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ORIGINAL ARTICLE

Effect of combined fiber-microcrystalline cellulose reinforcement on the rheology and hydration kinetics of cementitious composites

Efeito do reforço combinado de fibra de celulose-microcelulose cristalina no comportamento reológico e na cinética de hidratação de compósitos cimentícios

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Abstract: The influence of the combined addition of cellulose fibers (FC) and microcrystalline celluloses (MCC) on the fresh properties and hydration kinetics of cementitious composites was investigated. For this purpose, sixteen different formulations of FC-MCC celluloses in the cement matrix were analyzed, in which various cellulose fibers were 0.5%, 1.0%, and 1.5% and microcrystalline cellulose in 0.4%, 0.6% and 0.8% about the cement mass. The cementitious composites with the addition of FC-MCC celluloses were characterized in terms of rheological behavior, which was determined through the *Squeeze flow* method, fluidity through the *mini-slump* test, and hydration kinetics determined through the temporal evolution of the temperature of the mixtures. The initial hydration tests showed that the maximum addition of MCC (0.8%) used in this work reduced the maximum temperature of the cementitious composites, as well as the combination of FCs with MCC 0.8. Cellulose fibers took a longer time to reach the maximum temperature. The combined contents of FC 1.0-MCC 0.4 and FC 0.5-MCC 0.6 promoted an increase in the maximum temperature, which could indicate a dispersive effect of the cellulose particles with the cementitious system. The results of the compression flow showed that the studied samples presented a flow with very low loads and extended for a large part of the curve. Te increase in the amount of cellulose fiber alters the main phenomena related to flow: with a high cellulose content (FC 1.5%) there is an increase in friction between the particles, leading to the conclusion that the amount of cellulose fibers in the cementitious system influences on the rheological behavior and the occurrence of phase separation.

Keywords: nanomaterials, *squeeze flow*, cement system, phase separation.

Resumo: Foi investigado a influência da adição combinada de fibras de celulose (FC) e microceluloses cristalinas (MCC) nas propriedades no estado fresco e cinética de hidratação dos compósitos cimentícios. Para isso, foram analisadas dezesseis diferentes formulações de celuloses FC-MCC na matriz cimentícia, no qual foram variadas fibras de celulose em 0.5%, 1.0% e 1.5% e microceluloses cristalinas em 0.4%, 0.6% e 0.8% em relação a massa do cimento. Os compósitos cimentícios com adição das celuloses FC-MCC foram caracterizados em termos de comportamento reológico, no qual foi determinado por meio do método *Squeeze flow*, de fluidez através do ensaio de *mini-slump* e de cinética de hidratação determinada por meio da evolução temporal da temperatura das misturas. Os testes de hidratação inicial mostraram que a adição máxima de MCC (0.8%) empregada nesse trabalho reduziu a temperatura máxima dos compósitos cimentícios, assim como a combinação das FC's com a MCC 0.8. As fibras de celulose levaram um tempo maior para atingir a temperatura máxima. Os teores combinados de FC 1.0-MCC 0.4 e FC 0.5-MCC 0.6 promoveram um aumento da temperatura máxima podendo indicar um efeito dispersivo das partículas de celulose com o sistema cimentício. Os resultados do fluxo de compressão mostraram que as amostras estudadas apresentaram um fluxo com cargas muito baixas e se estendendo por grande parte da curva. O aumento da quantidade de fibra de celulose altera os principais fenômenos relacionados ao fluxo: com alto teor de celulose (FC 1.5%) há um aumento do atrito entre as partículas, podendo concluir que a quantidade de fibras de celulose no sistema cimentício influencia no comportamento reológico e na ocorrência de separação de fases.

Palavras-chave: nanomateriais, *squeeze flow*, sistema cimentício, separação de fases.

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1 INTRODUCTION

Studies on cellulose have been of great importance. It is the most abundant polymer in the world, with an estimated production of 10^{14} tons per year, and can be found in different forms of life such as green plants, fungi, protozoa, and prokaryotes [1]. In the wood industry, cellulose-based materials are generally used in paper production as a possible emulsifier and dispersing agent, which can minimize the carbon footprint, generating interest in biodegradable materials [2], [3]. Although it is a natural product, it has the potential to replace synthetic polymers, since they are non-petroleum-derived materials and can also be used to produce micromaterials [4], [5].

There are different types of processing (mechanical defibrillation; enzymatic treatment; intensive hydrolysis chemical treatment) to isolate cellulose particles, to produce different materials, such as, for example, fibrillated nanocelluloses (CNF), crystalline nanocelluloses (CNC) and crystalline microcelluloses (MCC) [6], [7].

The addition of micromaterials and nanomaterials in fragile matrices can be considered relevant, as it aims to increase particle packing and improve properties [8]–[10]. Cellulose microparticles can increase chemical interactions with matrices based on mineral binders, acting as a rigid particle in suspensions within composites, and altering the rheological behavior of cementitious systems [1], [10], [11].

For cementitious binders, it was demonstrated that additions of cellulose particles have a great impact on the rheological properties, even with low levels of micro or nanoparticles, being more efficient when compared to cellulose fibers [11]–[13]. In low doses (0.2%), cellulose particles can be used, interacting with cement particles in a similar way to water-reducing additives, improving the dispersion of composites. At high dosage levels (0.5%) cellulose particles can be used as a viscosity modifier in mixtures [14].

Another cellulose-based product, cellulosic fibers, has been used as reinforcement in cementitious composites since ancient times and has been growing in fragile building material. This is due to its numerous advantages in rheological behavior, such as the internal curing process and control of cracks in shrinkage through the water retention mechanism of cellulosic fibers [1], [10], [15], [16].

One of the limiting factors in the use of cellulose fibers in cementitious matrices is the significant variations in chemical composition, diameter, length, surface roughness, and amount of cellulose fibers, resulting in difficulties in mixing and dispersion, as well as a significant reduction in plasticity, of fresh composites [16], [17].

The evaluation of fresh properties in materials based on mineral binders is extremely important, as cementitious components play a crucial role in the civil construction market. They can be produced by different methods that require different rheological characteristics. A proposed method to measure the rheological behavior of cementitious composites is the *squeeze flow*, a rheometric technique based on compressing samples in compression-tension presses. The results are analyzed in terms of the flow behavior, assuming that fresh cement matrices behave like a non-Newtonian liquid [18]–[20].

The study of the interaction between the matrix based on mineral agglomerates and cellulosic materials is of great importance to be used in civil construction. Its application depends on the determination of chemical compatibility, assessed through the temporal evolution of temperature in the mixture. The incorporation of cellulose-based materials in the cement mass can affect the thermal balance of the composite and the intensity of hydration reactions, which is why the maximum temperature of the composite hydration reaction is used as an indicator of compatibility [21].

The compounds formed by binders and cellulose fibers can reduce induced cracking due to less heat generation during hydration [22], [23]. On the other hand, cellulose microparticles, have reactive surfaces, presenting a different behavior from cellulose fibers, and may offer new possibilities in cementitious matrices, as a result, they have a potential to increase resistance, and cellulose microparticles generally increase the degree of hydration of the cement [24], [25].

Thus, given the aforementioned context and the relevance of the theme, the present work aims to evaluate the combined effect of cellulose fiber and crystalline microcellulose, on the evolution of the properties of cementitious systems in terms of fresh properties and hydration kinetics. The goal is to development of formulations that meet the desired performance for cementitious building materials.

2 MATERIALS AND METHODS

2.1 Materials

2.1.1 Cellulose fiber (FC)

Cellulose fibers (Figure 1a) were obtained by a commercial company and were chemically characterized through X-ray diffraction (Figure 1b). Cellulose microfiber was obtained by reprocessing industrial paper waste. Cellulose fiber was obtained by reprocessing industrial paper waste. The material was ground, separated by aero separators, passed through magnetic and heavy particle filters, and moistened to form a pulp. Afterward, it underwent washing, sieving, and drying in an oven. The resulting material was further processed through separators and then ground to a size of 400 micrometers. Finally, calcium carbonate was added to facilitate the mixing of the fiber when applying cementitious materials.

The test was carried out in a diffractometer using CuKα1 (λ = 1,54056 Å), in the range of 10 to 70°, with a step size of 0.015° and counting time of 100s every 1.05°. According to the manufacturer's data, the apparent density is 1.2 g/cm^3 and the average particle diameter is 400 μm.

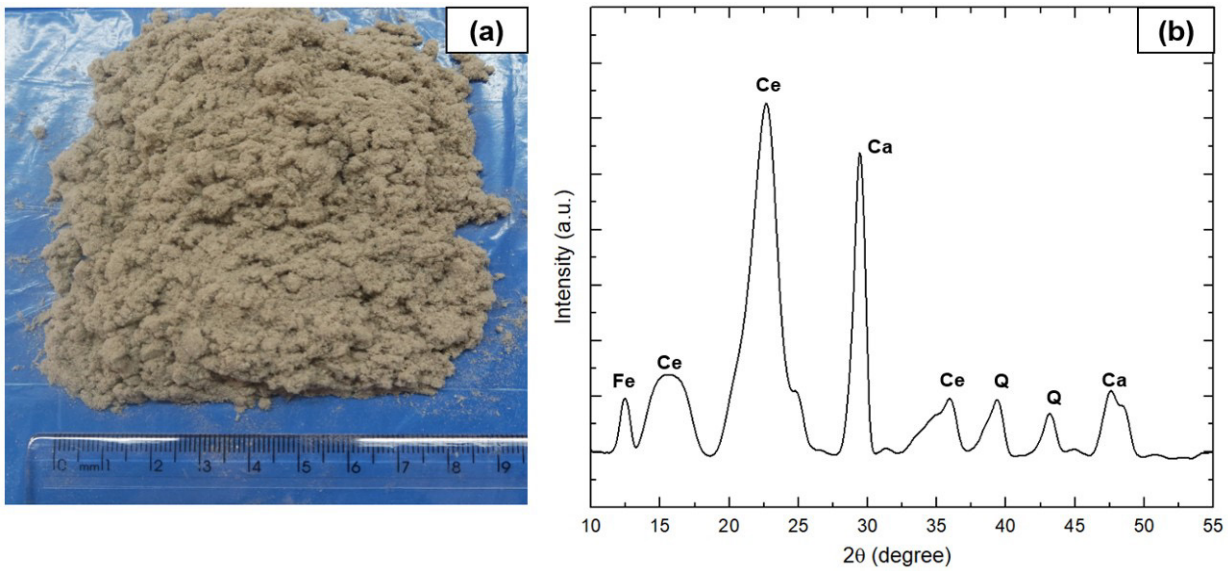

Figure 1. Samples of cellulose fiber (a) and XRD of cellulose fiber (b). Note: Fe = ferrite, Ce = Cellulose, Q = Quartz, Ca = Calcium carbonate.

XRD analysis of the cellulose fibers identified the three peaks of crystalline cellulose (2 θ = 15; 22; 35). In addition to cellulose (Ce), quartz (Q), calcium carbonate (Ca), and tetracalcium ferroaluminate (Fe) were found, components that are not found in native cellulose fibers.

To evaluate the inorganic components of the cellulose fiber, the ash content was determined according to TAPPI standard T211 om-02 [26]. The ash content of the cellulose fiber obtained was 16,49%.

Migneault et al. [27] evaluated the ash content as a measure to determine the degree of contamination of natural cellulose fibers extracted from different types of wood. A content of less than 1% ash was found, being typical of clean wood and due to the minerals present in cell walls and extractives. The wood barks presented ash contents in the range of 1% to 5%. Ash contents of 15% to 50% are found in lignocellulosic materials that have undergone chemical treatments.

To identify the chemical components of the ashes of the cellulose fibers, a semi-qualitative chemical analysis was carried out by X-ray fluorescence spectrometry. Ashes were prepared at 700°C. Table 1 shows the results of the chemical analysis of the cellulose fiber ash.

Table 1. Chemical composition semi-quantitative of cellulose fiber ash

	CaO (%)	SiO_2 (%)	Al_2O_3 (%)	MgO (%)	TiO_2 (%)	Fe_2O_3 (%)	Na_2O (%)	K_2O (%)
Cellulose fiber ash	49.2	22.9	13.6	1.8	1.2	1.1	1.0	0.9
	SO_3 (%)	Cl (%)	P_2O_5 (%)	SrO (%)	CuO (%)	ZnO (%)	ZrO_2 (%)	LOI (%)
	0.7	0.2	0.2	0.1	0.1	< 0.1	< 0.1	7.01

With the chemical analysis by X-ray fluorescence, in which the results are expressed in percentage oxides, it is observed that the ashes of the cellulose fibers are mainly composed of calcium oxide, silica and alumina (85.7%).

The high levels of silica (22.9%). alumina (13.6%) and calcium oxide (49.2%) found in the ash of cellulose fibers are not commonly found in wood ash. The chemical compositions of natural cellulose fibers and cement are different and the interaction between them is generally complex, causing this interaction to cause incompatibility along the interface of cementitious systems [28]. Treatments to modify cellulose fibers are used to optimize an improvement in the interface between fibers and cementitious systems [29], Biswas et al. [30] modified the surface of the cellulose fiber with calcium silicate and found that there are large amounts of Ca and Si bonds in the cellulosic hydroxyls. Demonstrating affinity between the molecules, Li et al. [31] produced nanocomposites based on calcium silicate and nanofibrillated cellulose and verified high interaction with the cementitious matrix.

2.1.2 Microcrystalline cellulose (MCC)

Microcrystalline cellulose, as shown in Figure 2a, was also purchased through a commercial company. The chemical characterization of MCC's was performed using XRD (Figure 2b). Crystalline microcellulose was obtained from purified cellulose fiber subjected to acid hydrolysis under controlled conditions. In the first phase, the cellulose fiber pulp was treated with a dilute acid solution. During hydrolysis, the acid molecules acted on the amorphous regions and broke the β bonds. The resulting water-soluble cellulose oligosaccharides and glucoses were removed in the next process. Subsequently, the pulp was washed, filtered, or ground to obtain the particle.

According to the manufacturer's data, the apparent density is 0.40 g/cm^3 and the average particle diameter is 150 nm.

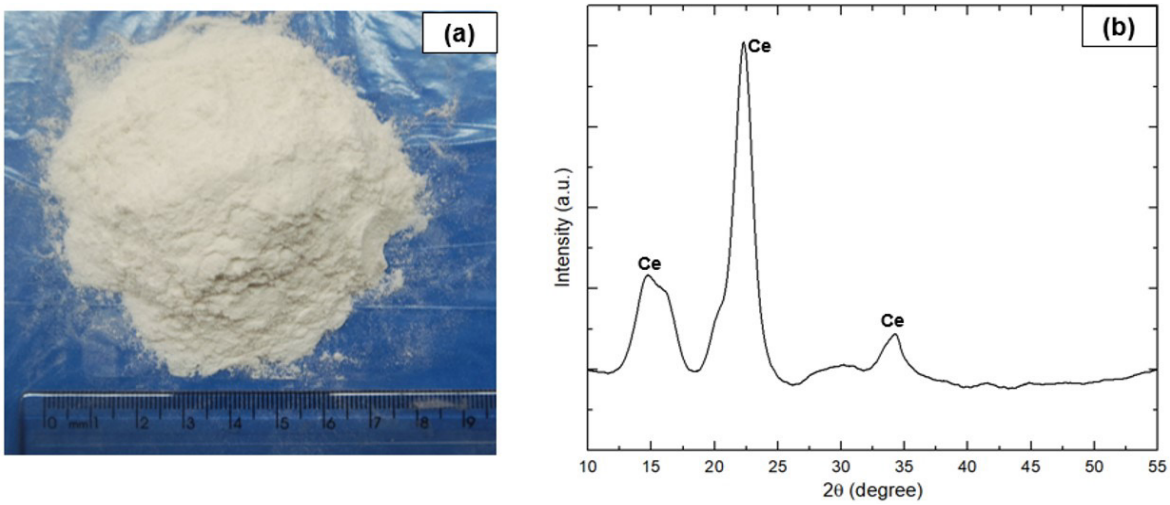

Figure 2. Samples of microcrystalline cellulose (a) and XRD of microcrystalline cellulose (b). Note: Ce = Cellulose.

Through XRD analysis, it was possible to identify that the samples peaked around 2θ = 14.5; 22; 34. These assignments are characteristic of the Miller indices, respectively, attributable to the crystalline cellulose I component. Microcrystalline celluloses present a typical diffractogram of cellulose I, with peaks in the amorphous region (18° ≤ 2θ ≤ 19°) and a maximum peak in the crystalline region (22° ≤ 2θ ≤ 23°) [32].

2.1.3 Cement Portland

The type of Portland cement used in the research is CP V ARI, justified by the fact that it does not present physical and chemical interference from mineral additions and thus does not influence the performance results of FC-MCC pulps in the production of composites. The oxide content and chemical characterization are shown in Table 2.

Table 2. Ordinary Portland cement oxide contents and chemical description

Components	Percent by mass (%)
SiO_2	19.12
Al_2O_3	4.51
Fe_2O_3	2.88
CaO	62.72
MgO	2.82
SO_3	2.62
Loss on Ignition	3.31
Free Lime	1.43
Insoluble Residue	0.67
Equivalent Alkali (NaEq%)	0.73
Blaine Fineness (cm^2/g)	4.302

2.2 Methods

2.2.1 Specific surface of the materials used

The characterization of cement particles. cellulose fibers and microcrystalline cellulose were determined through the specific area by the BET test (Brunauer-Emett-Teller), for joint analysis with the behavior of the fresh state of cementitious composites.

In the BET test, the gas used in the adsorption was nitrogen and the tests were carried out at a temperature of 77K. The sample was treated in a vacuum (100 mm of Hg) and heated at a temperature of 200°C for at least 8 hours for degassing and elimination of possible surface contaminants.

Suspensions containing particles with a high surface area are more susceptible to surface phenomena that may cause agglomeration between the particles. Consequently, increases the viscosity of the mixture, on the other hand, may increase the hydration kinetics [33]. The results of the specific surface are shown in Table 3.

Table 3. Results of the surface area (BET) of the materials used in the research

Materials	Specific surface (m^2/g)
Cement	3.567
Cellulose fiber	3.288
Cellulose Microcrystalline	4.554

Microcrystalline cellulose has a higher surface area compared to cement and cellulose fiber. The high specific surface of cellulose microparticles promotes a strong interface with cement hydration products [23], [34]. Fine particles tend to become dense packing, desirable in obtaining cementious systems. However, the excessive insertion of fines can lead to a high increase in the surface area and, consequently, a greater amount of water needed to cover the particles, which would reduce the free water for their removal, reducing the fluidity of the system [35], [36].

2.2.2 Preparation of aqueous MCC suspensions

The microcrystalline cellulose (MCC) dispersion process in the cementitious system was based on the work of Alshaghel et al. [37]; Parveen et al. [38]; Silva et al. [39]; Gómez Hoyos et al. [22]; Lisboa et al. [40]. The present work, however, focused on the development of a simple and less intensive physical technique to achieve homogeneous dispersion of MCC. The microcrystalline celluloses (0.4%; 0.6% and 0.8%) were added to the mixture water, then the

solution was stored for 24 hours for microparticle hydration. Subsequently, they were manually stirred for 5 minutes and the aqueous solution was added immediately to the dry materials in the mortar. Figure 3 shows the different aqueous solutions: (a) water/MCC (0.4), (b) water/MCC (0.6), and (c) water/MCC (0.8), respectively, as well as the analysis of the dispersion through optical microscopy that were used in the research.

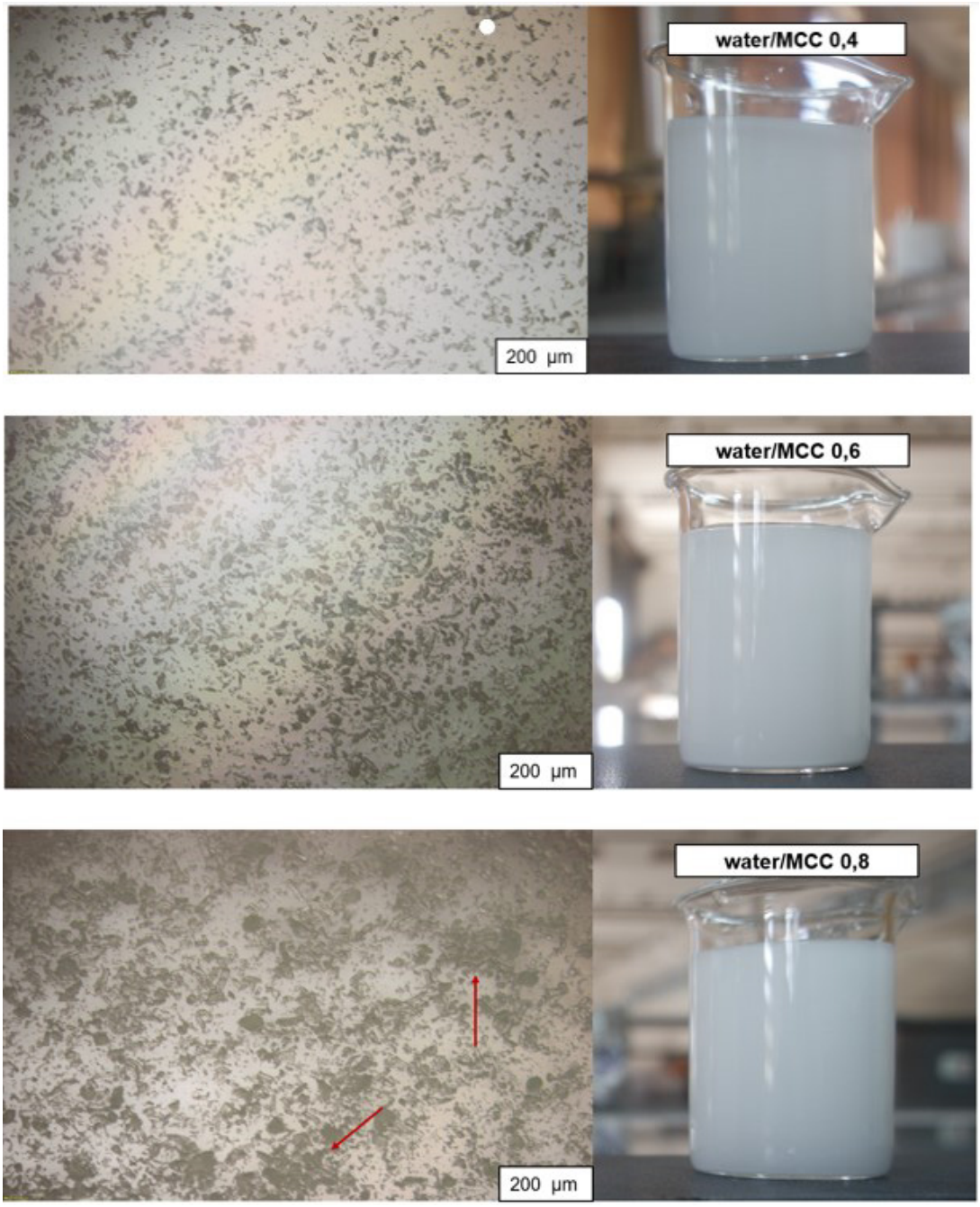

Figure 3. Aqueous suspension of MCC and Optical micrographs of MCC suspensions: (a) 0.4% MCC. (b) 0.6% MCC. (c) 0.8% MCC.

Through the optical microscopy test, it was possible to evaluate that the samples with water/MCC (0.4) and water/MCC (0.6) are well dispersed and homogeneous, with a slight increase in agglomeration at a higher concentration (water/MCC (0.8)).

2.2.3 Cement composites elaboration

The mixing procedure for the cementitious composites developed was based on the Brazilian standard NBR 16541 [41]. The standardization in the mixing procedure was extremely important so that the cellulose fibers were homogeneously dispersed, avoiding any type of agglomeration between them, and disturbance of the results obtained.

The dry materials (cement + cellulose fiber) were mixed inside plastic bags until complete homogenization. After the dry materials (cement + cellulose fiber) and the aqueous solution (water + microcrystalline cellulose) were completely homogenized, the dry materials were added to the mortar. Then, the aqueous solution was gradually added to the mixture, being homogenized for 60 seconds. In the final step, the equipment was turned off so that the bowl could be scraped with the aid of a spatula and, finally, another 60 seconds of mixing at a slow speed.

2.2.4 Mix proportions

To meet the objective of this research. an experimental program was implemented in pulp to correlate the effect of two types of cellulose (FC and MCC) combined. Table 4 shows the layout of the addition systems in cementitious pastes.

Table 4. Mix design showing the quantity of different components used for the preparation of cementitious composites

Samples	Cement (g)	Water Ratio	FC (g)	MCC (g)
Reference	100	0.45	-	-
FC (0.5)	100	0.45	0.5	-
FC (1.0)	100	0.45	1.0	-
FC (1.5)	100	0.45	1.5	-
MCC (0.4)	100	0.45	-	0.4
MCC (0.6)	100	0.45	-	0.6
MCC (0.8)	100	0.45	-	0.8
FC (0.5) + MCC (0.4)	100	0.45	0.5	0.4
FC (0.5) + MCC (0.6)	100	0.45	0.5	0.6
FC (0.5) + MCC (0.8)	100	0.45	0.5	0.8
FC (1.0) + MCC (0.4)	100	0.45	1.0	0.4
FC (1.0) + MCC (0.6)	100	0.45	1.0	0.6
FC (1.0) + MCC (0.8)	100	0.45	1.0	0.8
FC (1.5) + MCC (0.4)	100	0.45	1.5	0.4
FC (1.5) + MCC (0.6)	100	0.45	1.5	0.6
FC (1.5) + MCC (0.8)	100	0.45	1.5	0.8

The evaluation of the initial hydration and rheological measurements of the cementitious composites was carried out in 5 stages. In step 1, the influence of cellulose fibers on the cementitious system was verified. In step 2, the cementitious system was analyzed with the addition of microcrystalline cellulose. In steps 3, 4, and 5, combinations of cellulose fibers and microcrystalline celluloses were studied in FC's-MCC0.4; FC's-MCC0.6; and FC's-MCC0.8, respectively. The objective of carrying out the study in different parts was to investigate the effect of each addition (FC and MCC) and thus be able to understand the combination of the two celluloses in cementitious composites.

2.2.5 Monitoring early hydration reaction for cement composites from temperature versus time

The calorimetry test was performed using the Warme A202 data acquisition device. In plastic bags, the fresh mixtures of cementitious composites with the addition of FC-MCC celluloses. Then, the “K” type thermocouple cable (temperature measuring instrument) with silicone protection model KMO AFD 1P x 24 AWG was introduced into the mixture. Each plastic bag with the mixture was placed in a thermal container. The thermocouple cable was connected to a signal receiver and the data were read and converted into temperature values by a computer program (Lynx). Readings were collected at ten-second intervals over 24 hours. The test was performed with four replicates for each formulation.

2.2.6 Rheological measurements

The rheological behavior was evaluated through the *squeeze-flow* test. *Squeeze-flow* tests without lateral were conducted using a universal testing machine (Emic) equipped with a 1 kN load cell. The configuration used for the *squeeze-flow* test was a 50.8 mm punch, a 101.6 mm restrictive mold (Figure 4), applying a displacement rate of 1 m/s and tightening to a depth of 9 mm. performed in a machine universal that allowed a controlled displacement rate [18]. The test was performed with three replicates for each formulation.

Figure 4. Illustration of the *squeeze-flow* configuration

2.2.7 Fluidity test

The fluidity of cementitious composites with the addition of FC-MCC celluloses was evaluated using the *mini-slump* test. as proposed by Kantro [42]. The fresh mixtures were poured into a metallic mold, with dimensions of 37.5 × 32.5 × 40 mm, next to a flat glass plate in millimeters and a digital caliper to take the readings of the composites. After filling the mold with the cementitious composites, the material was compacted and leveled. Then, the mold was removed, and three diameter readings of the spread material were taken. The test was performed with three replicates for each formulation.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Monitoring initial hydration reaction of cement composite

The measurement of the initial phase of hydration evaluates the release of heat, following the kinetics of the chemical reactions of the cementitious system. The main differences in the evolution of hydration in the first hours occur during three stages: (i) induction period, in which two processes are happening simultaneously (dissolution of ions and formation

of ettringite) which results in the period of chemical dormancy; (ii) period of acceleration or phase transformation, is mainly associated with the reaction of Alita and, therefore, the formation of C-S-H and $Ca(OH)_2$ and, period of deceleration (iii) characterized by a gradual reduction in the rate of reaction, due to the decrease of water content and accumulation of hydration products [43], [44]. Figure 5 shows the results of steps 1, 2, 3, 4, and 5:

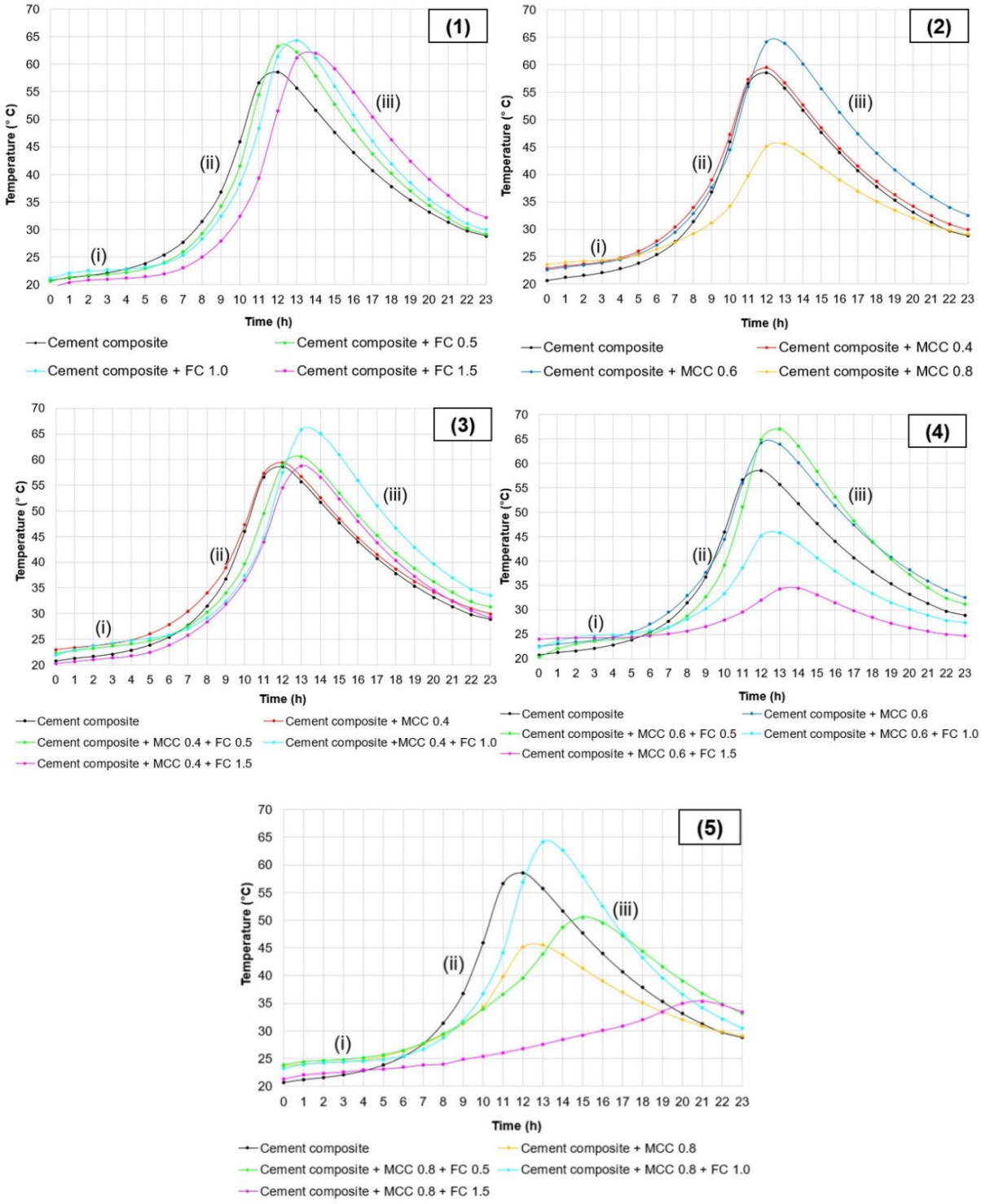

Figure 5. Temperature curves during the hydration reaction. steps 1, 2, 3, 4, and 5.

Table 5. Temperature and time maximal

Samples	Time (h)	T max (ºC)
Reference	12.0	58.5
FC (0.5)	12.2	63.1
FC (1.0)	13.0	64.3
FC (1.5)	14.0	62.0
MCC (0.4)	11.4	59.4
MCC (0.6)	12.4	64.1
MCC (0.8)	12.5	45.5
FC (0.5) + MCC (0.4)	13.0	60.6
FC (0.5) + MCC (0.6)	13.3	65.8
FC (0.5) + MCC (0.8)	12.9	58.7
FC (1.0) + MCC (0.4)	12.8	67.0
FC (1.0) + MCC (0.6)	12.7	45.8
FC (1.0) + MCC (0.8)	13.2	34.3
FC (1.5) + MCC (0.4)	14.8	50.5
FC (1.5) + MCC (0.6)	12.9	47.1
FC (1.5) + MCC (0.8)	20.6	35.3

Fig. **5** (1) shows the time it took each mixture to reach the maximum initial hydration temperature. It was possible to observe that the increase in the fiber content prolonged the occurrence of the maximum temperature peak, however, the cellulose fibers increased the magnitude of the maximum peak.

According to Table **5**, the incorporation of FCs showed an increase in temperature, about the reference. FC 1.0 reached the highest maximum temperature (64.3 ºC) about the reference samples (cement composite), samples with FC 0.5 and FC 1.5, but the time to reach the maximum temperature increased by one hour, when compared to the reference. The FC 1.5 additions took the longest time to reach the maximum temperature, around 14 hours. two hours later than the reference.

Previous research [45]–[48] highlighted that lignocellulosic fibers presented inhibitory substances for cement hydration (hemicelluloses and carbohydrates), whereas Raabe et al. [49] showed that bleached cellulosic pulp fibers did not negatively interfere with the initial hydration of hydrated cement, due to the treatment carried out on the fibers ("kraft" process) which remove the inhibitory substances.

The FCs used in this research have a high silica content (22.9%) (Table 1), not commonly found in lignocellulosic fibers. Silica accelerates the hydration rate of C_3S [50], which corroborates the increase in maximum temperatures of cementitious pastes with the addition of FCs.

Based on Figure 5(2) it is possible to observe that the temperature peaks of the cementitious composites with MCC 0.4 were similar to the peaks of the reference sample. Cementitious composites with the addition of MCC 0.6 reached the highest maximum temperature (64.1 ºC) when compared to the reference, samples with MCC 0.4 and MCC 0.8. This increase may be related to the reaction between MCC cellulose and water (solubilization), the energy activation of alkaline hydrolysis, which is also an exothermic reaction [22], [51], [52]. In addition, the higher maximum temperature increase of cementitious composites with the addition of MCC 0.6 may indicate a spherical stabilization which is responsible for the dispersion of cement particles, and thus, an increase in the degree of hydration occurs [53].

As found by Gómez Hoyos et al. [22], the addition of MCC 0.8 reached the lowest maximum temperature (45 ºC), about the other treatments, which can be attributed to the effective water reduction of the cementitious composites due to the micromaterial agglomeration tendency (Figure 3). which can trap the mixing water. In addition, the reduction of the maximum temperature influences the molecular structure due to the hydroxyl groups present in the cellulose molecules, generating a greater number of interactions between the cellulose molecules and cement hydration products [54].

Unlike the additions of FC's, the maximum temperatures took the same time to be reached in all additions of MCC's, as shown in Table 5.

Figures 5(3), 5(4) and 5(5) show the results of the influence of the combination of microcrystalline cellulose with cellulose fibers, on the initial hydration of cementitious composites.

The combined action of cellulose fibers (0.5% and 1.5%) with microcrystalline cellulose with a content of 0.4% showed curves similar to the reference. as shown in Figure 5(3). This similarity indicated that the FC0.5-MCC0.4 and FC1.5-MCC0.4 formulations did not influence the initial hydration of the cementitious system.

The FC1.0-MCC0.4 formulation showed a peak temperature of 7.3ºC and 6.4ºC higher than the reference samples and with MCC 0.4 samples, respectively. This shows that cellulosic fibers and microcrystalline cellulose promoted the dispersion of cement particles and consequently led to more heat release [55].

In Figure 5(4) it is possible to observe that the maximum hydration temperature peaks for the combined samples FC1.0-MCC0.6 and FC1.5-MCC0.6 suffered a reduction of 12.7ºC and 24.2ºC, respectively, when compared to the reference sample. This behavior can be attributed to the reduction of the effective w/c ratio of these composites, due to the existence of carboxyl groups on the surface of the FC-MCC celluloses that have a hydrophilic behavior and can adsorb the water in the mixture.

The combined sample of FC0.5-MCC0.6 showed an increase in maximum temperature of 8.5ºC and 2.9ºC about the reference sample and MCC 0.6, respectively. This could indicate a dispersive effect of the FC-MCC celluloses, as previously mentioned.

The combined addition of FC's-MCC 0.8 delayed the maximum peaks of the curve and presented lower maximum temperature values of the cementitious composites, as shown in Figure 5(5). The reduction in the maximum temperature may indicate a longer time for hardening due to the process of saturation of the FC-MCC cellulose content in cementitious composites [56].

3.2 *Squeeze-flow*

Squeeze-flow results are given from compressive stress (calculated from compressive strength divided by sample area) vs. displacement of the upper plate for each test. Figure 6 shows the diagram of the results of the *squeeze-flow* test of cementitious composites with the addition of two types of cellulose (FC and MCC).

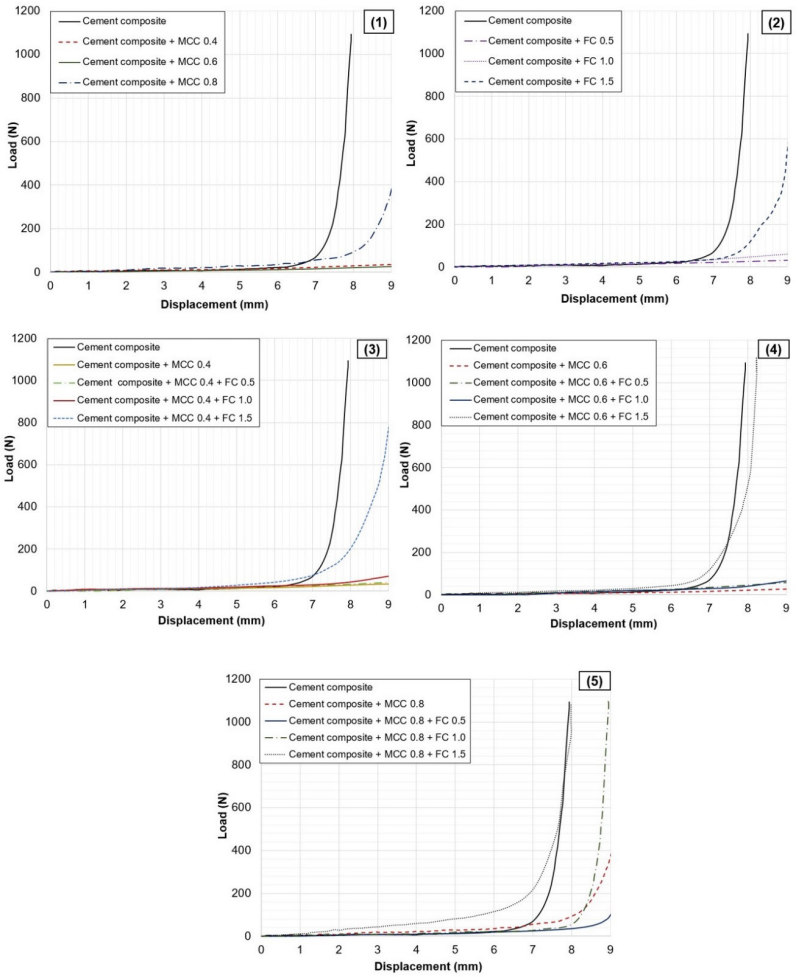

Figure 6. *Squeeze-flow* test performed in cement paste with cellulose FC-MCC (1) MCC's (2) FC's (3) FC's- MCC 0.4 (4) FC's-MCC 0.6 (5) FC's-MCC 0.8

The compression flow load-displacement curves presented in Figure 6 shows two distinct stages of behavior: (a) in this (intermediate) stage, the system flows by plastic deformation or viscous flow, being characterized by considerable deformations without significant increase in force necessary for displacement and, (b) known as strain hardening, in which there is a significant increase in the load necessary for material deformation [18], [20], [57]–[59].

All the studied formulations showed high workability where the cementitious composites flowed easily, with a viscous flow stage with very low loads and extending for most of the curve. The studied samples showed a predominant profile in stage (a).

The transition to the strain hardening stage did not occur in some formulations (MCC 0.4; MCC 0.6; FC 0.5; FC 1.0; FC 0.5-MCC 0.4; FC 1.0-MCC 0.4; FC 0.5-MCC 0.6; FC 1.0-MCC 0.6 and FC 0.5-MCC 0.8). At this stage, the system is more stressed due to the approximation between the particles, where the frictional forces become more intense. Cardoso et al. [18] evaluated that the cementitious systems that presented a predominant profile in stage (b) have a drier consistency, which may correlate with an increase in the coarse fraction in their composition. In the formulations used in the research. coarse fraction components (aggregates) were not used.

In Figure 6(1) it was possible to observe that the cementitious composites with microcrystalline celluloses in 0.4% and 0.6% remained in stage (a) until the end of the test. This occurs because the microcrystalline celluloses interact with the cement particles, releasing trapped water molecules to cement hydration products through electrostatic and spherical stabilization. This interaction improves the dispersion of the particles and consequently enhances the fluidity of the mixture. This behavior has also been observed in the works of Cao et al. [13] and Montes et al. [14], who studied cement composites with the addition of cellulose nanocrystals. Similar effects were reported by Hisseine et al. [60] and Grandes et al. [61] when employing cellulose filaments and cellulose ether, respectively, in cement paste. This mechanism is also exhibited by water-reducing additives to improve workability.

The addition of MCC 0.8, on the other hand, presented a different behavior than the MCC 0.4 and MCC 0.6 formulations, in which the stiffening due to deformation occurred in 8 mm. The addition of MCC 0.8 increased the viscosity and was unable to reduce the friction between particles.

The results obtained from the cementitious composites with the addition of cellulose fiber, as shown in Figure 6(2) showed that the FC 0.5 and FC 1.0 formulations did not show the transition to strain hardening. Cellulose fibers act as a lubricant in cementitious systems, reducing friction between solid particles and thus delaying the transition phase to hardening [62].

In the FC 1.5 formulation, the transition to the stiffening stage occurred at large displacements (N8 mm). Therefore, the concentration of fiber added to the mixture is important for plasticity performance [62]–[64]. Excess cellulose fiber can restrict the flow of cementitious composites [12], [65].

The effect of combining the two types of cellulose (MCC and FC) are shown in Figures 6(3), 6(4) and 6(5). The formulations with FC 1.5-MCC 0.4; FC 1.5-MCC 0.6 and FC 1.5-MCC 0.8 and the additions FC 1.5-MCC 0.6 and FC 1.5-MCC 0.8 had their displacements reduced by 0.4 mm and 0.5 mm, respectively, when compared to the reference (hydrated cement).

The stiffening behavior is associated with friction between particles due to the increased in concentration of solids in the central region between the plates. This behavior may correlate with fluid-solid segregation [58], [66], [67]. Cementitious composites with addition combined with FC 1.5-MCC 0.6 formulations and FC 1.5-MCC 0.8 experienced flow-induced heterogeneity due to phase separation between the fluid and the cellulose particles. It can be concluded that the formulations containing FC 1.5 have a significant impact on the rheological behavior of cementitious components.

The FC 0.5-MCC 0.4; FC 1.0-MCC 0.4; FC 0.5-MCC 0.6; FC 1.0-MCC 0.6 and FC 0.5-MCC 0.8 predominantly showed stage (a) until the end of the test, concluding that microcrystalline celluloses and cellulose fibers can act by improving the rheological behavior. even combined. and in correct dosages. in cementitious components.

3.3 *Mini-slump* test

Figure 7 shows the results presents the *mini-slump* test values and viscosity of the cementitious composites with the incorporation of FC-MCC celluloses evaluated in this work.

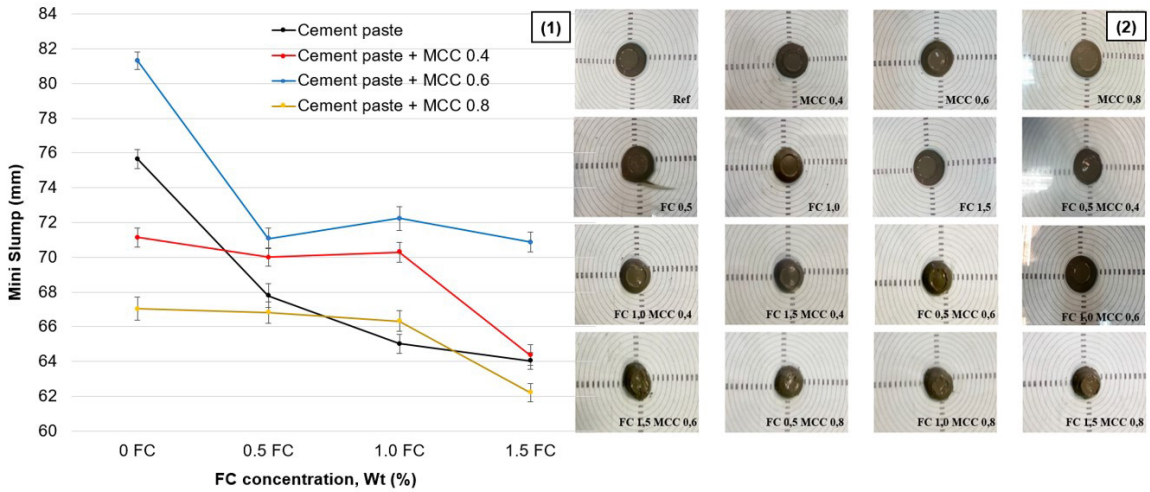

Figure 7. (1) Spread diameter (mm) and (2) Photo schematic of cement composites with cellulose in fibers and microcrystalline in different amounts measured.

In general, it is verified that the FC and MCC and the combination action of the addition of FC-MCC celluloses contributed to less expressive reductions about the reference samples (cement composite) in the *mini-slump* results of the cementitious composites (Figure 7(1)), where the greatest reduction in scattering was the addition of FC 1.5-MCC 0.8. with 17.44% compared to the reference sample.

The results indicate a gradual reduction in the *mini-slump* values with the increase in the cellulose fiber and microcrystalline cellulose contents, in which this behavior shows a decrease in the fluidity of the cementitious composites. However, the addition of MCC 0.6 showed an increase of 7.5% in the spreading diameter compared to reference samples (cement composite), which may correlate with the increase in flow dispersion of cementitious composites [22].

At higher dosages of microcrystalline cellulose (MCC 0.8), it is possible to perceive a reduction of 11.4% in the *mini-slump* values about the reference sample. This behavior may be associated with inadequate dispersion, due to the high specific surface of MCCs, in cementitious systems. Higher levels of cellulose particles promote agglomeration and reduced mobility, which may reduce the adsorption efficiency of the mineral phases of cement [51]–[68].

4 CONCLUSIONS

This article presents the study of the combination of two promising types of cellulose-based materials, FC and MCC, on the fresh properties of cementitious composites.

Based on the experimental results presented, the following conclusions are presented:

- The incorporation of cellulose fibers and crystalline micro cellulose increased the maximum temperatures of the cementitious composites in the first hours of hydration, except the MCC 0.8 formulation. The addition of MCC 0.8 reduced the maximum temperature, justified by the tendency of agglomeration of particles with high surface area in cementitious composites.

- The cellulose fibers took a longer time to reach the maximum temperature, due to components such as lignin and hemicellulose present in the fiber.

- The combined addition of FC 1.0-MCC 0.4 and FC 0.5-MCC 0.6 showed an increase in the maximum temperature, which may indicate that these formulations promoted a dispersive effect and consequently an increase in the maximum temperature. The FC 1.0-MCC 0.6; FC 1.5-MCC 0.6; and FC's-MCC 0.8 showed an inhibitory behavior, which may correlate with the saturation of cellulosic components in the cementitious matrix.

- The spreading capacity measured through compression tests showed that the crystalline microcelluloses MCC 0.4 and MCC 0.6 did not show the transition to strain hardening. This is because crystalline microcelluloses interact with cement particles, improving the fluidity of cementitious composites.

- The addition of FC 1.5 had a different behavior from the additions of FC 0.5 and FC 1.0 in the Squeeze flow test, where the displacement was lower when compared to the reference sample. The formulations combined with FC 1.5-MCC 0.4, FC 1.5-MCC 0.6 and FC 1.5-MCC 0.8 also had their displacements reduced. Thus, it can be concluded that the cellulose fiber content added to the mixture is important for the plasticity performance of cementitious composites.

- The combined FC 0.5-MCC 0.4. FC 1.0-MCC 0.4. FC 0.5-MCC 0.6 and FC 0.5-MCC 0.8 formulations resulted in a long deformation stage plastic, presenting a more homogeneous behavior, concluding that crystalline micro celluloses and cellulose fibers can act as dispersants, even combined, and in correct dosages, in cementitious components.

- The FCs and MCC's and the combination action of the addition of FC-MCC cellulose contributed to less expressive reductions about the reference samples in the *mini-slump* results.

In general, the combinations of CFs with MCC 0.8 reduced the peak temperature of the cement pastes. The study made it possible to identify the saturation of the cellulosic components in this cementitious system, delaying the initial hydration of the cement. The combinations of MCCs with FC 1.5 had an impact on the main phenomena related to elongational flow, which may indicate an increase in friction between the particles and a reduction in the plasticity of the cementitious composites.

The combined contents of FC 1.0-MCC 0.4 and FC 0.5-MCC 0.6 promoted an increase in the maximum temperature, increasing the degree of hydration of the cementitious pastes and did not show a transition to stiffening, concluding micro celluloses and cellulose fibers can act, by improving the fresh state properties in cementitious composites.

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