Mortars with residues of cementitious materials with different fineness

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Abstract

The growing environmental issues about excessive consumption of natural resources, as well as the generation and disposal of landfills, together with a worrying increase in greenhouse gas emissions lead to a new direction for the production and updating of building materials.

The main purpose of the present paper is the production of recycled cement from sufficiently hydrated pastes, representative of existing concrete matrices, and subsequent evaluation of their mechanical efficiency in mortars. To this end, an experimental program was planned, initially involving the treatment of pulp residues, the second evaluation of the material through X-ray diffraction and thermogravimetry, its thermo-activation and the production and characterization in the fresh and hardened state from mortars produced with different incorporations of the recycled cement. In the present work the influence of the thermo-activation temperature is still considered, not forgetting the degree of fineness of the recycled cement and the composition of the mortar.

It was possible to demonstrate the potential reactivity of cement paste initially hydrated and that was subjected to high thermo-activation temperatures, reaching recycled cements of resistance classes similar to those of normal Portland cement (NPC), but more sustainable and associated with less emissions.

***Keywords**:* Recycled cement, thermo-activation, mechanical strength, hydration, mortar.

1 Introduction

1.1 Previous considerations

The growing environmental worries about the excessive consumption of natural resources, as well as the generation and disposal of landfills, and a worrying increase in greenhouse gas emissions lead to a new direction for the production and updating of building materials. [1] [2]

In addition to the use of high quantities of raw materials and the generation of a significant share of the current construction waste, the concrete is responsible for the emission of more than 5% of the total man-made CO_2 emissions. Most of these, about 90%, derive from cement production, with a greater emphasis on the stage related to the burning of raw materials for the production of clinker [3] [4] [5].

In this aspect, in particular in the last 5 years, the experts have been triying to develop recycled cements resulting from the thermo-activation of concrete residues, taking advantage of the fact that the resistance capacity of the concrete when exposed to fire action.

1.2 Dissertation purposes

The main objective of the present work is the production of recycled cement from sufficiently hydrated pastes, representative of existing concrete matrices, and subsequent evaluation of their mechanical efficiency in mortars, when incorporated in different percentages of replacement of normal Portland cement (NPC). This objective expands within the framework of the general guidelines defined in the research project that suits the present work, aiming at the production of more sustainable low carbon recycled cements.

Bearing in mind this, an experimental program was planned, involving in the first phase the treatment of paste residues thermo-activation of which, in a second stage the production and characterization of mortars produced with different incorporations of the recycled cement. In the present work we also considered the influence of the thermo-activation temperature, the degree of fineness of the recycled cement and the composition of the mortar. Within this scope already have been realized works by several authors like [6] [7] [8] [9] [10] [11] [12] [13].

2 Experimental design, materials and methods

2.1 Original paste

In order to represent sufficiently mature cements, the original paste (OP) was produced. This paste was made through two kneading and with a w/c = 0.,40 since it is a ratio that allows handling and compaction in the fresh state without undesirable phenomena occurring as exudation. The Portland cement (CPN) used was provided by the SECIL cement and falls within the strength class of 42.5R. A total of 12 cubic test pieces with a 15 cm edge were produced by kneading. [2]Additionally, 3 cubes of each batch were removed with the purpose of characterizing its mechanical strength on the 28^{th} day, from where the values of 51 MPa and 45 MPa were obtained for the first and second batch, respectively. After this, the remaining specimens were stored under controlled conditions, more precisely in the humid chamber, for about 120 days.

2.2 Production of recycled cement

The production of recycled cement begins with the crushing process. The cubes stored for 120 days in the damp chamber are introduced into the jaw crusher, thus creating particles measuring a maximum diameter of 7 cm and being an elongated laminar shape.

After crushing, the particles were subjected to two different types of milling. In an early stage the roller mill was used in order to reduce the particle size to one of less than 2 mm. After this, all material from the roller mill was placed in the ball mill where about 300 steel balls were rotated at 40 revolutions per minute for 2 hours.

Then a sieving was carried out, where three classes were defined: 1st) material greater than 125 μm; 2nd) material with dimensions between 45 μm and 125 μm; 3rd) material smaller than 45 μm. The first class was again submitted to the ball mill.

Two different types of analysis were then tested. The thermogravimetric analysis was obtained by using the SETARAM equipment, model TGA92, in order to monitor the mass variation of a given material as a function of temperature. The heating rate used was 10°C/min and measurements were carried out up to 1000°C.

In another perspective an X-ray diffraction analysis was carried out, allowing the identification of the crystalline phases in a sample of material, conveying an idea of the evolution of the phases formed and of the reactivation potential of the cements produced. For this purpose, the PANalytical X'Pert Pro was used.

Based on the literature and with the aid of previous analyzes, it was possible to define the treatment temperature of 700ºC, as it is a range that suggests effective dehydration of the ground cement without significant decarbonation. The heating levels were: 1st) 1 hour at 100ºC; 2nd) 3 hours and 30 minutes at a temperature of 350°C, 3rd) Final stage for 5 hours according to the desired temperature. The materials with the highest granulometry (CP) were only submitted to a final plateau of 700ºC while the FP at levels varying between 400ºC and 900ºC. The shape of the particles using the electromagnetic microscope were also analyzed.

We observed through the electron microscope a clear distinction between NPC particles and those of recycled cements, with NPC particles having a compact structure and crystalline appearance. On the other hand, NT particles and thermo-activated cement have a porous structure and a rough surface. The development of hydration products with nano-crystalline structure significantly increases the surface area of these particles, which should have consequences for their water requirement

2.3 Production of pastes and mortars

In the production of pastes and mortars through recycled cement, the various characteristics of recycled cements began to be defined. The density of material CP, FP and NT was determined: M_{CP}=2650 [Kg/m^3], M_{FP}=2690 [Kg/m^3] e M_{NT}=2200 [Kg/m^3]. It is assumed that for material FP the density variations as a function of temperature are extremely small. The mortars were produced with a 1:3 trace (binder: sand) in which 35% is thin sand and 65% coarse sand.

According to standard EN 1015-2, prismatic mortar specimens with 160x40x40 mm were molded.

The molding of the test pieces was carried out in two layers using the compaction table, with 60 strokes per layer, according to EN 196-1. The specimens were then held in the mold for 48 hours in the laboratory environment and protected by a plastic film on the surface. After this time, the samples were demolded and placed in the humid chamber, with a relative humidity of more than 95%. Table 1 shows the mixtures used for the formulation of mortars.

Table 1: Compositions for one liter used for production of mortars with their respective constituents.

Mortars compositions	w/c	Recycled Cement [%]	Recycled Cement [gr]	CEM [gr]	Water [gr]	Thin sand [gr]	Coarse sand[gr]
100%CEM.CN	0,50	0	0	482	241	506	939
80%CEM+20%CP.CN	0,50	20	96	383	240	503	934
50%CEM+50%CP.CN	0,56	50	231	231	258	484	900
100%CP.CN	0,67	100	432	0	290	454	843
100%CEM.HW	0,67	0	0	444	297	466	865
80%CEM+20%NT	0,50	20	95	380	238	499	927
100%CEM.MW	0,58	0	0	463	269	486	903
80%CEM+20%CP.MW	0,58	20	92	368	267	484	898
50%CEM+50%CP.MW	0,58	50	228	228	265	480	891
100%CP.MW	0,58	100	451	0	262	473	879
100%CEM.LW	0,40	0	0	507	203	533	990
80%CEM+20%CP.LW	0,40	20	101	404	202	530	984
50%CEM+50%CP.LW	0,40	50	250	250	200	525	975
400FP	0,70	100	428	0	299	449	834
500FP	0,70	100	428	0	299	449	834
600FP	0,70	100	428	0	299	449	834
700FP	0,72	100	424	0	306	445	827
800FP	0,76	100	416	0	317	437	812
900FP	0,77	100	415	0	320	435	809

2.4 Characterization of mortars in the fresh and hardened state

In the first place, the normal consistencies for the pastes produced with the thermally activated cements was determined, we carried out a similar procedure for the NPC. The normal consistency of a paste is measured on the basis of the penetration resistance of a standard size probe with the aid of the *Vicat* apparatus. The determination of the holding times is then carried out. Through the *Vicat* needle and the folders of normal consistencies obtained, it is possible to estimate the setting time, according to NP EN 196-3 (2006).

For a better characterization of the cements was done also the expansion test, present in the same directive as the aforementioned tests, is carried out, using the aid of *Le Châtelier*.

In order to monitor the rate of heat of hydration released by the recycled cement and to evaluate its capacity and hydration speed, isothermal calorimetry tests were carried out. This test was performed with T700FP material and w/c = 0,70 due to the high-water requirement of the furnace materials.

Subsequently the mortars were characterized. According to the compositions shown in Table 1 the desired mortars were produced and several tests were carried out. In the fresh state the characterization begins by the flow test, it has the objective of measuring the consistency of a mortar through the ability of the mortar to flow when it is subjected to an external action.

Measuring the average spreading diameter when subjected to a number of strokes at a specific table. The fresh density of the mortars was also determined according to EN 1015-6 (1998). This parameter is the quotient between the mass of the material contained within the cup and the value of the cup volume. In addition, the volume of voids was also calculated.

For the characterization of the mortars in the hardened state only two tests were carried out: bending strength and compressive strength. Both were performed using the Seider Form+Test equipment model 505/200/100 DM1. The flexural test is performed with the specimens simply supported on fitted supports, the load is applied half-way at a speed of about 0.5 KN/s and conducted to a 10 KN load cell. On the other hand, each half of the specimens coming from the flexion is tested under compression, using the same equipment, but for a load cell of 200 KN.

3 Results and discussion

3.1 TG/DTG and DRX

As would be expected, cements treated at higher temperatures develop higher amounts of CaO, resulting from dehydroxylation of CH and eventual decarbonation of $CaCO_3$. Sufficiently dehydrated cements with no significant CO_2 release occur at 650 to 700°C. Differential gravimetric (DTG) and differential heat flux (dHF) curves demonstrate the effective dehydration of the main cement paste compounds, in particular CSH, ettringite, CH and gypsum. There is also an apparent carbonation after the dehydroxylation of Portlandite during the burning of the recycled cement, with consequent increase of the calcite content in the final product. Figure 1 shows the results obtained for thermogravimetry.

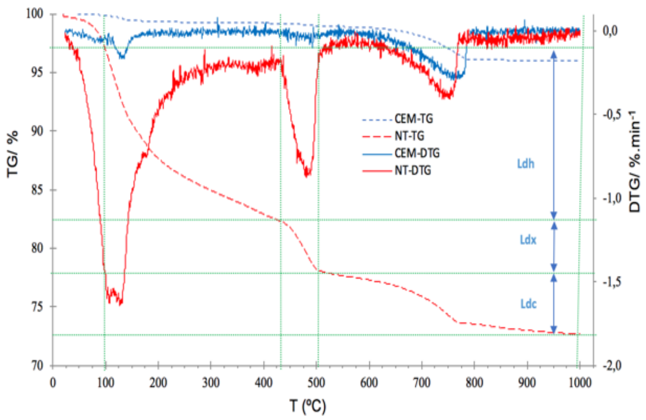

Figure 1: Thermogravimetric analysis of the untreated material with dimensions between 45 µm and 125 µm and of the ordinary Portland cement used.

The XRD tests, performed at different treatment temperatures, highlight the progressive depolymerisation of CSH and the reduction of Portlandite with temperature, as well as the consequent formation of low crystallinity nano-products and a new neurosilicate of stoichiometry similar to larnite (C_2S). For temperatures above 800 ºC, as observed by other authors, it is verified that the obtained bicalcium silicates are better defined and associated with greater crystallinity, being able to become less reactive. This may justify the worse results of mechanical strength obtained in the present work for mortars with cement treated at 900 ºC.

3.2 Characterization of recycled cement and pastes

The water requirement of pastes of normal consistency increased with the percentage of incorporation of recycled cement and with the increase of its thermos-activation temperature. The higher porosity and surface area of the recycled cement particles, the eventual agglomeration of these particles and the presence of CaO contribute to this trend. However, in the binders incorporating recycled cement up to 50%, the water requirement was not significantly increased, and there is a synergistic effect between the recycled cement and the CPN, which maintains an adequate workability of the system.

The setting times in the recycled cements at 700ºC were inferior to those of the NPC. In pastes composed only by recycled cement, the start of setting time was lower than the 45 minutes recommended in EN 197-1 (2001), which requires the use of retarders. This is justified by the high initial reactivity of the dehydrated cements, as evidenced by the analysis of the evolution of the rate of heat of hydration. However, contrary to what was observed by other authors, for percentages of incorporation up to 50% or in cements treated above 800°C, there were no shorter setting times than in the pastes with NPC. The possible agglomeration in the recycled cement and the available CaO content may explain this tendency. On the one hand, as reported by [9] a false setting time phenomenon was observed, in which initial hardening of the paste surface changes after remixing. On the other hand, due to greater difficulty in dispersion, the cement treated at 600ºC presented a shorter setting time, which corroborates the higher mechanical strength of these cements at the early ages.

Although recycled cements were associated with higher expansibility rates due to the presence of CaO, the maximum limit specified in EN 196-1 (2005) was not exceeded. The pH of the recycled cements was also measured immediately after their rehydration, showing that the pastes have alkalinity values similar to those of reference, with only CPN.

The analysis of the evolution of the heat of hydration allowed to verify that if there is no agglomeration, the recycled cements tend to present a higher initial reactivity during the first 2 hours, which then dilutes to later ages, leading to lower values of heat of hydration , after 6 hours, than the NPC. Although recycled cements involve the same hydration steps as NPC, the exothermic reactions concentrate more in the initial period after contact of the water with the binder. To do so, it contributes to the greater surface area of the recycled cement and its greater amount of free lime. According to the XRD and SEM analyzes performed on the hydrated cements, the reactivity of the aluminate and carbonate phases may also contribute to this phenomenon.

3.3 Characterization of fresh mortars

As expected, the density decreased with the percentage of incorporation of recycled cement, due to the lower density of this binder. There is an apparent increase in voids content in mortars with recycled cement, which also contributes to the reduction of the density of the mortar and is justified by its lower workability and greater difficulty of compaction. Figure 2 shows the results obtained for the fresh mortar tests: Flow, fresh density and voids content.

Table 2: Results of fresh density, flow and void volume tests.

Mortars compositions	w/c	Fresh density [m^3]	Teorical density [m^3]	Flow [mm]	Voids contente [%]
100%CEM.CN	0,50	2176	2168	187	4,8
80%CEM+20%CP.CN	0,50	2135	2156	171	6,1
50%CEM+50%CP.CN	0,56	2104	2104	183	5,3
100%CP.CN	0,67	1970	2019	169	7,8
100%CEM.HW	0,67	2157	2072	fluido	1,2
80%CEM+20%NT	0,50	2136	2139	168	6,1
100%CEM.MW	0,58	2211	2121	195	1,1
80%CEM+20%CP.MW	0,58	2191	2109	194	1,5
50%CEM+50%CP.MW	0,58	2141	2092	166	3,1
100%CP.MW	0,58	2060	2065	163	5,7
100%CEM.LW	0,40	2148	2233	189	8,7
80%CEM+20%CP.LW	0,40	2128	2221	177	9,1
50%CEM+50%CP.LW	0,40	2031	2200	161	12,6
400FP	0,70	1689	2010	151	20,3
500FP	0,70	1704	2010	145	19,6
600FP	0,70	1728	2010	181	18,5
700FP	0,72	1813	2002	179	14,1
800FP	0,76	1861	1982	167	11,0
900FP	0,77	1869	1979	163	10,4

Since recycled cement is associated with a higher water demand, mortars of equal to w/c with a higher percentage of incorporation of this binder presented worse workability or, for the same workability, required more water of kneading with consequent increase of w/c. Still, for percentages of incorporation of up to 20% of recycled cement, the change in workability was not significant. The best workability was obtained for treated cement between 600ºC and 700ºC. For lower temperatures the mixtures were difficult to work with and showed little cohesion between their constituents. For temperatures above 700°C, the significant increase in CaO led to a high-water requirement.

3.4 Mechanical strength of mortars

For the mechanical resistance the interpretation and description of the results will be carried out essentially for the compressive strength, since in relation to the flexural strength the obtained tendencies were similar considering that the behavior is identical.

Table 3 summarizes the average values of the compressive strength and flexural strength of the mortars produced, for the ages in which they were tested. In general, the coefficients of variation were less than 5% in the compressive strength and 10% in the flexural strength. For mortars produced with the same workability (100%CEM.CN; 80%CEM+20%CP.CN, 50%CEM+50%CP.CN and 100%CP.CN), it is verified that the compressive strength on the 28th day of mortars with 20%, 50% and 100% incorporation of recycled cement (CP) were 92%, 72% and 36% of the resistance of the reference mortar, respectively. Nevertheless, after the high w/c of the mortar with 100% CP, it was able to reach 23.1 MPa on the 28th day, demonstrating high reactivity.

Table 3: Mean values of the compressive and flexural strengths and coefficients of variance on the 28th day of mortars produced [MPa].

Mortars	3 days		7 days		28 days			90 days	
Compositions	μ_{fcm}	μ_{fctm}	μ_{fcm}	μ_{fctm}	μ_{fcm}	cv_{fcm}	μ_{fctm}	μ_{fcm}	μ_{fctm}
100%CEM.CN	26,6	4,6	44,7	7,9	65,1	2,5	10,8	80,9	11,6
80%CEM+20%CP.CN	33,2	6,1	48,1	7,6	59,7	1,8	8,4	71,3	9,6
50%CEM+50%CP.CN	18,1	3,3	33	5,6	47,1	2,9	8,2	56,3	9,3
100%CP.CN	3,4	1,3	6	2,1	23,1	8,7	5	30,4	5,9
100%CEM.HW	17,5	3,9	27,1	5,6	44,2	5,9	8,7	-	-
80%CEM+20%NT	27,4	4,9	36	7,1	48,4	2,3	8,1	-	-
100%CEM.MW	26,3	5,6	39,9	7,1	51,3	4,9	9,2	-	-
80%CEM+20%CP.MW	22,1	4,9	32,2	7,5	45,6	6,8	8,2	-	-
50%CEM+50%CP.MW	23,8	4,5	29,4	6	39,7	3	7,8	-	-
100%CP.MW	16,4	3,7	21,6	4,3	27,1	6	6,2	-	-
100%CEM.LW	49,1	8,1	61,5	10,1	76,5	4,1	11,9	-	-
80%CEM+20%CP.LW	46,1	8,7	61,7	9,6	74,9	2,9	10,7	-	-
50%CEM+50%CP.LW	35,2	6,3	49,1	8,7	66,9	3	9,2	-	-
400FP	0,6	0,3	-	-	1,2	8,5	0,6	-	-
500FP	4,6	1,5	-	-	9,3	2,3	2,6	-	-
600FP	15,3	3,4	-	-	21,6	2,4	4,6	-	-
700FP	4,9	1,7	-	-	15,9	1,7	4	-	-
800FP	5,6	1,7	-	-	26,4	3,4	4	-	-
900FP	5,1	1,1	-	-	24,6	4,4	6,3	-	-

Then mortars with w/c of 0.58 and different percentages of substitution were produced: 100% CEM.MW; 80%CEM + 20%CP.MW; 50%CEM+50%CP.MW; 100%CP.MW. On the 28th day, the compressive strength for 20%, 50% and 100% replacement were 89%, 77% and 53% of that obtained in the reference mortar, respectively. When compared to mortars 100%CEM.CN; 80%CEM+20%CP.CN, 50%CEM+50%CP.CN and 100%CP.CN, One can concluded that the efficiency level of the recycled cement was not significantly affected by the w/c ratio. In Figure 2 the compressive strength on the 28th day as a function of the w/c ratio for mortars with only cement or mortars produced with different percentages of treated cement at 700ºC of greater granulometry (CP).

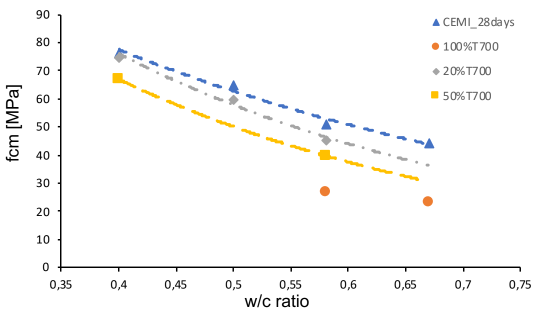

Figure 2: Compressive strength on the 28th day, for mortars with different percentages of substitution, varying the relation a / l.

Mortars with recycled cement, for the same workability, show a slower evolution in the compressive strength up to 28 days than the reference mortar with NPC. On the other hand, for low percentages of incorporation of recycled cement in mortars of equal w/c, the evolution of strength was faster than in the reference mortar. Thus, other factors such as the w/c ratio and the quality of the dispersion attained in the binder may have an influence on this behavior.

In Figure 3, it is verified that for identical w/c of 0,58 the resistance of the mortar with 100%CP was already 62% and 53% of the resistance of the reference mortar on the 3rd and 28th days, respectively. In this case, the fact that superplasticizers have been added in the mixture with 100%CP in order to have a smaller w/c than the reference mortar. This is in accordance with the study of [10], which shows that the use of additives to improve the dispersion of recycled cement leads to a more rapid development of resistance.

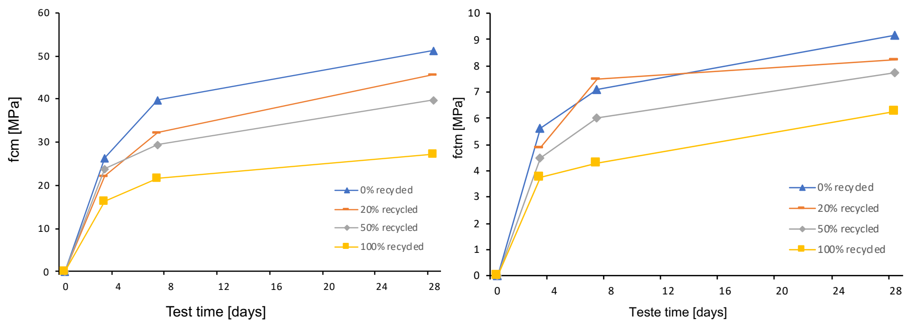

Figure 3: Mechanical strength over the test time for mortars produced with w /c = 0.58: 100%CEM.MW, 80% CEM+20% CP.MW, 50% CEM+50% CP.MW and 100%CP.MW

Figure 4 shows the results of the compressive strength and flexural strength of the mortars produced with 100% recycled cement of particle size less than 45 μm (FP):

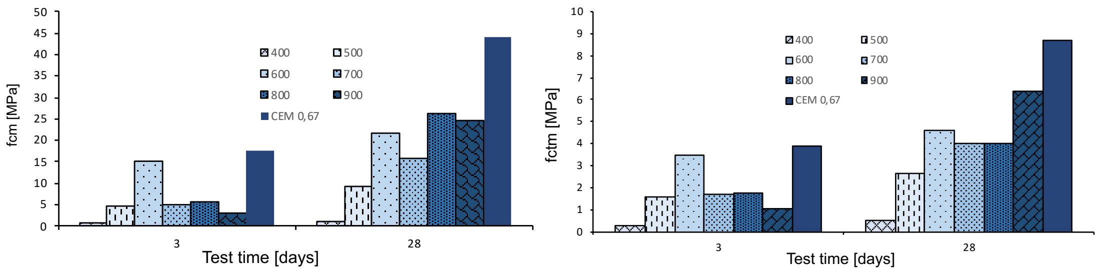

Figure 4: Mechanical strength for recycled cement mortars produced with dimensions over 45: A.R400FP; A.R500FP; 600FP; 700FP; 800FP; 900FP; 100%CEM.HW.

We could observe that, except for cement mortar treated at 400ºC and at 600ºC, the initial resistance on the 3rd day was similar for the different treatment temperatures. Cement treated at 400 °C revealed low reactive capacity at all test ages, according to [13]. In the remaining cases, only cement treated at 600ºC presented initial reactivity compatible with what is referred by other authors [11] [7]. Regarding the results at 28 days of Figure 4, an abnormally lower value occurs at 700ºC, which escapes the aforementioned trend of increasing resistance with temperature. This phenomenon may be related to the greater susceptibility of the recycled cements to rehydrate partially during their storage, especially due to the presence of free CaO in their constitution. The mechanical resistance at 28 days increased with the activation temperature up to 800ºC, decreasing slightly to 900°C. This trend, which is also documented by other authors, can be attributed to the alteration in the degree of crystallinity and reactivity of the formed compounds, namely the type of bicalcium silicates.

The reduction of the fineness of recycled cement from 45-125 μm to a dimension of less than 45 μm did not affect positively the mechanical strength. The greater difficulty of dispersion associated with the finer cements and the fact that the cement granulometry decreases after heat treatment justifies these results, as can be seen in the Figure 5.

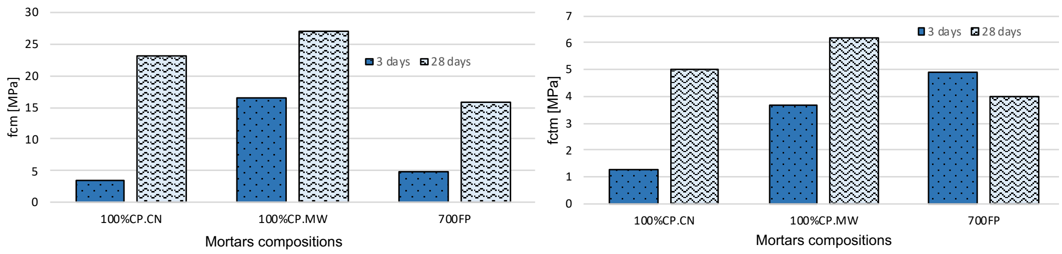

Figure 5: Compressive and flexural strength for mortars produced with different fineness and w/c, treated at 700ºC

4 Conclusion and future developments

The main conclusions of this work are presented in this chapter. The water requirements of normal consistency pastes increased with the percentage of incorporation of recycled cement and with the increase of its thermo-activation temperature. The density decreased with the percentage of incorporation of recycled cement. For equal w/c the mortars with higher percentage of incorporation of this binder presented lower workability. Through this work it was possible to demonstrate the potential reactivity of cement paste initially hydrated and that was subjected to high thermo-activation temperatures, reaching recycled cements of resistance classes similar to those of normal Portland cement (NPC), but more sustainable and associated with lower CO_2 emissions. For cements treated at 700°C and maximum size up to 125 μm, binders with 20%, 50% or 100% of this constituent can be classified in class 52.5, 42.5 and 32.5, respectively. It is concluded to, that due to the more reactive nature of its components and the presence of CaO, recycled cements tend to be more susceptible to storage conditions.

Some studies can lead to the development in the future: To overcome the problem of reduced setting time and low workability of recycled cements, as well as the initial development of high heats of hydration; Identification of the various phases created in the recycled cements and analysis and monitoring of the subsequent development of hydration products; Microstructural characterization of the cementitious matrix developed at various ages in the presence of recycled cements; Production and characterization in the fresh and hardened state of mortars and concretes produced with recycled cement; Analysis of the influence of recycled cements on the deferred behavior of mortars and concretes; Analysis of the durability of concrete produced with the incorporation of recycled cement; Analysis of the influence of the introduction of mineral additions in cementitious mixtures produced with recycled cement.

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