PFS). A relação água/aglomerante adotada foi de 0,20 para todas as misturas. Os concretos foram submetidos a dois tipos de cura: úmida até a idade de ensaio e úmida combinada com cura térmica. Os concretos foram avaliados quanto à resistência à compressão axial e à microestrutura utilizando técnicas de análise termogravimétrica (TG/DTG) e espectroscopia de infravermelho por transformada de Fourier (FTIR). Os resultados mostraram que todas as misturas atingiram níveis de resistência desejados para CUAD (150 Mpa). Até 28 dias, as misturas com RHA apresentaram maior resistência em comparação com as misturas com FA, independentemente do tipo de cura. Contudo, aos 91 dias, as misturas com FA e cura térmica foram mais resistentes que as misturas com RHA. Com os resultados obtidos percebe-se que a PFS pode ser usada como agregado de UHPC nos níveis de substituição testados.

PALAVRAS-CHAVE

CUAD; microestrutura; adições minerais; areia fenólica de fundição.

ABSTRACT

The use of ultra-high-performance concretes (UHPC) has become increasingly common in various civil engineering solutions, mainly due to their composition of materials that provide strength and durability properties significantly surpassing those of conventional concretes. The aim of this study was to produce UHPC with phenolic foundry waste

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sand (PFS). Five mixtures of concrete were tested. One reference UHPC, labeled as REF, with 85% white Portland cement (WPC) and 15% silica fume (SF), as binders, and aggregates in a proportion of 36% quartz powder and 84% industrial sand. And four eco-friendly mixtures with mineral additions: RHA15, with 85% WPC and 15% RHA; FA15, with 85% WPC and 15% FA; RHA10-FA5, with 85% WPC, 10% RHA, and 5% FA; and RHA10-LF5, with 85% WPC, 10% RHA, and 5% LF. Except for the REF mixture, all other mixtures had the addition of 15% of lime, CH I, along with aggregates composed of 36% quartz powder and 84% phenolic sand (PFS). The water/binder ratio adopted was 0.20 for all mixtures. The mixtures underwent two types of curing: moist curing until the test age and moist curing combined with thermal curing. The concretes were evaluated for axial compressive strength and microstructure using thermogravimetric analysis (TG/DTG) and Fourier-transform infrared spectroscopy (FTIR) techniques. The results showed that all mixtures achieved the desired UHPC strength levels (150 MPa). Up to 28 days, the RHA-containing mixtures exhibited higher strength compared to the FA-containing mixtures, regardless of the curing method. However, at 91 days, the FA-containing mixtures with thermal curing were stronger than the RHA-containing ones. With the results obtained, it is clear that PFS can be used as an aggregate of UHPC in the tested substitution levels.

KEYWORDS

UHPC; microstructure; mineral additions; waste foundry sand.

RESUMEN

El uso de concreto de ultra alta resistencia (UCAD) se ha vuelto cada vez más común en diversas soluciones de ingeniería civil, principalmente debido a su composición de materiales que brindan propiedades de resistencia y durabilidad que superan significativamente las clases de concreto convencional. En este estudio, se ha buscado producir CUAD utilizando arena de desecho de fundición fenólica (PFS). Se ensayaron cinco mezclas de hormigón. Un CUAD de referencia, denominado REF, con un 85% de cemento Portland blanco (WPC) y un 15% de humo de silica (SF) como ligantes, y áridos en una proporción de 36% polvo de cuarzo y 84% arena industrial. Y cuatro mezclas eco-amigables con adiciones minerales: RHA15, con 85% WPC y 15% RHA; FA15, con 85% WPC y 15% FA; RHA10-FA5, con 85% WPC, 10% RHA y 5% FA; y RHA10-LF5, con 85% WPC, 10% RHA y 5% LF. A excepción de la mezcla REF, todas las demás mezclas tenían la adición de un 15% de cal, tipo CH I, junto con áridos compuestos por un 36% de polvo de cuarzo y un 84% de arena fenólica (PFS). La relación agua/ligante adoptada fue de 0,20 para todas las mezclas. Los hormigones se sometieron a dos tipos de curado: húmedo hasta la edad de ensayo y húmedo combinado con curado térmico. Se evaluó la resistencia a la compresión axial y la microestructura de los hormigones mediante análisis termogravimétrico (TG/DTG) y técnicas de espectroscopia infrarroja transformada de Fourier (FTIR). Los resultados mostraron que todas las mezclas lograron los niveles de resistencia deseados para CUAD (150 MPa). Hasta los 28 días, las mezclas con RHA mostraron mayor resistencia en comparación con las mezclas con FA, independientemente del tipo de curado. Sin embargo, a los 91 días, las mezclas con FA y curado térmico fueron más resistentes que las mezclas con RHA. Con los resultados obtenidos, queda claro que PFS se puede utilizar como agregado de UHPC en los niveles de sustitución probados.

PALABRAS CLAVE

CUAD; microestructura; adiciones minerales; arena de fundición fenólica.

1. INTRODUCTION

To reduce cement consumption, promoting the reduction of pollutant emissions into the atmosphere and environment sustainability, the use of mineral additions (especially pozzolans and blast furnace slag) in partial replacement of cement in concrete has been an alternative researched and implemented in the construction industry. Among these pozzolans there is rice husk ash (RHA), which is an agro-industrial waste generated by the burning of rice husk, used as an energy source, and fly ash, which is the burning coal waste from the thermoelectric power industry.

Mineral additions promote physical (particle packing), chemical, and synergistic effects for ternary and quaternary mixtures. The physical effect is characterized by the added particles acting as nucleation points for the cement hydration products [1]. The type of mineral addition and mineralogical composition (crystalline and amorphous phases) are responsible for the chemical effect [2]. Isaia *et al*. [3] verified that the combination of pozzolans with different characteristics promotes a synergistic effect, increasing the levels of mechanical strength, i.e., the strength of mixtures with two additions is higher than the one presented by single addition mixtures with the same level of cement replacement.

RHA has reactivity similar to SF, refining the microstructure of the cementitious matrix at early ages, and this reactivity is due to the high amorphous content and its large specific surface area [4]. The RHA and LF mineral additions' composition promotes better performance in chemical and morphological properties, providing higher strength levels, densifying the cementitious matrix [5].

The pozzolanic reaction promoted by FA results in the reduction of Portlandite (CH) [6], for ashes with alumina contents between 15 and 35%, provides the formation of higher contents of hydrated alumina phases [7], and, the great FA influence for usual curing conditions, increases strength at later ages, especially after 28 days.

Limestone filler insertion in the cement matrix in concretes with FA improves the mechanical properties due to the lime interaction with aluminates from cement hydration, forming carboaluminates. The aluminates provided by FA in the pozzolanic reaction process enhance the lime interaction's effect [8].

Ultra high-performance concrete (UHPC) is a special concrete that cannot be compared with conventional or high-performance concrete (HPC). The unit cement consumption is high and, consequently, its cost is very high. However, the achieved mechanical strength and densification of the cementitious matrix are so high that the UHPC can be used in situations where other types of concretes would not be feasible. Thus, the attempt to produce UHPC with industrial by-products is justifiable, for it reduces the cement consumption and the environmental impact, besides adding the aforementioned technical advantages.

In fact, there are studies that seek to substitute part of the Portland cement in the UHPC production, mainly aiming at reducing its cost and environmental impact. Yu *et al*. [9] state that due to the low water/cementitious material ratio (w/cm) and the high cement content, the hydration degree in ultra high-performance concretes is low. Therefore, the substitution of part of the cement, which ends up not reacting, by mineral additions, usually industrial by-products, can be an advantage.

Yazici *et al*. [10] evaluated the behavior of reactive powders UHPC with partial replacement of cement by silica fume combined with blast furnace slag and fly ash, in ternary or quaternary mixtures. The contents of blast furnace slag and ash varied between 10 and 30%, while they tested the silica fume in two possibilities, at a constant content equal to 26% or variable keeping the CaO/SiO2 ratio equal to 1.30. The w/cm ratio remained constant and equal to 0.13. The concretes reached compressive strengths above 200 MPa, with the highest results when the CaO/SiO2 ratio was constant.

Hassan *et al*. [11] evaluated the mechanical strength and stress-strain behavior of UHPC concretes with cement replacement by silica fume and blast furnace slag at 10% and 35% replacement levels, respectively. At 28 days the concretes reached strengths of around 121 MPa.

Le Than *et al*. [12] evaluated the compressive strength of UHPC in quaternary mixtures with cement, silica fume, fly ash, and rice husk ash. The strengths were very close to 130 MPa at 28 days. Van and Ludwig [13] performed a similar study with rice husk ash and blast furnace slag, whose compressive strengths in ternary mixtures were higher than with silica fume in a binary mixture.

Yu *et al*. [9] state that the use of models that maximize particle packing allows producing ultra

high-performance concretes with relatively low cement consumption. The authors achieved compressive strengths of around 100 MPa at 28 days using concretes with partial replacement of Portland cement by 5% silica fume and 29% limestone filler.

Lampropoulos *et al*. [14] evaluated the use of beam reinforcement with ultra high-performance concrete. The UHPC they tested had portland cement partially replaced by 10% silica fume and 35% blast furnace slag. The water/cementitious material ratio was 0.15. The authors achieved compressive strengths of around 164 MPa at 28 days.

On the other hand, foundry sand is the residue generated in the manufacturing process of molds used in the pouring process of ferrous and non-ferrous metals in foundries. When phenolic resins are used as binders, the generated sand is phenolic. One of the environmentally correct alternatives for the use of phenolic sands is its inertization within cementitious matrices.

In this study, it was used silica fume, rice husk ash and fly ash, lime, limestone filler, industrial sand, and waste foundry sand associated with structural white cement to produce ultra high-performance concretes (UHPC). The content of cement replacement by mineral additions was 15%, and, except for the reference mixture, it was added 15% hydrated lime (HL) to all other mixtures to catalyze the pozzolanic reaction. The water/cementitious material ratio (w/cm) was 0.20 and it was evaluated the samples regarding axial compressive strength, thermogravimetry (TG/DTG) and fourier transform spectroscopy (FTIR).

2. MATERIALS AND EXPERIMENTAL PROGRAM

In this study, ternary and quaternary mixtures were cast using white structural Portland cement (WPC), silica fume (SF), hydrated lime (CH I), rice husk ash (RHA), fly ash (FA), and limestone filler (LF), in combination with industrial sand (IS) and discarded phenolic foundry sand (PFS), where the maximum particle packing was evaluated. The tested mixtures included a reference Ultra High-Performance Concrete (UHPC) labeled as REF, with 85% WPC and 15% SF as binders and aggregates in a proportion of 36% quartz powder and 84% industrial sand; RHA15, with 85% WPC and 15% RHA; FA15, with 85% WPC and 15% FA; RHA10-FA5, with 85% WPC, 10% RHA, and 5% FA; and RHA10-LF5, with 85% WPC, 10% RHA, and 5% LF. Except for the REF mixture, all other mixtures had the addition of 15% CH I, along with aggregates composed of 36% quartz powder and 84% phenolic sand (PFS). The water/binder ratio adopted was 0.20.

The curing of the specimens was carried out in two ways: moist curing and moist curing combined with thermal curing, with the first two days in moist curing, followed by five days in thermal curing at 80 °C, and then again in moist curing until the testing age. Subsequently, the specimens were evaluated for axial compressive strength at ages of 7, 28, and 91 days. Therefore, the molding matrix included 5 different types of mixtures, with 2 distinct curing processes, for 3 concrete ages, totaling 30 samples. The evaluated concretes lacked coarse aggregate due to high expected strengths and equipment limitations. Test specimens were limited to 5cm diameter, 10cm height cylinders, making coarse aggregate impractical.

It was used white structural cement, of Portuguese manufacture, with a 28-day strength of 52.5 MPa. According to the literature, this is the most suitable cement for making Reactive Powder Concrete due to its low C3A content and its specific surface area, which is smaller than those of any commercial CP-V, decreasing the water consumption required for a given workability. In partial replacement of cement it was used rice husk ash (RHA) from industries in the region of Santa Maria/RS, burned without temperature control, class E [15], fly ash (FA) from the thermoelectric plant of Candiota/RS, class C [15], hydrated lime (HL) [16], [17], and [18], limestone filler (LF) from the region of Caçapava do Sul/RS, quartz powder and an industrial sand from Analandia/SP, and a waste foundry sand from the city of Santa Rosa/RS. The superplasticizer TecFlow 8000 from GCP Applied Technologies was used.

To compare the mineral additions, it was tested a binary mixture (cement and silica fume), considered the reference mixture (REF), because it is a commonly used composition in the UHPC production, two ternary mixtures (cement, lime, and RHA or FA), and two quaternary mixtures (cement, lime, RHA and FA or LF).

To test the mineral additions in similar physical condition, it was sought to approximate the particle size distributions. Thus, the mineral additions were subjected to grinding in a ball mill with different grinding times, being RHA for two hours, FA for one hour, and LF for three hours. To determine the particle size

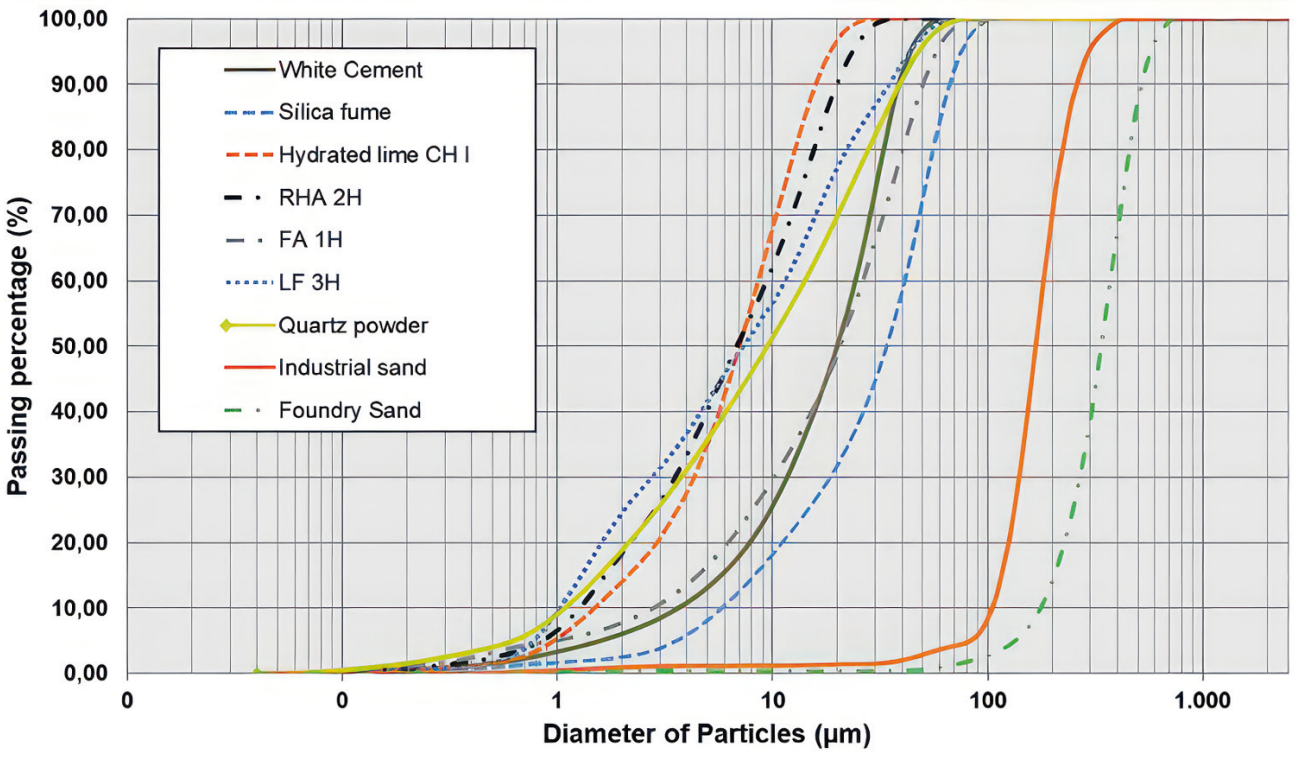

Figure 01: Particle size curves of UHPC component materials.
Source: Authors.

Material	Diameter at 10% (µm)	Diameter at 50% (µm)	Diameter at 90% (µm)	Average diameter (µm)
Cement CP V ARI	1.07	8.90	26.01	11.54
White Portland Cement (WPC)	3.62	19.95	38.72	20.86
Silica fume (SF)	5.76	33.79	66.84	35.16
Hydrated lime CH I	1.48	7.02	15.88	8.02
Rice Husk Ash (RHA) - 2H	1.19	5.92	18.17	7.97
Fly ash (FA) - 1H	2.75	20.60	60.03	23.84
Limestone filler (LF) - 3H	5.84	73.54	310.85	119.01
Quartz powder	1.08	9.45	39.00	15.33
Industrial sand	104.77	167.27	261.85	175.57
Foundry sand (PFS)	175.89	336.76	526.51	346.26

Table 01: Average diameters of materials.
Source: Authors.

curves of the mineral additions, it was used a laser diffraction granulometer, by the PO-GT-1043 method, employing dispersion in anhydrous alcohol and ultrasound for 60 seconds. Figure 01 presents the granulometric curves while Table 01 presents the average diameters of the mineral additions. Of all the materials used, the one with the highest fineness is RHA, with a specific area BET (m^2/g) of 49.25 and an average diameter of 7.97 µm.

Table 02 presents the results of the binders' physical and chemical characterization, with RHA and FA meeting the chemical requirements of ABNT NBR 12653 [15]. The LF milled for 3 hours obtained a performance index of 85%, which is considered satisfactory because it is a fine inert material.

Properties	WPC	CH I	SF	RHA	FA	LF
Physical						
Especific mass (g/cm^3)	3.00	2.30	2.20	2.09	2.36	2.92
Specific area BET (m^2/g)	-	-	1.90	49.25	1.04	2.64
specific surface Blaine (cm^2/g)	4900	7200	-	-	-	-
Residue #0.075 mm (%)	-	< 7.00	-	-	-	-
Residue # 32 µm (%)	2.70	-	-	-	-	-
Start of catch (min)	125	-	-	-	-	-
End of catch (min)	170	-	-	-	-	-
Reflectance index (0 a 100)	86.50	-	-	-	-	-
Portland Cement Performance Index (IAP)	-	-	-	107	92	85
Compressive strength						
3 days (MPa)	33.00					
7 days (MPa)	50.10					
28 days (MPa)	62.00					
Chemical analysis (%)						
Loss to fire	8.85	24.51	-	0.25	0.10	34.44
SiO2	18.75	1.77 + RI	94.30	94.84	68.81	14.18
Al2O3	2.30	0.36	0.09	0.39	23.51	1.54
Fe2O3	0.22	0.16	0.10	2.58	4.70	0.87
CaO	66.50	72.37	0.30	1.32	1.00	28.89
CaO available	-	65.92	-	-	-	-
MgO	0.50	0.39	0.43	0.40	2.16	18.28
SO3	2.95	0.21	-	0.01	-	-
S	-	0.084	-	-	-	-
Na2O	0.06	-	0.27	0.11	-	0.34
K2O	0.24	-	0.83	1.45	0.39	0.39
MnO	-	-	-	-	0.68	-
TiO2	-	-	-	-	0.16	-
CO2	-	4.16	-	-	-	-
P2O5	-	-	-	-	-	-

Table 02: Physical. mechanical and chemical characteristics of binders.
Source: Authors.

Figure 02 illustrates the diffractograms of the RHA, FA, and LF additions. RHA exhibits few crystalline peaks, displaying the amorphism. Crystalline cristobalite peaks were also evident, which indicates burning without temperature control and aeration.

This pozzolan presented a greater amorphism

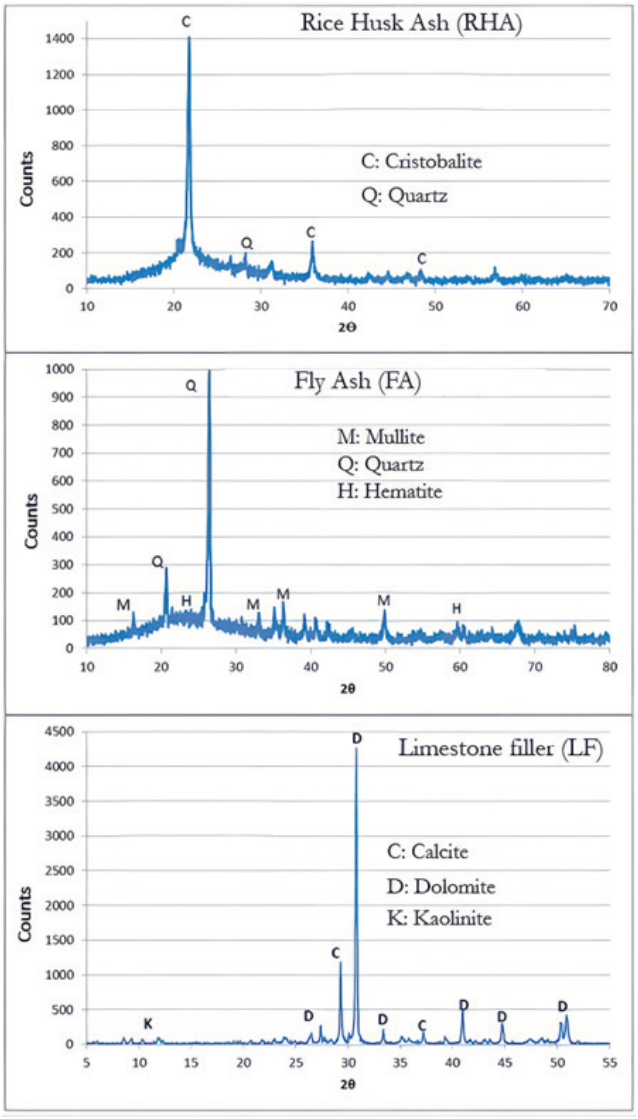

Figure 02: Diffractogram of rice husk ash (RHA), fly ash (FA), limestone filler (LF).
Source: Authors.

halo, especially between angles 2θ, 15 and 30°. The FA presented peaks of quartz (Q), mullite (M), and hematite (H). The LF, on the other hand, presented high dolomite and calcite peaks.

Figure 01 illustrates the particle size distribution of the aggregates and Table 03 presents the physical and chemical characteristics. The average diameters of quartz powder, industrial sand, and foundry sand are 15.33 µm, 175.57 µm, and 346.26 µm, respectively.

2.1. Definition of mixtures, molding, and curing

It was performed the theoretical particle packing of the aggregates and binders. The packing was evaluated using the Elkem Materials Mixture Analyser (EMMA) software, seeking a higher densification of the concrete.

The main input data for EMMA are the particle size curve (obtained by laser granulometry), the diameter at 50%, the maximum and minimum particle sizes, and the materials' specific mass. After including the constituents' data, the mixtures' proportions were informed, with the amount of each material that composes the concrete and the amount of water. For the evaluation of the packing, it is necessary to define the value of the distribution modulus (q), one of the modified Andreassen method's parameters.

The distribution modulus represents the fine (powder) and coarse particles ratio. Hüsken and Brouwers [19] state that values of q lower than 0.25 are ideal to obtain concretes rich in fine particles. Brouwers and Radix [20] consider that a value of q equal to 0.25 is ideal to obtain self-compacting concretes, even when using coarse aggregates. Yu *et al.* [9] used a value of q equal to 0.23 for the production of ultra high-performance concretes employing cement and silica fume as binders, with water/cementitious material ratios ranging from 0.23 to 0.33.

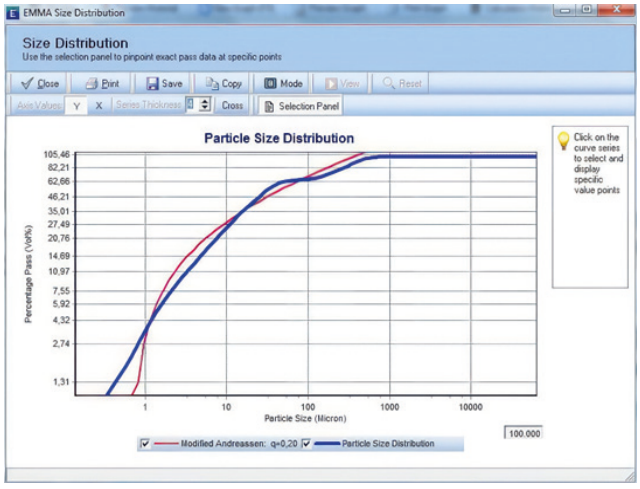

Figure 03: Packaging by Modified Andreassen method for RHA10-FAV5 mixture in EMMA.
Source: Authors.

In this study, it was adopted q equal to 0.20, because the concretes produced were reactive powders and self-compacting. Figure 03 presents the packing of the RHA10-FA5 mixture as an example.

For all mixtures it was set w/cm ratio of 0.20, based on the dosage studies of [21], [22], and [23]. The final strokes were determined from the previous results. Table 04 presents the mixtures in 1:1.2 (cementitious materials, aggregates) mass ratio. This value was adopted based on previous research and preliminary workability and strength tests.

Properties	Quartz powder	Industrial sand	Foundry sand
Physical			
Especific mass (g/cm³)	2.65	2.64	2.60
Unit mass (g/cm³)	0.92	1.48	1.46
#18 = 1.000 mm (%)	0.10	0.00	-
#20 = 0.850 mm (%)	-	0.00	-
#30 = 0.600 mm (%)	7.60	0.00	-
#40 = 0.425 mm (%)	-	0.10	-
#50 = 0.300 mm (%)	-	0.20	-
#70 = 0.212 mm (%)	-	1.50	-
#100=0.150 mm (%)	-	33.70	-
#140 = 0.106 mm (%)	-	59.50	-
#200 = 0.075 mm (%)	-	4.60	-
#270 = 0.053 mm (%)	-	0.50	-
# 325 µm (%)	7.60	-	-
Fineness modulus AFS (American Foundry Society)	-	91.40	-
Clay AFS (American Foundry Society)	-	0.12	-
Fines - sum of the #200. 270 e fundos (%)	-	5.10	-
Humidity (%)	0.10	0.04	-
Temperature (°C)	25.00	26.00	-
Absorption (%)	0.05	-	-
Chemical analysis (%)			
Loss to fire	0.11	0.09	-
SiO2	99.68	99.58	-
Al2O3	0.23	-	-
Fe2O3	0.045	-	-
TiO2	0.028	-	-
pH	-	5.50	-
C	-	-	44.04
O	-	-	42.10
Mg	-	5.50	1.56
Al	-	-	2.09
Si	-	-	5.40
P	-	-	1.25
K	-	-	1.35
Ca	-	-	0.39
Zn	-	-	1.82

Table 03: Physical and chemical characteristics of aggregates.
Source: Authors.

The concretes were molded in a high rotation energy mortar mixer. The chemical admixture used was the superplasticizer TecFlow 8000, with solid contents ranging from 1.23% and 2.14% of the cementitious materials mass. The specimens were subjected to wet curing, until the test age or thermal curing at 80°C beginning. In the latter, after demolding (day after molding), the specimens were immersed in water for 24 hours at room temperature, and from the third to the seventh day, it was subjected to thermal curing. After the thermal treatment, the samples returned to wet curing until the test age.

Material (Mass ratio)	REF	RHA15	FA15	RHA 10-FA5	RHA 10-LF5
White Portland Cement	0.85	0.85	0.85	0.85	0.85
Sílica fume	0.15	-	-	-	-
Hydrated lime CH I	-	0.15	0.15	0.15	0.15
RHA - 2H	-	0.15	-	0.10	0.10
FA - 1H	-	-	0.15	0.05	-
LF - 3H	-	-	-	-	0.05
Sum of cementitious materials	1.00	1.15	1.15	1.15	1.15
Quartz powder	0.36	0.36	0.36	0.36	0.36
Industrial sand	0.84	-	-	-	-
Foundry sand	-	0.84	0.84	0.84	0.84
Sum of aggregates	1.20	1.20	1.20	1.20	1.20
Ratio water/ cementitious materials (w/cm)	0.20	0.20	0.20	0.20	0.20
Superplasticizer – total content (%)	2.51	4.37	3.28	3.64	3.64
Superplasticizer – solids content (%)	1.23	2.14	1.61	1.79	1.79

Table 04: New values entered in EMMA for dosage by the modified Andreassen packing method - unitary, in mass.
Source: Authors.

2.2. Testing

For the concrete axial compressive strength test, it was molded nine (9) 5 x 10 cm cylindrical specimens for each test age, according to the guidelines of ABNT NBR 5738 [24] and ABNT NBR 5739 [25]. The specimens were rectified to ensure the loading surfaces' flatness. The ruptures were performed in an Instron hydraulic press, model HDX 1500, with BlueHill 3.0 data acquisition software. The tests

were performed with displacement control, with a displacement rate of 0.01 mm/s.

For the DT/DTG and FTIR testing, after the specimens broke in the axial compression test, the samples were grinded with mortar and grit hand and sieved them on a #100 sieve (0.15 mm mesh). The procedure for stopping the hydration reactions was by immersing the sample for 15 minutes in isopropanol and then filtering and washing it with diethyl ether [26]. Afterwards, the powder was dried for 10 minutes in an oven at 40 °C and put it in a closed container.

In the DT/DTG test, it was used samples of 15 ± 1 mg conditioned in alumina crucible. The samples were heated in an inert nitrogen atmosphere at a flow rate of 50 mL/min. The test's temperature range was from 20 to 1000 °C, at a temperature rise rate of 20 °C/min.

For the FTIR technique, the infrared region absorption spectrometry was verified to identify the functional groups present in the samples. For each wavenumber (cm-1) there is a corresponding formation of each compound formed. The first mass losses refer to free water, and the temperature levels for this range go up to approximately 105°C. After this temperature, from 105°C up to 400°C, the mass loss corresponds to chemically combined water. This content was obtained through the Bhatty equation [26]. The mass loss from CH occurs between 400°C and 500°C. Carbonates, on the other hand, decompose between 550°C and 990°C. According to Thiery *et al.* [27], the calcium carbonate contents are determined in three ranges. Range I occurs between 780°C < T < 990°C, range II occurs between 680°C < T < 780°C, and range III occurs 550°C < T < 680°C.

3. RESULTS AND DISCUSSIONS

3.1. Axial compressive strength

Table 05 and Figures 04, 05 and 06 present the axial compressive strength results at 7, 28, and 91 days. Table 5 presents the results along with the ANOVA statistical analysis plus the Tukey-Kramer post hoc test. The significance level adopted in the analyses was 5%. The table has indicated the p-value. When the p-value was lower than 0.05, the difference was significant. In pairwise analyses (Tukey-Kramer), when the difference was significant, the calculated p-value was shaded, which indicates that the difference between those mixtures was statistically significant.

In Figures 04, 05 and 06 for each column is presented

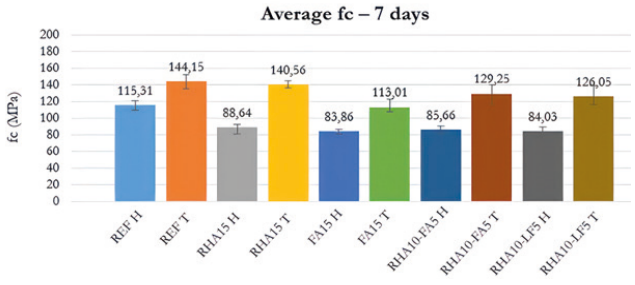

Figure 04: Axial compression strength at 7 days for the studied mixtures.
Source: Authors.

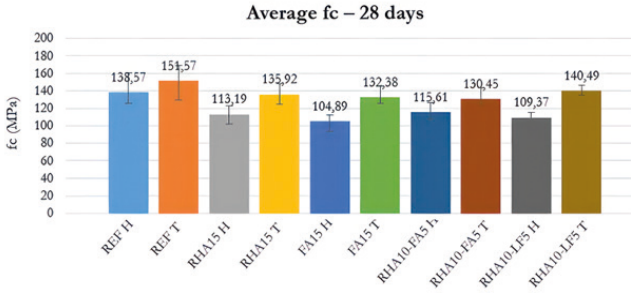

Figure 05: Axial compression strength at 28 days for the studied mixtures.
Source: Authors.

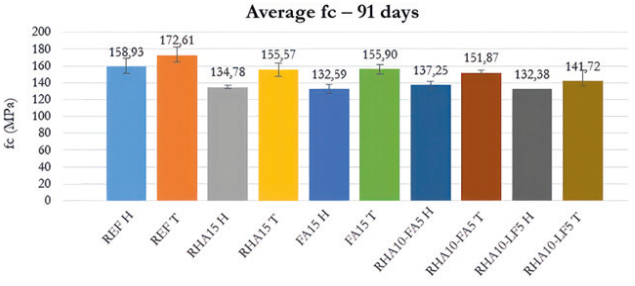

Figure 06: Axial compression strength at 91 days for the studied mixtures.
Source: Authors.

the standard deviation of the slab. In these figures U indicates humid curing and T indicates thermal curing.

It can observe that for both wet and thermal curing, the reference mixture obtained the highest results for all ages. It was evident that at 7 days with wet curing, the strength levels are not consistent with UHPC, the reference mixture reached 115.3 MPa, while the other mixtures did not reach 100 MPa, with the results of mixtures RHA15, FA15, RHA10-FA5, and RHA10-LF5 for wet curing, being statistically equal. Thus, the performance of the studied UHPC is due to a concomitant effect of the fines and the chemical reaction of forming hydrated compounds, the latter being the main one.

The explanation is that, even changing the mineral additions, the strengths were very close for wet curing, that is, the reactivity of each addition was not significant, and the effect of the fineness was preponderant for the performance. As the EMMA software performs the initial dosing process, it evaluates only the physical packing, and

AGE / CURE	MIXTURE						Significance value (ideal 5%)
	MIXTURE	REF	RHA15	FA15	RHA10-FA5	RHA10-LF5	p
7 days – humid cure	REF		0.000	0.000	0.000	0.000	1.06E-10
	RHA15	14.380		0.049	0.252	0.163	
	FA15	20.010	4.261		0.907	0.978	
	RHA10-FA5	18.160	3.000	1.219		0.999	
	RHA10-LF5	18.520	3.370	0.809	0.392		
AGE / CURE	**MIXTURE**	**REF**	**RHA15**	**FA15**	**RHA10-FA5**	**RHA10-LF5**	**p**
7 days – thermal cure	REF		0.952	0.000	0.037	0.028	7.52E-05
	RHA15	1.000		0.001	0.282	0.233	
	FA15	9.507	7.109		0.038	0.050	
	RHA10-FA5	4.548	2.918	4.527		1.000	
	RHA10-LF5	4.757	3.095	4.336	0.191		
AGE / CURE	**MIXTURE**	**REF**	**RHA15**	**FA15**	**RHA10-FA5**	**RHA10-LF5**	**p**
28 days - humid cure	REF		0.001	0.000	0.001	0.000	2.02E-05
	RHA15	6.851		0.558	0.997	0.821	
	FA15	8.616	2.157		0.345	0.976	
	RHA10-FA5	6.673	0.482	2.694		0.588	
	RHA10-LF5	8.664	1.509	0.826	2.088		
AGE / CURE	**MIXTURE**	**REF**	**RHA15**	**FA15**	**RHA10-FA5**	**RHA10-LF5**	**p**
28 days - thermal cure	REF		0.006	0.003	0.001	0.368	5.22E-04
	RHA15	5.522		0.998	0.824	0.316	
	FA15	5.916	0.414		0.933	0.202	
	RHA10-FA5	6.723	1.500	1.105		0.058	
	RHA10-LF5	2.628	2.776	3.171	4.094		
AGE / CURE	**MIXTURE**	**REF**	**RHA15**	**FA15**	**RHA10-FA5**	**RHA10-LF5**	**p**
91 days - Humid cure	REF		0.000	0.000	0.000	0.000	7.08E-07
	RHA15	9.789		0.973	0.959	0.957	
	FA15	11.630	0.851		0.647	1.000	
	RHA10-FA5	9.565	0.958	1.954		0.566	
	RHA10-LF5	12.420	0.970	0.088	2.147		
AGE / CURE	**MIXTURE**	**REF**	**RHA15**	**FA15**	**RHA10-FA5**	**RHA10-LF5**	**p**
91 days - thermal cure	REF		0.001	0.006	0.026	0.000	2.34E-04
	RHA15	6.687		0.776	0.874	0.953	
	FA15	5.668	1.632		1.000	0.301	
	RHA10-FA5	4.740	1.342	0.041		0.493	
		8.149	0.996	2.843	2.318		

Table 05: Statistical analysis ANOVA and the Tukey-Kramer method of the studied mixtures.
Source: Authors.

since the UHPC has many fines, at 7 days many of these materials are still inert in the matrix, not achieving the expected performance.

On the other hand, it was noticed the thermal curing's efficiency at 7 days. The RHA15 mixture presented strength statistically equal to the REF and superior to the FA15, RHA10-FA5, and RHA10-LF5 mixtures. The RHA15 mixture had the highest RHA contents, the thinnest and most reactive addition used, and which was sensitive to thermal curing. In thermal curing at 7 days, the mixture that presented the lowest resistance was FA15. Unlike RHA, this addition is less reactive, due to its chemical and mineralogical composition and its lower fineness. Heat treatment generally results in a reduction of pores in the nanometer range and an increase in compressive strength, compared to the same sample cured under ambient conditions [28].

As expected, at 28 days, all the mixtures presented strength gains when compared to 7 days, with the exception of the thermal RHA15 mixture. Probably, this addition's high reactivity was almost entirely mobilized at early ages, being exhausted, and there was little evolution in the formation of secondary C-S-H at higher ages. As for wet curing, this mixture presented significant growth between the ages of 7 and 28 days. Indicating the mixture's lower reactivity under thermal curing. For wet curing, all mixtures with additions presented significantly lower strengths than the reference, but statistically equal to each other. As for the thermal curing, only the RHA10-LF5 mixture was statistically equal to the reference, the others were lower.

For the 91 days, thermal curing proved to be significantly influential for all mixtures except for the RHA15 mixture. The REF T mixture achieved the highest strength value, reaching 172.6 MPa, while the REF U mixture achieved 158.9 MPa. The other mixtures, for the same type of cure, presented statistically equal strengths among themselves, but lower than the reference. The RHA10-LF5 U mixture presented the lowest average measured strength, 132.38 MPa.

3.2. Thermogravimetric Analysis (TG/DTG)

Figure 07 presents the REF U mixture's TG-DTG graphic at 28 days and the REF U mixture's TG graphic with mass loss, also at 28 days. Figure 08 presents the chemically combined water (CW), CH (calcium hydroxide), and carbonate content at 28 and 91 days. The curves were separated from the test into temperature ranges. Up to 105°C free water loss occurred, between 105°C and 300°C,

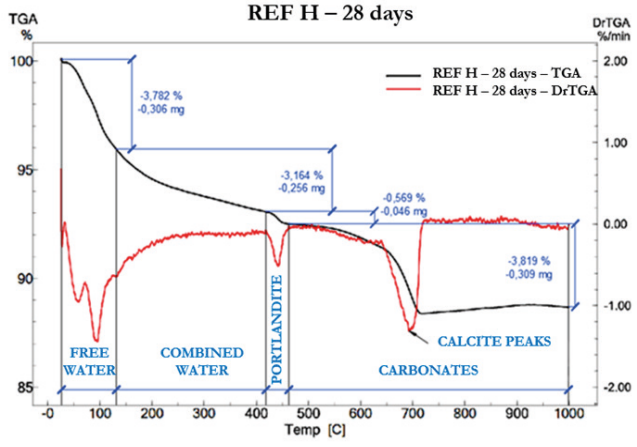

Figure 07: Axial compression strength at 91 days for the studied mixtures.
Source: Authors.

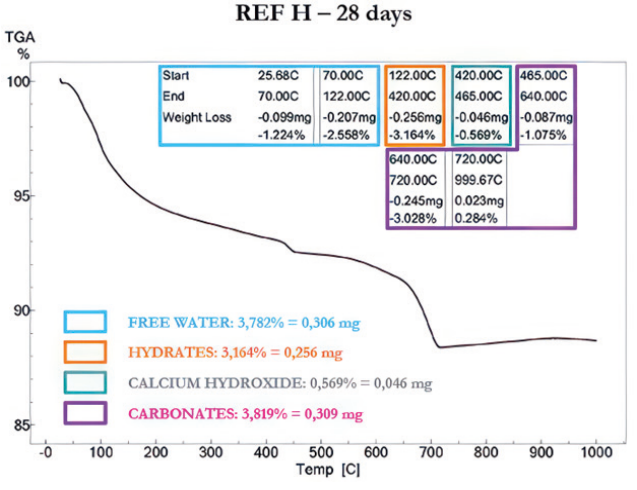

Figure 08: TG graph with mass loss of the REF U mixture at 28 days.
Source: Authors.

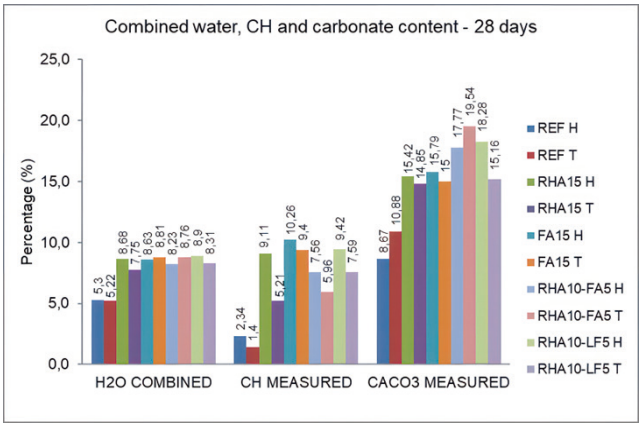

Figure 09: Chemically combined water, CH and carbonate content at 28 days.
Source: Authors.

chemically combined water loss from the decomposition of C-S-H and hydrated carboaluminates occurred [29]. Between 400°C and 500°C mass loss from the portlandite (Ca(OH)2) occurred. For temperatures above 600°C the mass loss corresponded to calcite. It can be seen in Figure 09 that at 28 days the lowest chemically combined water (CW) levels were for the REF mixtures. The pozzolanic

reaction increases CW levels, tending to consume calcium hydroxide. The reason that the REF mixture presented lower CW levels is that it has no lime addition, and it is the only mixture where the SF was used. The aim was to make a comparison of a usual UHPC, which contains SF, with the other proposed mixtures using lime, RHA, FA, LF, and foundry sand. So, in these mixtures some of the CH was not consumed by the pozzolanic reaction.

This was visible in the higher CW contents for the mixtures with mineral additions, this behavior being evidenced by the remaining CH content.

Lime was added to the mixtures, with the exception of REF, to react with the pozzolans, but due to the low w/cm ratio and the large content of fine materials, part of the cementitious materials did not hydrate, functioning as filler. The failure to completely hydrate cementitious materials was observed by [30]. These authors evaluated the degree of hydration achieved in ultra high-performance concretes with 20% partial replacement of cement by silica fume, quartz sand as aggregate and w/cm ratio of 0.14. They concluded that the degree of hydration achieved was only 31%. According to [31], complete cement hydration is only possible for w/cm ratios greater than 0.42. In concretes with mineral additions, where there is enough water for the pozzolan to react with the calcium hydroxide, there is a tendency to reduce the levels of CH in the cementitious matrix. However, in this study, a significant portion of the supplied lime did not react with the pozzolans.

It can be seen that there was no significant difference in the CW of the REF mixture's wet and thermal cures. For the mixtures with mineral additions, all presented higher CW levels when compared to the REF mixture. RHA, due to its porous structure, can absorb free water inside the molecule during mixing [32], and at later ages, when the paste's relative humidity reduces due to cement hydration, it slowly releases this water, causing pozzolanic reactions to occur at later ages.

The CW content is indicative of the amount of hydrated compounds formed, the main ones being C-S-H and CH. One hypothesis is that lime would providing CH to react with the pozzolans and refine the concrete's microstructure, improving the mechanical properties. However, as already mentioned, this hypothesis was partially verified in the results. In this way, part of the CH was not consumed by the pozzolanic reaction. This was visible in the CW contents for the mixtures with mineral additions, this behavior being evidenced by the remaining CH content. The studied UHPC's performance is due

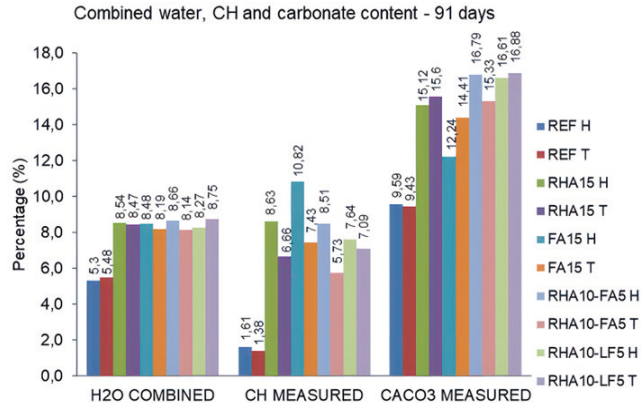

Figure 10: Chemically combined water, CH and carbonate content at 91 days.
Source: Authors.

to a concomitant effect of the fines and the forming chemical reaction of hydrated compounds.

Regarding the CHMEASURED content at 28 days, all mixtures with admixtures presented higher contents than the REF mixture. This behavior is in line with that observed for chemically combined water, where it was found the highest CW content for the mixtures with mineral additions. The highlight in CH levels was for the FA15 U mixture, with 10.82%. It is noteworthy that the FA15 mixture features 15% FA as a cement substitute and 15% lime addition. When compared to the RHA15 mixture, in which RHA was used instead of FA, it was found that RHA consumed more CH due to its greater fineness and reactivity. Moreover, the thermal treatment accelerated the pozzolanic reactions, consuming CH more intensely when compared to wet curing.

When the carbonates at 28 days were evaluated, the lowest contents were for the REF mixture, similarly to CW and CH. In contrast, the highest levels were for the RHA10-FA5 mixture, which combined FA and RHA. It is worth noting that there was no significant difference for the mixtures with mineral additions, the variation being 14.85% (RHA15 T) and 19.54% (RHA10-FA5 U). The explanation for this similar behavior is that the lime insertion would react with the pozzolans to refine the microstructure. However, this occurred in part due to the low w/cm ratio and the reduced space for the pozzolanic reaction to occur. Then a portion of the additions became inert in the mixture, evidencing the similar behavior for the carbonates.

For the 91 days it was observed the same behavior trend for the 28 days. For the REF mixture, the levels were lower than the other mixtures, with CW content of 5.30% for REF U and 5.48% for REF T. The mixtures with mineral additions presented very close CW contents, which is an indication that the physical effect was

predominant. Regarding CH, the highest content was in the FA15 U blend, with 10.82%. For carbonates, the FA15 T mixture presented the greatest increase from 28 to 91 days. It is noteworthy that this increase was similar for mixtures with mineral additions, due to the low w/cm ratio and the reduced space for pozzolanic reactions.

3.3. Fourier Transform Infrared (FTIR)

Figures 11 and 12 illustrate the FTIR spectra's results for the mixtures studied at 28 and 91 days, respectively. Between 400 and 600 cm-1, C-S-H has a maximum at 450 cm-1, the position for the main band of tobermorites is 490 cm-1 and internal deformations of the SiO4 tetrahedra occurred. At 450 cm-1 the rocking mode occurs to determine the Si-O-Si incorporation rate according to [33] and [34].

Among the peaks it was observed, at 670 cm-1 the C-S-H gel change occurred. This change is due to exposure to high sodium concentrations [34] and it is likely to have occurred due to the sample's dilution by precipitated CaCO3. Since foundry sand was used in this study, which has significant sodium and potassium contents, the behavior is within the expected range. The peaks at 694 and 778 cm-1 correspond to elongated vibrations of SiO4, compatible with quartz [35]. Between 714 and 874 cm-1 the decomposition of carbonates occurs, indicating the reaction between Ca(OH)2 and CO2, and the vibration of CO2 from CaCO3 also occurs [36]. For the peaks between 870 and 1420 cm-1, the bands are characteristic of the O-C-O bonds of carbonates [37], [38]. It is noteworthy that these bands are due to the asymmetric stretching of CO32- [39].

The peaks between 970 and 1004 cm-1 depict the asymmetric stretching vibration of Si-O-Si and Al-O-Si, and these elements are bound to silicates (quartz, amorphous silica, and glassy phases) and aluminosilicates (mullite) [44, 45]. There is an association of these peaks associated with C-S-H gel [40], which present higher levels at 91 days compared to 28 days. This behavior is due to the stretching vibrations of the SiO4 tetrahedron's SiA- in the C-S-H gel.

For all the mixtures studied, it was observed that at 91 days the curves are modified more intensely compared to 28 days, with special emphasis on the RHA15 mixture (thermal curing). It is worth noting that this mixture presents 15% RHA, associated with the lime present, also in the 15% content, with the temperature rise provided the pozzolanic reaction generating secondary hydrated compounds.

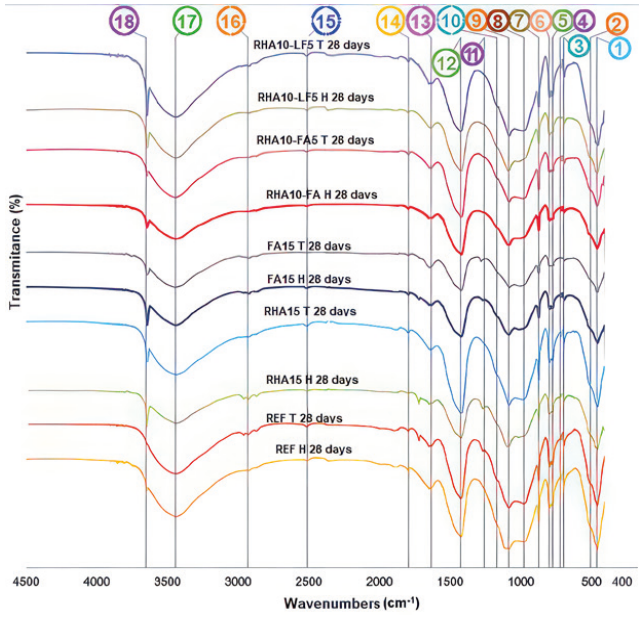

Figure 11: FTIR spectra for the studied mixtures at 28 days.
Source: Authors.

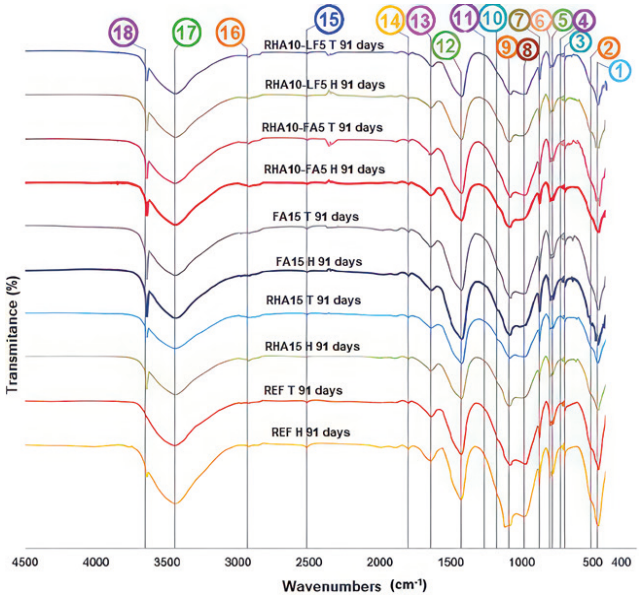

Figure 12: FTIR spectra for the studied mixtures at 91 days.
Source: Authors.

Between 1200 and 1700 cm-1, the vibrations refer to the out-of-plane deformation (n2) of the sulfates' and/or ettringite's O-H vibration and the bands of the CaCO3 added to the cement. In the range of 1300 and 1600 cm-1 there is an increase in the intensity of the bands referring to O-H bound to sulfates and ettringite [41]. Between 1400 and 1450 cm-1 occur C-O stretching vibrations that we can attribute to calcium carbonates [34]. It can be seen that the mixtures FA15 U and FA15 T were the ones that presented the most relevant peaks of CaCO3, with this sample presenting 15% of FA in substitution for cement and 15% of lime in addition to the system.

The presence of chemically combined water occurs in

the peaks at 1640 and 3445 cm-1 [35], [39], and [42]. The peaks at 3465 cm-1 correspond to CW [43]. All the mixtures in this study presented characteristic CW peaks, being more significant for the thermally cured mixtures.

The peaks at 1700 and 1795 cm-1 are compatible with C-O vibrations in Ca(CO)3 [44]. The calcite peaks, on the other hand, are quite characteristic at 2514 cm-1 [45]. The C-O vibrations in CO2, restricted in the amorphous phase, occur in peaks at 2920 cm-1 [42].

The peaks at 3715 cm-1 represent the OH group's stretching and deformation vibration [46]. The stretching vibrations generated by the O-H bonds in the portlandite (Ca(OH)2), due to the Ca/Si ratio in the gel, causes a certain portion of portlandite to precipitate with the C-S-H gel [22], the same behavior found by [37] and [42].

In this study it was observed that at both 28 and 91 days, the REF mixtures presented low portlandite levels. This performance ratifies the results obtained in the DRX and TG/DTG analyses. The REF mixture has the lowest portlandite content when compared to the others, due to the absence of lime addition in the mixture, in such a way that practically all the CH produced by cement hydration was consumed by the pozzolanic reaction resulting from the silica fume presence. For all mixtures with added lime, there are portlandite peaks, which indicates that the lime did not fully react with the pozzolan. At 28 days, the mixture that presented the most pronounced portlandite peaks was RHA10-FA5 T (thermal curing), however this peak reduced at 91 days, proving that there was a pozzolanic reaction. At 91 days, the most intense portlandite peak was for the FA15 U mixture (wet curing).

4. CONCLUSIONS

It was evaluated the compressive strength and microstructure of the UHPC samples with mineral additions of the RHA, FA, LF, and lime. The microstructure was evaluated with thermogravimetry (TG/DTG) and Fourier transform infrared (FTIR) techniques.

It was possible to develop eco-friendly ultra high-performance concrete using mineral additions and phenolic foundry sand, reaching strength levels compatible with this type of concrete.

All mixtures with mineral additions presented inferior performance in axial compressive strength, with the RHA15 T (thermal curing) mixture reaching levels very close to the REF, being superior to the FA15, RHA10-FA5, and RHA10-LF5 mixtures. The RHA, with the temperature elevation to 80ºC, promoted a significant increase in strength through pozzolanic reactions. At 91 days there was a tendency for the strengths for wet and thermal curing to approximate.

The use of pozzolans with different reactivity (FA: less reactive), (RHA: more reactive), and (LF: inert), promoted the synergistic effect due to the chemical interaction, making the secondary C-S-H levels high, improving as a whole the concrete's microstructure.

Regarding TG-DTG, the mixture that presented the greatest evolution in CW and CH levels was the RHA15 T mixture. The lime insertion to provide CH for the pozzolanic reaction was partially effective. There was an increase in CW levels, promoting the formation of secondary hydrated compounds, especially for the heat-treated samples.

Regarding the FTIR, for all mixtures studied, it was observed that at 91 days the curves are modified more intensely compared to 28 days, with emphasis on the RHA15 T mixture (thermal curing), highlighting the mixture with 15% RHA, which associated with the provided lime, also at 15%, with the temperature rise, provided the pozzolanic reaction generating secondary hydrated compounds.

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