Subject: Experimental research for evaluating the performance of ***MetacaulimHP*** as addition to Portland Cement Concrete

Sponsor: METACAULIM do Brasil Indústria e Comércio Ltda.
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Progress Report II: Results obtained until August 2002. This report modifies and substitutes the progress report I.

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1. INTRODUCTION

At the request of the Sponsor it had performed, since the beginning of 2002, to the evaluation of the performance of the ***MetacaulimHP*** as a high efficiency addition to Portland Cement concrete. This is the second report on the progress of the experimental lab research.

This report presents part of the studies that are in progress in the laboratory of the Escola Politécnica of the University of São Paulo, in the IPT-Institute for Technological Research of the State of São Paulo and in the L.A. Falcão Bauer, under supervision and design of the Professor Paulo Helene.

Engineer Charles Siervi Lacerda has carried out the experimental works as part of his activities as a graduate student at the Escola Politécnica of the University of São Paulo (EPUSP).

This report modifies and substitutes the previous one.

According to the Sponsor, the Metacaulim do Brasil Ltda, is the first Brazilian company to produce and to commercialize the ***MetacaulimHP*** in industrial scale, being this product derived from carefully selected kaolinite clay and its calcinations at very controlled temperatures.

MetacaulimHP is mostly constituted by silica (SiO_2) and alumina (Al_2O_3) in the amorphous phase (glass) resulting a high reactivity material that reacts with calcium hydroxide $Ca(OH)_2$ provided by the hydration of the cement composites.

According to the Sponsor the ***MetacaulimHP*** is recommended for indiscriminate use in Portland-cement concrete because its physical and chemical characteristics improve the concrete mechanical and durability properties. The ***MetacaulimHP*** is still recommended to various applications in the industry of refractory, ceramic, metallurgy of iron and steel, chemistry and others.

The results obtained are promising and indicating that the ***MetacaulimHP*** represents an strong interesting option in the search of quality, economy, sustainable development and durability of the Portland-cement concretes.

MetacaulimHP **TECHNOLOGICAL DATA**

2.1 Chemical analysis (ASTM C 114, adapted)

The chemical analysis of the ***MetacaulimHP*** was carried through in the Laboratory of Technological Materials Characterization of the Department of Engineering of Mines and Oil of the Escola Politécnica of the University of São Paulo.

The half-quantitative for fluorescence of rays-x is presented in Table 2.1.1, transcribed from the certificate EPUSP LCT 204/02.

Table 2.1.1 Results in % of oxides, normalized at 100%.

SiO_2	51,2
Al_2O_3	35,3
Fe_2O_3	4,00
CaO	2,62
K_2O	0,97
TiO_2	0,41
MgO	0,40
P_2O_5	0,20
MnO	0,16
SO_3	0,09
SrO	0,02
ZrO_2	0,02
PbO	0,02
ZnO	0,01
Ga_2O_3	0,01
Rb_2O	0,01
Y_2O_3	0,01
Cr_2O_3	<<
Nb_2O_5	<<
ThO_2	<<
PF	4,57

<< values below 0,00%

The results of Table 2.1.1, shows that the ***MetacaulimHP*** is composed mainly by siliceous and aluminous materials and lesser components, characterizing itself as a typical reactive material with pozzolanic characteristics with Portland Cement concretes.

2.2 Analysis of Particle Size

The analysis of particle size of the ***MetacaulimHP*** was carried through in the Laboratory of Technological Materials Characterization of the Department of Engineering of Mines and Oil of the Escola Politécnica of the University of São Paulo.

The resultant grain size distribution is graphically represented in Fig. 2.2.1, transcribed from the certificate EPUSP LCT 288/02.

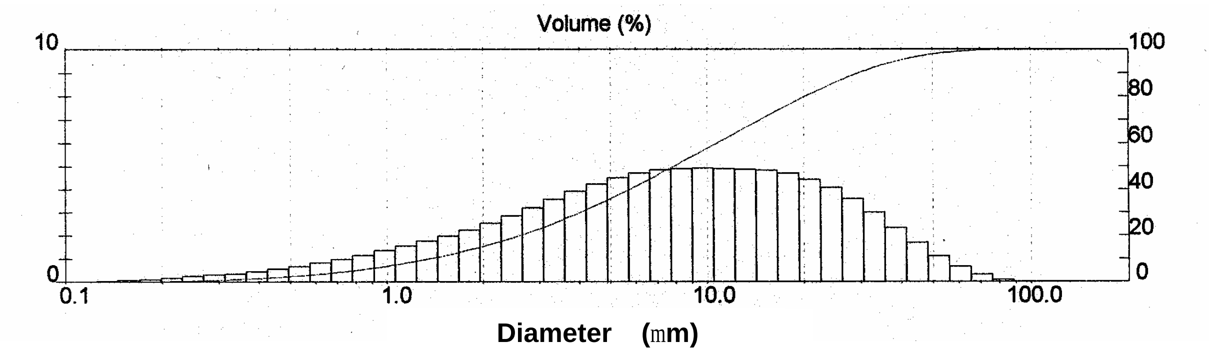


Figure 2.2.1 Particles size distribution of ***MetacaulimHP***

In Table 2.2.1 it can be observed the values of average diameter, density and specific superficial area obtained from the analysis of particle size.

Table 2.2.1 Analysis of particle size

Average diameter	12.4	µm
Density	165.4	lb/ft^3
Specific superficial area	738	m^2/kg

As showed in Table 2.2.1, the ***MetacaulimHP*** is a very fine material. It is finer than the Portland cements produced in Brazil.

2.3 X-Ray Powder Diffraction Analysis (ASTM C 1365, adapted)

The X-Ray Powder Diffraction Analysis of the ***MetacaulimHP*** was carried through in the Laboratory of Technological Materials Characterization of the Department of Engineering of Mines and Oil of the Escola Politécnica of the University of São Paulo. The used equipment was of the dust, using the X-Ray diffractrometer, Philips, model MPD 1880.

Through the analysis of the difratogram of the sample and its comparison with the database of the *ICCD - International Center for Diffraction Data* had been identified the constant crystalline phases of Table 2.3.1, transcribed from the certificate EPUSP LCT 083/02.

This analysis proves the pozzolanic nature of the material already evidenced by the chemical analysis.

Table 2.3.1 X-Ray Powder Diffraction Analysis

Name of the composition	Chemical formula	Name of the mineral
Silica	SiO_2	Quartz
Kaolinite	$Al_2Si_2O_5(OH)_4$	Kaolinite
Phlogopite	$KMg_3Si_3AlO_{10}(F,OH)_2$	Phlogopite
Hematite	Fe_2O_3	Hematite
Ilmenite	$FeTiO_3$	Ilmenite

2.4 Determination of Pozzolanic Activity - Method of modified Chapelle (ASTM C 618, adapted)

The determination of pozzolanic activity was carried by the IPT - Institute of Technological Research of the State of São Paulo through the DEC-LQM-PE-041 procedure "Determination of pozzolanic activity – Modified Chapelle Method", based on the publication "Appréciation de l'activité pouzzolanique des constituants secondaires" - 7^{e}. Congrès International de la Chimie des Ciments, Paris - 1980, Vol. III, IV-36/41, by Raverdy M., Brivot F., Paillere A.M., Dron R.

The result is showed in the Table 2.4.1, transcribed from the report of assay IPT 890 816.

Table 2.4.1 ***MetacaulimHP*** pozzolanic activity

Sample identification	Result of pozzolanic activity at $(90\pm5)^{o}$C (mg CaO/g sample)
MetacaulimHP (caulinitic calcined clay)	729,4

According to Raverdy, mentioned previously, the difference between the compressive strength of mortars of cement with and without pozzolanic material is practically equal to zero for materials of low reactivity less than 330 CaO/g sample, corresponding of calcium oxide (CaO) reacted in 182 days.

As the analyzed material, ***MetacaulimHP*** reacted 729,4 mg CaO/g sample, over the value to the limit of 330 mg CaO/g sample, as described above. It can be concluded that ***MetacaulimHP*** is a high reactivity pozzolanic material.

3. EXPERIMENT, RESULTS and INITIAL ANALYSIS

In this chapter we describe the experimental design that was built to evaluate the behavior of the ***MetacaulimHP*** as a mineral addition to be used in normal and high performance concrete.

It is opportune to explain that concrete ones considered here are those that present high compression strength above 4,350 psi (30 MPa), in average, at 28 days.

3.1 Experimental design

Three different mixture proportions with cement-to-aggregate ratio of 1:3, 1:4 e 1:5, in mass of dry material, were chosen for comprising the ordinary structural concrete.

A substitution, in mass, of eight percent (8%) of the cement content by ***MetacaulimHP*** was adopted.

In all performed concrete was incorporated an admixture 395 N produced by MBT-Master Builders Technology Brasil, because that one is an admixture normally used in the concrete ready mix plants in the region of the great São Paulo. This admixture can act as a water reducer, as a plasticizer and as a retarder, according ASTM C 494 type D and E.

The fine aggregate used was the natural sand with fineness modulus of 1,21 and bulk density of 165.44 lb/ft^3 (2.650 kg/m^3). Granite crushed with fineness modulus of 6,91 and bulk density of 166.06 lb/ft^3 (2660 kg/m^3) was used as a coarse aggregate. The coarse aggregate was washed previously and dried in greenhouse. The fine aggregate was only dried in greenhouse. The aggregates are according to ASTM C 294.

The cement used in this research was the ASTM C 150 type III, made by HOLCIM, characterized in Table 3.1.1. It was chosen for being used by the concrete ready mix plants in São Paulo region.

Table 3.1.1 - Physical characteristics of the cement used in the studies

Tests	**Standards**	**Results**	**Specification ASTM C 150**
Fineness – fraction of cement retained on a 75µm test sieve(%)	ASTM C 184	1,6$^{(3)}$	-
Bulk density (lb/ft^3)	ASTM C 188	189.16	-
Specific surface area (m^2/kg)	ASTM C 204	379	-
Cement paste standard water (%)	ASTM C 187	31,0	-
Initial set (h:min)	ASTM C 191	3:25	$\geq$ 0:45
Final set (h:min)	ASTM C 191	5:15	$\leq$ 6:15
Le Chatelier’s expansibility – by heating (mm)		0,5	-

The compressive strength of the cement in the standardized mortar is indicated in Table 3.1.2.

Table 3.1.2 - Compressive strength of the cement used in the studies

Compressive strength (psi), in accordance with ASTM C 150							
Age (days)	Sample n. (MPa)				**Average** psi (MPa)	Maximum relative deviation (%)	specification ASTM C 150
	1	2	3	4			MPa
1	13.5	14.3	13.2	14.3	2.001 (13.8)	4.7	≥ 12
3	28.9	29.7	28.3	29.2	4,206 (29.0)	2.4	≥ 24
7	38.5	36.9	37.2	36.2	5,366 (37.2)	3.5	-

3.1.1 Mix Proportion of Concrete Performed

A summary of the mix proportion concrete studied in this research can be seen in Table 3.1.3.

Table 3.1.3 Synthesis of the mixture proportions.

Mixture proportion	name	substitution	cement : admixture : fine agg : coarse agg. ; w/c	Υ_c (lb/ft^3)	Air content (%)	Cement Content (lb/ft^3)
1 : 3	R	reference (0%)	1.00 : 0.00 : 0.96 : 2.04 ; 0.31	150.6	1.7	35.0
	M	*MetacaulimHP* (8%)	0.92 : 0.08 : 0.96 : 2.04 ; 0.34	149.3	1.2	31.7
1 : 4	R	reference (0%)	1.00 : 0.00 : 1.45 : 2.55 ; 0.42	148.7	2.0	27.4
	M	*MetacaulimHP* (8%)	0.92 : 0.08 : 1.45 : 2.55 ; 0.46	147.4	1.3	24.8
1 : 5	R	reference (0%)	1.00 : 0.00 : 1.94 : 3.06 ; 0.52	147.9	1.9	22.7
	M	*MetacaulimHP* (8%)	0.92 : 0.08 : 1.94 : 3.06 ; 0.58	146.1	1.9	20.4

3.2 Independent Variables

The following fixed independent variable had been considered:

- Cement ASTM C 150 type III, CIMINAS / HOLCIM
- Fine aggregates, fine natural quartz sand
- Coarse aggregates, granite
- Potable water
- Plasticizer 395 N produced by MBT , ASTM C 494 type D and E
- 3.15 in slump ASTM C 143
- Aggregate/cement ratio of 3, 4 and 5, in dry mass
- Substitution of 8% of cement content by the ***MetacaulimHP***, by mass (weight)

3.3 Dependents variables

Six tests had been initially elected so that, in the first phase of the studies of the ***MetacaulimHP***, it could be evaluated not only the behavior of the concrete on the point of view of the mechanical properties but also on the durability, as showed in Table 3.3.1.

Different ages for the tests, as described in Table 3.3.1, had been chosen so that the evolution of the pozzolanic activity of the ***MetacaulimHP*** in the concrete could be observed, mainly, through the changes of durability and mechanical properties.

Table 3.3.1 Dependents variables.

properties	method	age (days)
Compressive Strength	ASTM C 39 / 39M	1, 7, 28, 63, 91 e 182
Splitting Tensile Strength	ASTM C 496	7, 28, 63, 91 e 182
Static Modulus of Elasticity	ASTM C 469	7, 28, 91 e 182
Chloride Ion Penetration	ASTM C 1202	28, 63 e 91
Density, Absorption and Voids	ASTM C 642	28, 63 e 91
Electrical Resistivity	ASTM G 57, adapted	28, 63 e 91

3.4 Compressive Strength

The test of compressive strength had been executed in accordance with ASTM C 39 / 39M. The summary of the results obtained for the different mixture proportions considering workability with 80 mm slump is showed in Table 3.4.1.

Table 3.4.1 Compressive strength , 3.15 inch slump.

Mix proportion	1 day		7 days		28 days		63 days		91 days	
	(MPa)	Psi	(MPa)	psi	(MPa)	psi	(MPa)	psi	(MPa)	psi
1 : 3 R	(34.0)	**4931**	(47.6)	**6903**	(60.0)	**8702**	(61.7)	**8948**	(61.9)	**8977**
1 : 3 M	(27.6)	**4003**	(58.0)	**8412**	(68.8)	**9978**	(72.1)	**10457**	(76.4)	**11080**
1 : 4 R	(32.3)	**4684**	(40.3)	**5845**	(49.9)	**7237**	(50.7)	**7353**	(51.9)	**7527**
1 : 4 M	(20.2)	**2929**	(50.5)	**7324**	(56.7)	**8223**	(59.6)	**8644**	(62.2)	**9021**
1 : 5 R	(24.9)	**3611**	(35.4)	**5134**	(40.3)	**5845**	(41.9)	**6077**	(45.6)	**6613**
1 : 5 M	(20.1)	**2915**	(38.2)	**5540**	(42.8)	**6207**	(57.4)	**8325**	(60.3)	**8745**

3.5 Splitting Tensile Strength

The tensile strength was determined by splitting tensile method in accordance with ASTM C 496 where the cylindrical sample, with 4 in of diameter and 8 in of height, as can be seen in Fig. 3.5.1.

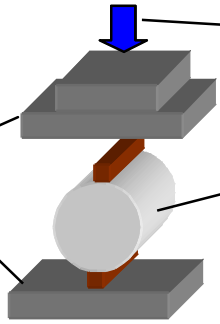


Figure 3.5.1 Simplified sketch of the splitting tensile method.

The results is showed in Table 3.5.1 and 3.5.2 followed by the values of fc_{tk}.min and fc_{tk},max that correspond to the minimum and maximum limits of the tensile characteristic strength for the concrete ones based on the values of compressive strength gotten in this study and using the equations, below, proposed by the CEB-FIP Model Code 1990 (CEB-Comité Euro-International du Beton, FIP-Fédération Internationale de la Précontrainte).

$$f_{ctk,\min} = 0{,}95\left(f_{ck}/f_{cko}\right)^{2/3}$$

$$f_{ctk,\max} = 1{,}85\left(f_{ck}/f_{cko}\right)^{2/3}$$

It is observed, initially, in Tables 3.5.1 and 3.5.2, that the values gotten for tensile strength of the concrete ones in study are inside of the limits foreseen for the *International Code* [***fib***(CEB-FIP) 1990]. It is still observed that the values of strength for the concrete of reference (R), without addition of ***MetacaulimHP***, are below of those gotten for the concrete ones with addition of ***MetacaulimHP***, in all the mixture proportions. That happens because the pozzolanic addition promote chemicals reactions that reduce the size and the concentration of crystals of calcium hydroxide in the transition zone, one of the most fragile point of the concrete when subjected to the tensile efforts, according to Mehta & Monteiro (2000).

Table 3.5.1 Tensile strength, 3.15 inch slump.

Mixture proportion	**$f_{ctk,min}$**		**Age (7 days)**		**$f_{ctk,max}$**		**$f_{ctk,min}$**		**Age (28 days)**		**$f_{ctk,max}$**	
	(MPa)	**psi**	(Mpa)	**psi**	(Mpa)	**psi**	(Mpa)	**psi**	(Mpa)	**psi**	(Mpa)	**psi**
1 : 3 R	(2.7)	392	(3.9)	**566**	(5.2)	754	(3.1)	450	(4.4)	**638**	(6.1)	885
1 : 3 M	(3.1)	450	(4.1)	**595**	(6.0)	870	(3.5)	508	(4.7)	**682**	(6.9)	1001
1 : 4 R	(2.6)	377	(3.8)	**551**	(5.1)	740	(2.8)	406	(4.1)	**595**	(5.4)	783
1 : 4 M	(2.8)	406	(4.1)	**595**	(5.4)	783	(3.0)	435	(4.2)	**609**	(5.9)	856
1 : 5 R	(2.2)	319	(3.2)	**464**	(4.3)	624	(2.4)	348	(3.9)	**566**	(4.7)	682
1 : 5 M	(2.3)	334	(3.8)	**551**	(4.5)	653	(2.5)	363	(4.0)	**580**	(4.9)	711

Table 3.5.2 Tensile strength, 3.15 inch slump

Mixture proportion	$f_{ctk,min}$		Age (63 days)		$f_{ctk,max}$		$f_{ctk,min}$		Age (91 days)		$f_{ctk,max}$	
	(MPa)	psi	(Mpa)	**psi**	(Mpa)	psi	(Mpa)	psi	(Mpa)	**psi**	(Mpa)	Psi
1 : 3 R	(2.9)	421	(4.4)	**638**	(5.7)	827	(2.7)	392	(4.5)	**653**	(5.2)	754
1 : 3 M	(3.0)	435	(4.9)	**711**	(5.9)	856	(3.2)	464	(5.5)	**798**	(6.2)	899
1 : 4 R	(2.9)	421	(4.1)	**595**	(5.7)	827	(2.9)	421	(4.3)	**624**	(5.6)	812
1 : 4 M	(2.8)	406	(4.3)	**624**	(5.4)	783	(2.9)	421	(4.7)	**682**	(5.6)	812
1 : 5 R	(2.7)	392	(3.9)	**566**	(5.2)	754	(2.8)	406	(4.1)	**595**	(5.5)	798
1 : 5 M	(2.9)	421	(4.1)	**595**	(5.7)	827	(2.9)	421	(4.5)	**653**	(5.7)	827

The tensile strength increases with the time as it can be noticed in Table 3.5.1 comparing the gotten values for 7 and 91 days on the same mixture proportion. That rising of values is justified by the fact that the cement hydration and the pozzolanic reactions are occurring with time. The ***MetacaulimHP*** plus water and calcium hydroxide $Ca(OH)_2$ provided by the cement hydration forms a new composition increasing the internal bindings of the concrete.

This increasing of tensile strength nor always follows the same ratio of growth of the compressive strength. The different speed of increasing is due not only to the cure of the concrete but also to factors related to the mixture of the concrete such as type of aggregates, admixture used and internal factors as microcracking.

3.6 Modulus of Elasticity

Forecast model according with the American Concrete Institute ACI 318

- item 8.5:

$E_c = 4733 \bullet f'_c{}^{0,5}$ (MPa)

it corresponds to tension about $0{,}45f'_c$.

The results gotten for the modulus of elasticity consist of Tables 3.6.1 and 3.6.2 where also it can be seen the forecast of modulus of elasticity calculated in accordance with the model presented above and the values of compression strength (f'_c) gotten in this study ($f'_c = f_{c,average}$ - 5 MPa).

It can be observed that the concrete with ***MetacaulimHP*** present higher values of modules than that of the concrete of reference, without addition. Where the ***MetacaulimHP*** substitutes the cement it still can be noticed that the modules of the concrete presents a higher growth percentile compared with the reference concrete. That happens because in the reference concrete, with the cement ASTM C 150 type III, the reaction of hydration occurs in the first days and the concrete quickly reaches most of its resistance. In concrete with ***MetacaulimHP*** the pozzolanic reactions continue after the initial ages.

3.7 Chloride Ion Penetration

The durability of the concrete has been more and more a concern of the engineers and of all the society due to economic reasons. The costs of the repairs have reached platforms so high that it has been concluded the significance in the improvement of the quality of the concrete related to the durability. On the other hand it exists an ambient and ecological question about the conservation of natural resources, as Freyermuth (Life-Cycle Cost Analysis. Concrete International, ACI, v. 23, n. 2, Feb. 2001. p.89-95) and Kumar Mehta (Reducing the Environmental Impact of Concrete. Concrete International. ACI, v.23, n. 10, Oct. 2001. p.61-66), besides the concrete ones have been used in environments too much hostile as maritime platforms and in diverse types of chemical and nuclear industries.

Table 3.6.1 Static modulus of elasticity, 3.15 inch slump.

	7 days			28 days			91 days		
Mix proportion	**GPa**	**10^6 psi**	**ASTM C 469 *10^6 psi***	**GPa**	**10^6 psi**	**ASTM C 469 *10^6 psi***	**GPa**	**10^6 psi**	**ASTM C 469 *10^6 psi***
1 : 3 R	37.6	**5.5**	5.9	40.8	**5.9**	6.1	41.7	**6.0**	6.2
1 : 3 M	39.4	**5.7**	6.0	43.0	**6.2**	6.3	44.8	**6.5**	6.4
1 : 4 R	33.1	**4.8**	5.5	35.7	**5.2**	5.7	38.6	**5.6**	5.9
1 : 4 M	34.5	**5.0**	5.6	37.6	**5.5**	5.9	39.6	**5.7**	6.0
1 : 5 R	27.9	**4.0**	5.1	29.8	**4.3**	5.2	30.8	**4.5**	5.3
1 : 5 M	29.7	**4.3**	5.2	32.4	**4.7**	5.5	33.5	**4.9**	5.5

The analysis of the durability involves not only the control of the contact between the aggressive medium with the concrete that it is responsible for many physical and chemical processes of degradation but also the control of its porosity through which the aggressive agents cannot penetrate into the concrete.

One of the most aggressive agents for concrete are ions chlorides that can appear mainly in seawater, coastal areas, industrial atmospheres, chemical treated water reservoirs, swimming pools, treatments in industries and others.

When the relation Cl/OH increase can cause the destruction of the protective film that is naturally formed on the steel surface of the reinforcement. When this occurs exists great possibility of corrosion of the steel. Also, when large amounts of chlorides are founded in concrete it tends to conserve more humidity that increases the risk of corrosion with the consequent reduction of the electric resistivity of the concrete.

The determination of the resistance to the penetration of ions chlorides was made following the Method ASTM C1202, at 28, 63 and 91 days of age. The results are showed in Table 3.7.1.

Table 3.7.1 Resistance to ions chlorides penetration, 3.15 inch slump, at 28, 63 and 91days.

mix	**28 days**		**63 days**		**91 days**	
proportion	**electrical charge (Coulombs)**	**resistance to ions chlorides penetration**	**electrical charge (Coulombs)**	**resistance to ions chlorides penetration**	**electrical charge (Coulombs)**	**resistance to ions chlorides penetration**
1 : 3 R	2072	moderate	1860	moderate	1304	high
1 : 3 M	**764**	**very high**	**809**	**very high**	**632**	**very high**
1 : 4 R	2163	moderate	1897	High	1522	high
1 : 4 M	**943**	**very high**	**721**	**very high**	**705**	**very high**
1 : 5 R	2282	moderate	2073	High	1796	high
1 : 5 M	**998**	**very high**	**865**	**very high**	**736**	**very high**

According to ASTM C 1202 the concrete ones with electrical charge between 100 and 1000 Coulombs are those one with very high resistance to the chloride penetration, that is, concrete with very low probability to occur corrosion. In the band that comprises the values of electrical charge between 1000 C and 2000 C are the concrete ones with high resistance to the chloride penetration and between 2000 C and 4000 C are the concrete ones with moderate resistance to the chloride penetration. Above of the value of 4000 Coulombs are the concrete ones of low resistance to the chloride penetration.

It is noticed, in Table 3.7.1, that the concrete with ***MetacaulimHP*** have very high resistance to the chloride penetration resulting that they are durable and recommendable for applications in aggressive environments with chloride predominance. It is also observed that the concrete ones of reference (R), with same materials however without the addition of ***MetacaulimHP***, had presented only moderate resistance to the chloride ion penetration, dissuading them for environments with chlorides.

3.8 Density, Absorption and Voids

This is an indirect way to measure the resistance of the concrete to the penetration of aggressive agents in submerged situations or permanently saturated and humid.

The determination of the absorption of water by immersion, density and voids was carried using the Method ASTM C 642, at 28 days of age. The results are showed in Table 3.8.1.

Table 3.8.1 Absorption, density and voids, 3.15 inch slump

Mix proportion	absorption (%)	density		voids (%)	concrete classification
		kg/m^3	**lb/ft^3**		
1 : 3 R	5.1	2414	150.7	12.5	normal
1 : 3 M	4.0	2420	**151.1**	9.6	**durable**
1 : 4 R	5.7	2410	150.5	13.7	normal
1 : 4 M	4.4	2407	**150.3**	10.5	**durable**
1 : 5 R	6.1	2408	150.3	14.5	deficient
1 : 5 M	5.2	2400	**149.8**	12.2	**normal**

It is clearly observed that the concrete with addition of ***MetacaulimHP*** can be considered a durable while the concrete without addition presents higher values of water absorption resulting therefore lesser service life in aggressive and humid environments.

3.9 Electrical Resistivity

The corrosion of the steel used in concrete structures is an electrochemical process that occurs in the electrolyte formed by the water solution present in the concrete's voids. Thus, the steel corrosion rates is directly related to the conductivity (inverse of resistivity) of the concrete that, in turn, depends on the present humidity and on the present amount of chlorides.

Once the chemical protective film over the surface of steel reinforcement concrete, has been destroyed, that is, has occurred the depassivation of the reinforcement, the corrosion rate will be controlled mainly by the electric resistivity and the availability of oxygen.

Electric resistivity controls the flow of ions that are spread out in the concrete through the water solution in the voids. Its measure is a basic and decisive parameter in the control of the rate of the corrosion reaction and can be made through the adaptation of the ASTM G 57 Standard Method for Field

Measurement of Soil Resistivity Using the Wenner Four-Electrode Method. The results are showed in Table 3.9.1.

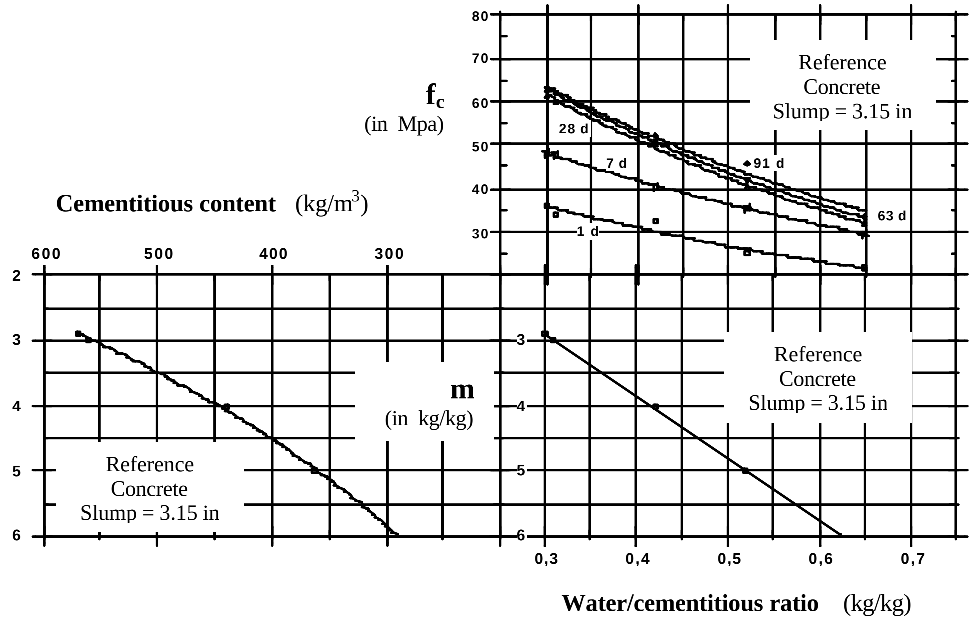
Table 3.9.1 Electrical/ionic resistivity , 3.15 inch slump concrete**.**

	28 days	**63 days**	**91 days**
Mix proportion	**(10^3 W.cm)**	**(10^3 W.cm)**	**(10^3 W.cm)**
1 : 3 R	30.7	36.5	37.7
1 : 3 M	72.4	92.4	136.8
1 : 4 R	30.5	34.8	34.3
1 : 4 M	71.3	76.7	81.3
1 : 5 R	26.7	31.5	44.7
1 : 5 M	62.2	71.2	74.5

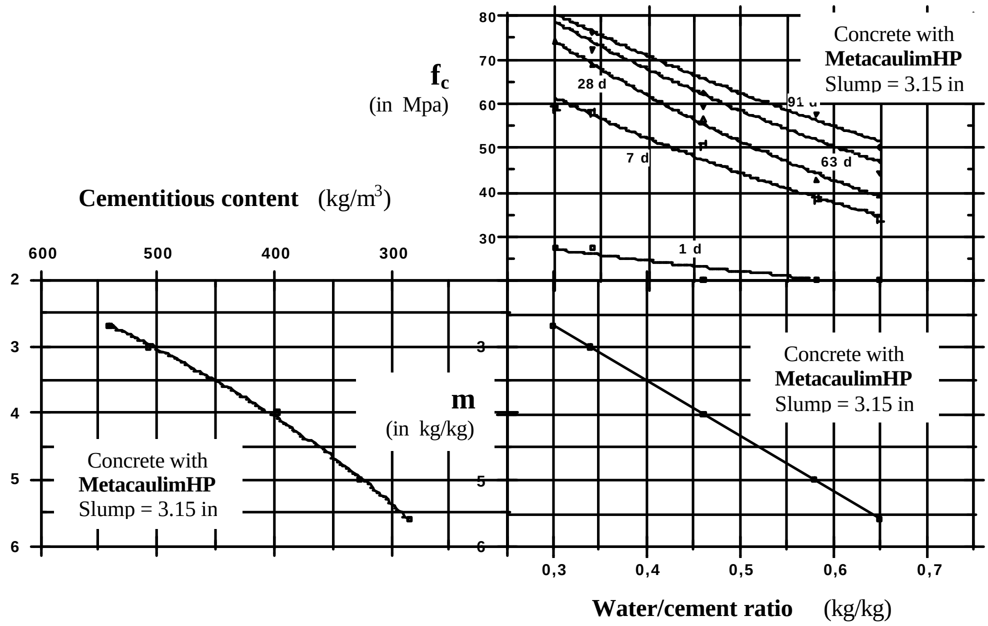
The fib (CEB-FIP), in its Bulletin of Information n. 192, establishes that for values of resistivity superior to 60 kΩ.cm the expected corrosion rate can be considered worthless. Observing the results presented in Table 3.9.1 it can be noticed that all the concrete with addition of ***MetacaulimHP*** can be considered of high resistivity concrete and therefore they are protective to the reinforcement when these are in aggressive environments. Also under this aspect, they can be considered durable concrete, while the concrete ones without addition of ***MetacaulimHP*** cannot be considered durable concrete strucure.

4 ANALYSIS OF THE RESULTS

4.1 Mix Design Nomogram → Reference concrete, without addition



4.2 Mix Design Nomogram → Concrete with *MetacaulimHP*



4.3 Mix Design Nomogram - Equations

Abrams' Law → Compressive Strength (MPa)

reference	MetacaulimHP
$f_{c,1day} = \frac{55}{4,3^{a/c}}$	$f_{c,1day} = \frac{41}{3,7^{a/c}}$
$f_{c,7days} = \frac{73}{4,1^{a/c}}$	$f_{c,7days} = \frac{107}{5,6^{a/c}}$
$f_{c,28days} = \frac{108}{6,6^{a/c}}$	$f_{c,28days} = \frac{137}{7,2^{a/c}}$
$f_{c,63days} = \frac{109}{6,3^{a/c}}$	$f_{c,63days} = \frac{97}{2,5^{a/c}}$
$f_{c,91days} = \frac{97}{4,3^{a/c}}$	$f_{c,91days} = \frac{104}{2,6^{a/c}}$

Lyse's Law → Water by cubic meter → Equations

$$m = 0,035 + 9,52 \cdot a/c$$
reference

$$m = 0,167 + 8,33 \cdot a/c$$
MetacaulimHP

Molinari's Law → Cement Content → Equations (kg/m³)

$$C = \frac{1000}{0,33 + 0,48 \cdot m}$$
reference

$$C = \frac{1000}{0,35 + 0,54 \cdot m}$$
MetacaulimHP

4.4 Compressive Strength → *MetacaulimHP* Advantage

Quantitative analysis using the equations and the Mix Design Nomogram.

Table 4.4.1 Approximate relationship between average compressive strength and water/cementitious ratio, at 28 days of age.

water/cementitious ratio	reference		*MetacaulimHP*		*MetacaulimHP/ reference %*
	MPa	**psi**	MPa	**psi**	
0.35	56	**8122**	69	**10008**	123
0.40	51	**7397**	62	**8992**	122
0.45	46	**6672**	56	**8122**	122
0.50	42	**6092**	51	**7397**	121
0.55	38	**5511**	46	**6672**	121
0.60	35	**5076**	42	**6092**	120
0.65	32	**4641**	38	**5511**	119

Table 4.4.2 Approximate relationship between average compressive strength and cementitious content, at 28 days of age.

cementitious content		reference		*MetacaulimHP*		*MetacaulimHP/ reference %*
kg/m^3	$\mathbf{lb/ft^3}$	MPa	**psi**	MPa	**Psi**	
300	18.7	31	**4496**	38	**5511**	122
350	21.8	38	**5511**	47	**6817**	124
400	25.0	44	**6382**	55	**7977**	125
450	28.1	50	**7252**	63	**9137**	126
500	31.2	55	**7977**	69	**10008**	127
550	34.3	59	**8557**	75	**10878**	127

It is noticed that for a given cementitious content the concrete with ***MetacaulimHP*** present higher compressive strength comparatively to the reference concrete.

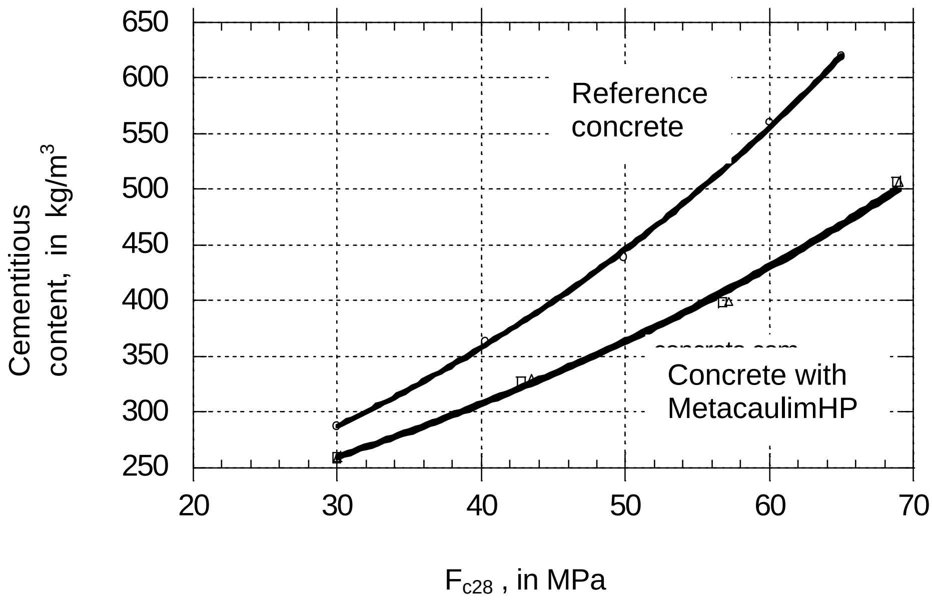


Figure 4.4.1 Savings that can be obtained by adding the ***MetacaulimHP*** in concrete, f_{c28}.

For example, a concrete with f'_c = 9,427 psi (65 MPa) with the addition of the ***MetacaulimHP*** can be obtained saving 30% in cement content compared to the reference concrete. Even for a f'_c = 4,351 psi (30 MPa) the cement economy can reach more than 10%.

As it is presented in Table 4.4.3, for any characteristic compression strength it is observed that the addition of ***MetacaulimHP*** leads to an economy of cement. By the way the cement economy does not only represent economy of money but it represents lesser risk of thermal movements on account of cement heat of hydration, minor risk of chloride ion penetration, larger durability, higher modulus of elasticity and minor deformations for loads. Also contribute to save the mineral resources preserving the environment and contributing to the sustainable development.

Table 4.4.3 Cementitious content considering same compressive strength

Compressive strength		**reference cement**		***MetacaulimHP Agglomeratives***		***MetacaulimHP cement***		***MetacaulimHP 8%substitution***	
Mpa	**psi**	kg/m^3	**lb/ft^3**	kg/m^3	**lb/ft^3**	kg/m^3	**lb/ft^3**	kg/m^3	**lb/ft^3**
30	**4351**	321	**20.0**	283	**17.7**	260	**16.2**	23	**1.4**
35	**5076**	358	**22.3**	306	**19.1**	281	**17.5**	25	**1.6**
40	**5802**	400	**25.0**	334	**20.9**	307	**19.2**	27	**1.7**
45	**6527**	446	**27.8**	363	**22.7**	334	**20.9**	29	**1.8**
50	**7252**	498	**31.1**	395	**24.7**	363	**22.7**	32	**2.0**
60	**8702**	620	**38.7**	467	**29.2**	429	**26.8**	38	**2.4**

4.5 Splitting Tensile Strength

The Fig. 4.5.1 is based on the data of Table 3.5.1 and shows clearly the positive influence of the addition of the ***MetacaulimHP*** by increasing the splitting tensile strength in Portland-cement concrete.

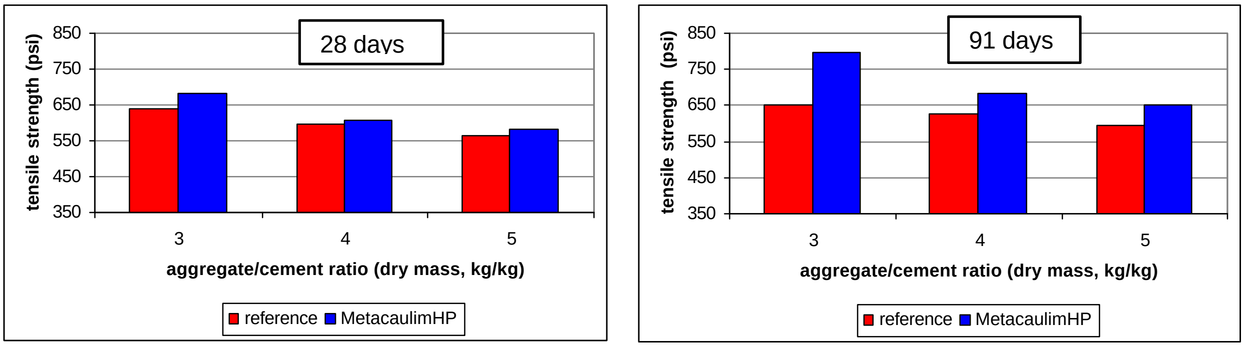


Figure 4.5.1 Splitting tensile strength at 28 and 91 days.

In Fig. 4.5.2 it is presented the evolution of the splitting tensile strength for the different mixture proportions. In that way it can be observed again the positive action of the addition of MetacaulimHP.

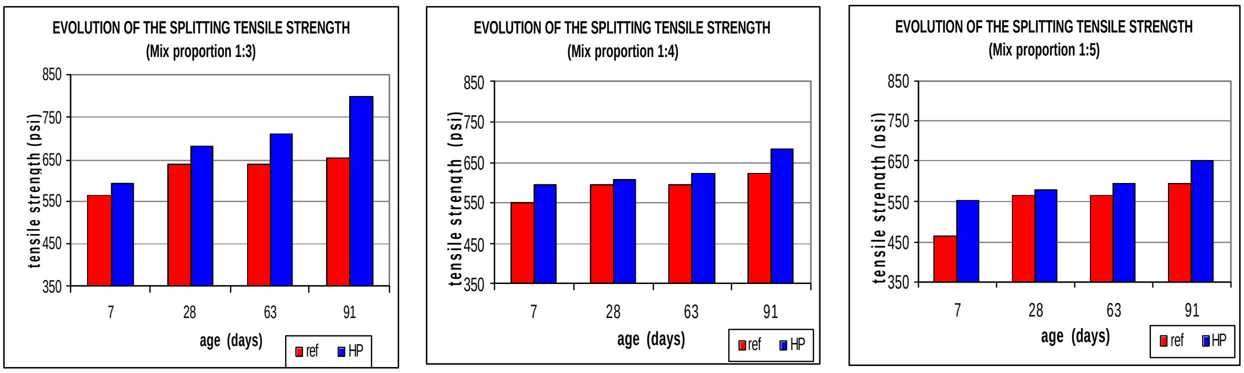


Figure 4.5.2 Evolution of the splitting tensile strength for different mixture proportions

4.6 Modulus of elasticity

The Fig. 4.6.1 was constructed based on the data of Table 3.6.1 and it shows clearly the positive influence of the addition of ***MetacaulimHP*** in the increase of the modulus of elasticity of the Portland-cement concrete.

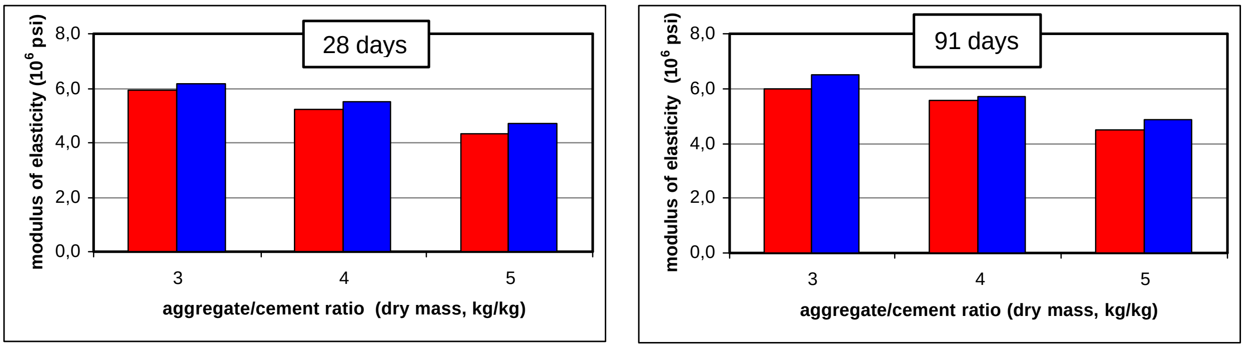


Figure 4.6.1 Modulus of elasticity at 28 and 91 days.

In Fig. 4.6.2 it is presented the evolution of the modulus of elasticity for different mixture proportions and at different ages. In all of them it is possible to verify the positive action when the ***MetacaulimHP*** is added.

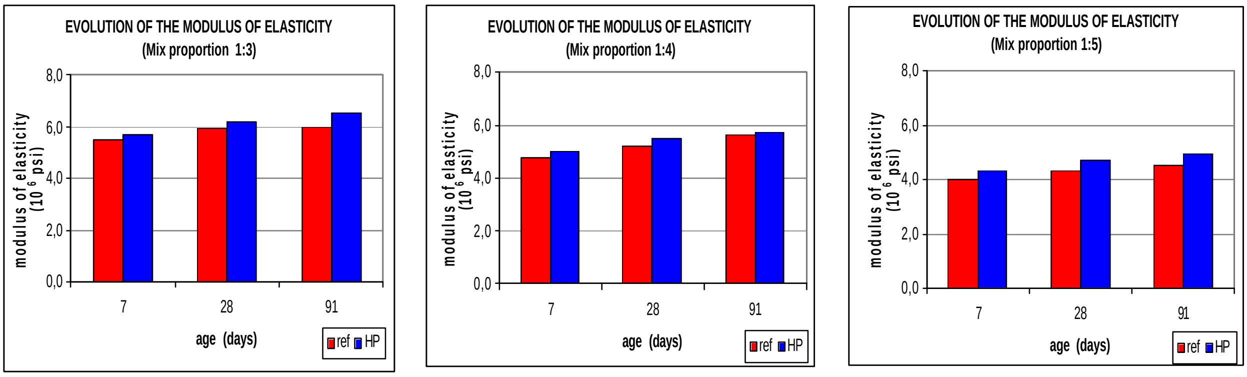


Figure 4.6.2 Evolution of the modulus of elasticity for different mixture proportions and ages.

4.7 Chloride Ion Penetration

The Fig. 4.6.1 was constructed based on the data of Table 3.6.1 and it shows clearly the positive influence of the addition of ***MetacaulimHP*** in the increase of the resistance to chloride ion penetration in Portland-cement concrete.

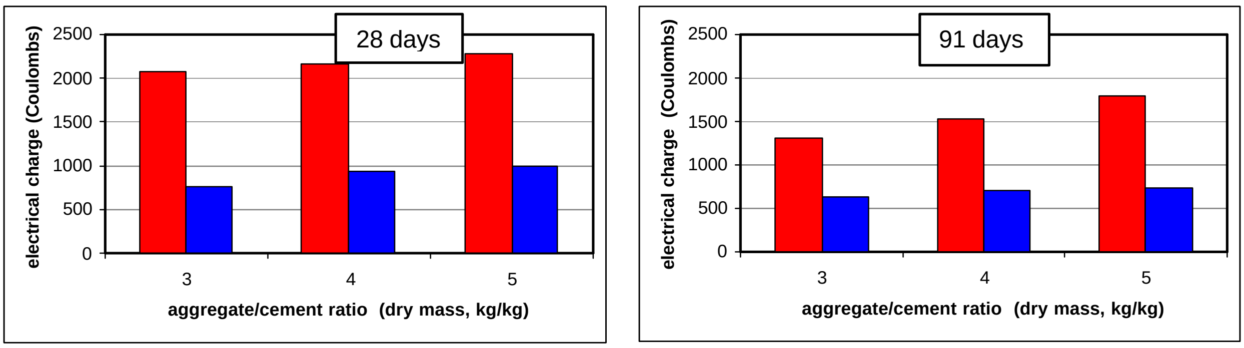


Figure 4.7.1 Resistance to the chloride ion penetration at 28 and 91 days of age.

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Departamento de Engenharia de Construção Civil

In Fig. 4.7.2 it is presented the evolution of the resistance to the chloride ion penetration for different mixture proportions at different ages. It can be observed again the positive results obtained when the ***MetacaulimHP*** is added to the Portland-cement concrete.

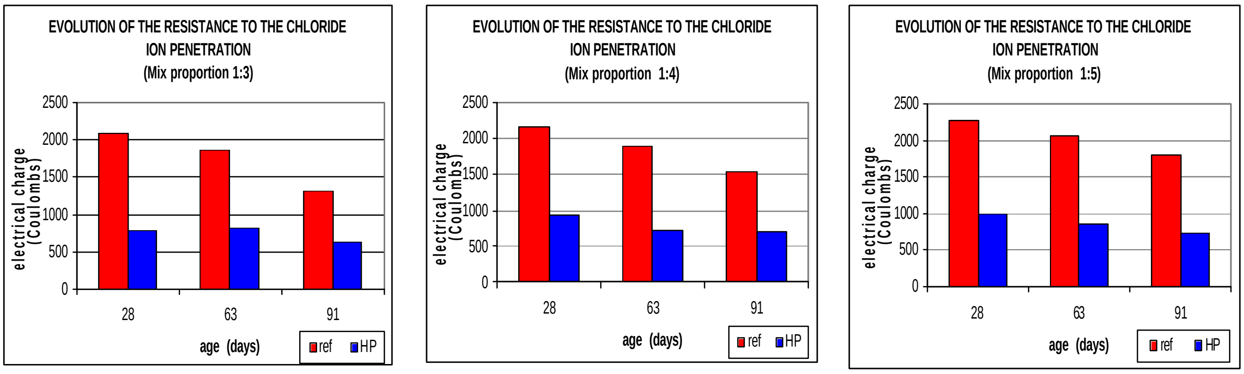

Figure 4.7.2 Evolution of the resistance to the chloride ion penetration

4.8 Absorption, Density and Voids

Based on the results of Table 3.8.1 it was constructed the Figures 4.8.1 and 4.8.2 through what it can be seen the positive influence of the addition of ***MetacaulimHP*** to the reduction of the water absorption after immersion and of the volume of permeable pore space in Portland-cement concrete.

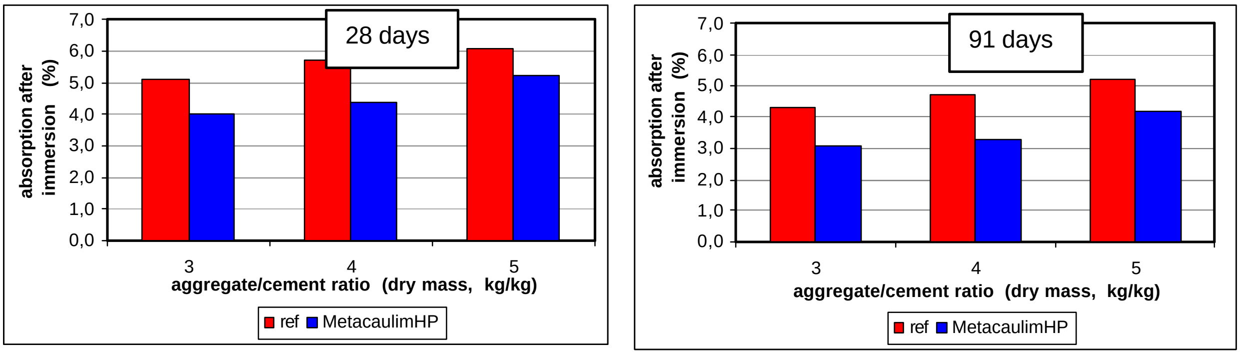

Figure 4.8.1 Water absorption after immersion at 28 and 91 days.

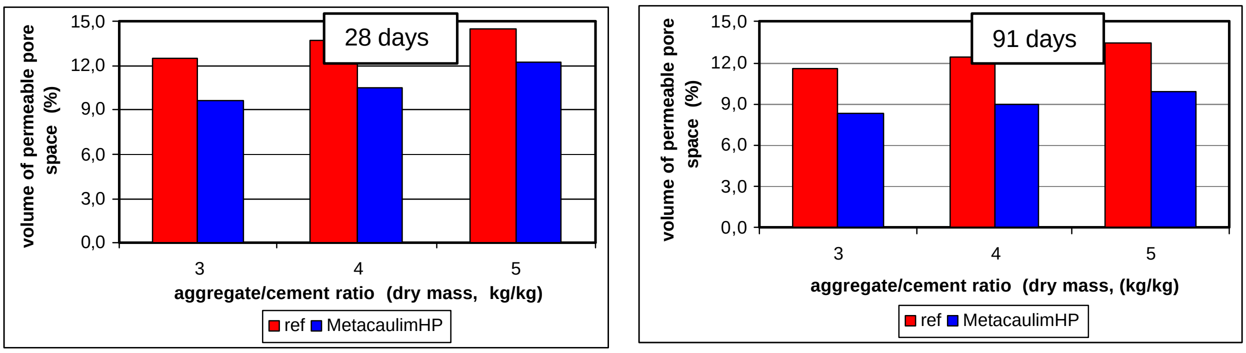

Figure 4.8.2 Volume of permeable pore space at 28 and 91 days.

In Fig. 4.8.3 it is presented evolution of the water absorption after immersion and in Fig. 4.8.4 the evolution of the volume of permeable pore space for different mixture proportions and ages having been able itself to observe again the positive action of the addition of ***MetacaulimHP***

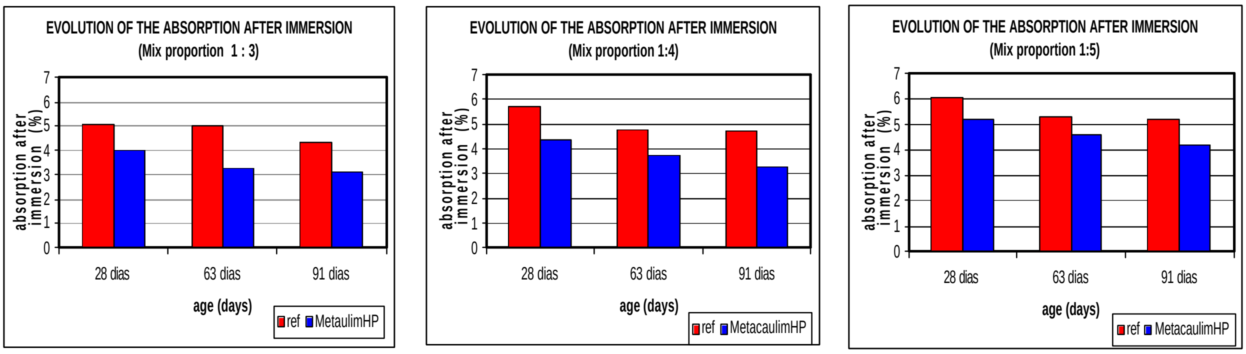

Figure 4.8.3 Evolution of the water absorption after immersion for different mixture proportions and ages.

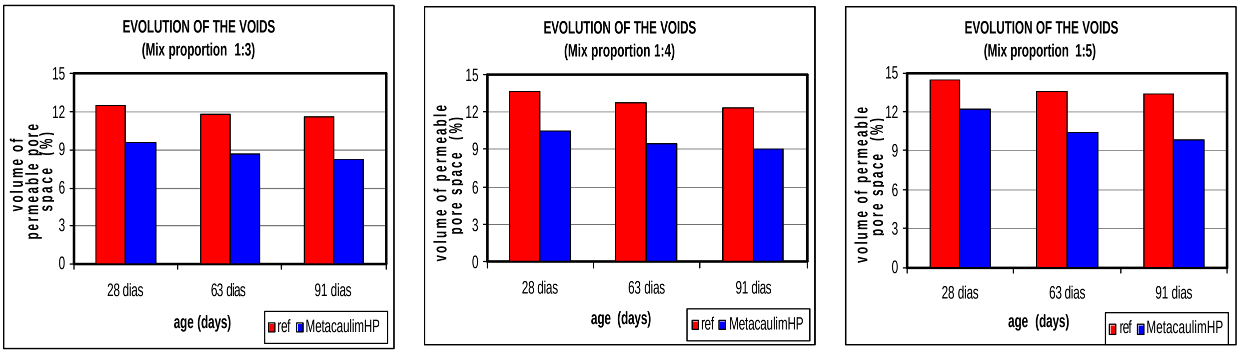

Figure 4.8.4 Evolution of the volume of permeable pore space for different mixture proportions and ages

4.9 Electrical resistivity

The Fig. 4.9.1 was constructed based on the data of Table 3.9.1 and it shows clearly the positive influence of the addition of ***MetacaulimHP*** increasing the electrical resisitivity of Portland-cement concrete.

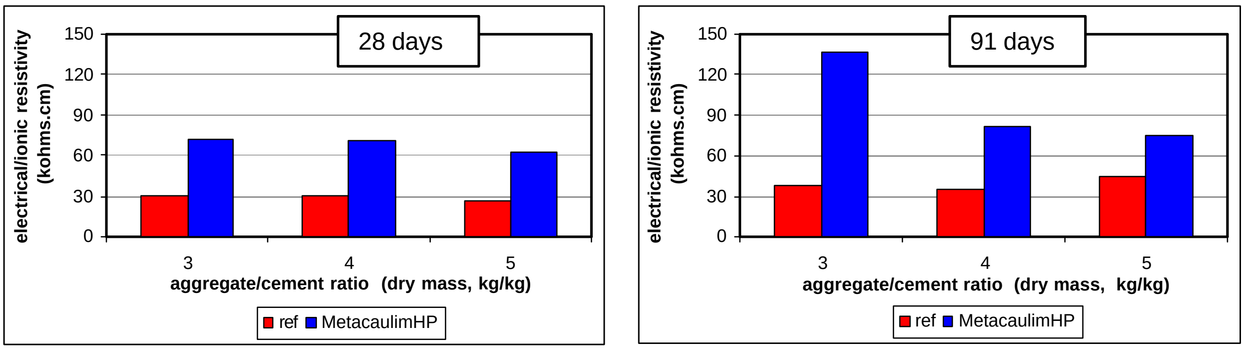

Figure 4.9.1 Electrical/ionic resistivity of the Portland-cement concrete at 28 and 91 days.

In Fig. 4.6.2 it is presented the evolution of the ionic/electrical resistivity for different mixture proportions and at different ages. In all of them it is possible to verify the positive action when the ***MetacaulimHP*** is added to Portland-cement concrete.

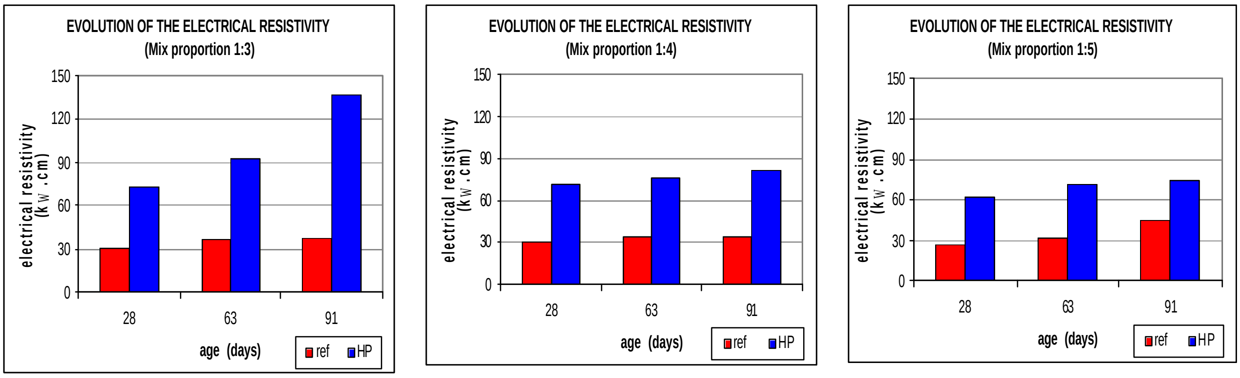

Figure 4.9.2 Evolution of the electrical/ionic resistivity in Portland-cement concrete for different mixture proportions and at different ages

5. FINAL CONCLUSIONS

The results obtained are promising and indicating that the ***MetacaulimHP*** represents an interesting option in the search of the improvement of the quality, the economy and the durability of the Portland-cement concrete. The positive action can be seen, for example, through the values presented in Table 5.1 where some concrete properties are shown with its respective values necessary to reach specific compression strength at 28 days.

Table 5.1 Properties of the Portland-cement concrete for the same compression strength, at 28 days, 3.15 in slump.

Properties	MPa	30	35	40	45	50	60
	psi	**4,351**	**5,076**	**5,802**	**6,527**	**7,252**	**8,702**
Cement content (lb/ft^3)	Reference	20.0	22.4	25.0	27.8	31.1	38.7
	MetacaulimHP	**16.2**	**17.5**	**19.2**	**20.9**	**22.7**	**26.8**
Resistance to ion chloride penetration (Coulombs)	Reference	2146	2182	2214	2243	2268	2314
	MetacaulimHP	**1079**	**1036**	**997**	**963**	**932**	**877**
Water absorption (%)	Reference	6,5	6,2	5,9	5,6	5,4	4,9
	MetacaulimHP	**5,8**	**5,5**	**5,2**	**4,9**	**4,7**	**4,3**
Electrical/ionic resistivity (10^3W.cm)	Reference	25.9	27.2	28.3	29.4	30.4	32.2
	MetacaulimHP	**58.9**	**61.1**	**63.1**	**65.0**	**66.7**	**69.9**

São Paulo, October, 30th 2002.

Paulo Helene   Charles Siervi Lacerda