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Efeito da adição de metacaulim na absorção capilar e carbonatação do concreto

Effect of metakaolin addition on capillary absorption and carbonation of concrete

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RESUMO

Este estudo avalia os efeitos da adição de metacaulim (MK) ao concreto com resistência à compressão de 30 MPa e 40 MPa, na absorção capilar e na profundidade da carbonatação. Para o teste de absorção capilar, as amostras foram secas no forno, pesadas, imersas em água por períodos de 1, 2, 3 e 24 horas. Após cada período, as amostras foram secas e pesadas, e esses valores foram utilizados para se obter a curva Abrams. O teste de carbonatação foi realizado após curar as amostras em uma câmara úmida, padronizando a umidade por duas horas e permanecendo na câmara de carbonatação por 30 dias com 5% de CO_2 a 23 ± 2 °C. Após este período, as amostras foram cortadas ao meio, pulverizadas com uma solução de fenolftaleína e uma análise de imagem foi feita. Para as amostras preparadas com cimento CPIII com um ou dois agregados, a adição de MK aumentou a absorção capilar. Para as amostras fabricadas com cimento CPV, a amostra sem adição de MK mostrou maior absorção capilar. O menor valor de profundidade de carbonatação foi observado para amostras fabricadas com cimento CPV e dois agregados. A maior profundidade de carbonatação foi observada para a amostra com cimento CPIII, pedra triturada 1, com adição de MK.

Palavras-chave: matacaulim, concreto, absorção capilar, carbonatação

ABSTRACT

This study evaluates the effects of metakaolin (MK) addition to concrete with compressive strength of 30MPa and 40MPa, on capillary absorption and depth of carbonation. For the capillary absorption test, the samples were oven dried, weighed, immersed in water for periods of 1, 2, 3 and 24 hours. After each period, the samples were dried and weighed, and these values were used to obtain the Abrams curve. The carbonation test was carried out after curing the specimens in a moist chamber, standardizing the humidity for two hours and remaining in the carbonation chamber for 30 days with 5% CO_2 at 23±2°C. After this period, the specimens were cut in half, sprayed with a solution of phenolphthalein and an image analysis was performed. For samples prepared with CPIII cement with one or two aggregates, the MK addition increased the capillary absorption. For the samples manufactured with CPV cement, the sample without MK addition showed a higher capillary absorption. The lowest value of carbonation depth was observed for samples manufactured with CPV cement and two aggregates. The highest carbonation depth was observed for the sample with CPIII cement, crushed stone 1, with metakaolin addition.

Keywords: metakaolin, concrete, capillary absorption, carbonation

1. INTRODUCTION

With the increasing need to reduce the liability of solid waste, and to reduce the environmental impact of the extraction of materials for the manufacture of concrete, the use of mineral additions in cement or pozzolanic concrete is increasing to improve performance characteristics such as volume and porous size, permeability

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and durability [1-7]. The utilization of calcined clay, in the form of metakaolin (MK), as a pozzolanic material for mortar and concrete has received considerable attention in recent years. This interest is part of the widely spread attention directed towards the utilization of wastes and industrial by-products in order to minimize Portland cement (PC) consumption, the manufacture of which being environmentally damaging. Mortar and concrete, which contain pozzolanic materials, exhibit considerable enhancement in durability properties [8-26]. Literature demonstrates that MK is an effective pozzolan which causes significant improvement in the pore structure and hence the resistance of the concrete to the action of harmful solutions [17].

Metakaolin improves the engineering properties of concrete because of its double effect on cementitious matrix (filler effect and pozzolanic properties). Moreover, MK can reduce impact of concrete on the environment due to its lower carbon dioxide emission than clinker. The development of a slurry form of MK opens new fields of investigations, such as its incorporation into self-compacting concrete (SCC) [8].

Kou *et al* [26] reported that MK contributed to both the short and long-term properties of concrete. As far as compressive strength is concerned, the replacement of cement by 15% of MK improved both the mechanical and durability performance. MK contributes to the short and long-term properties of concrete because the calcium hydroxide of cement reacts with the silica of the MK producing a more resistant compound [26].

Batis *et al* [15] concluded that metakaolin improves the compressive strength and the 10 wt.% addition shows the optimum contribution to the strength development. In addition, the use of MK, either as a sand replacement up to 20 wt.% , or as a cement replacement up to 10 wt.%, improves the corrosion behavior of mortar specimens, while when MK is added in greater percentages there is no positive effect [15].

Metakaolin is a valuable admixture for concrete/cement applications that can enhance the performance of cementitious composites through high pozzolanic reactivity such as silica fume (SF). While SF concrete is characterized by superior mechanical and durability performance, concrete containing MK achieves comparable properties at a lower price and with better workability. Highly durable self-compacting concrete (SCC) mixtures can be produced using a high MK content with an optimum percentage of 20 wt.% [9]. Results have also shown that the durability of SCC, especially with high MK content, is higher than that of SCC containing SF [9].

Badogrannis and Tsivilis [12] reported that MK concrete exhibits a lower chloride permeability, gas permeability and sorptivity. The addition of metakaolin refines the pore system of concrete, leading to a decreased mean pore size and improved uniformity of the pore size distribution [12].

Badogiannis et al [13] concluded that MK has a very positive effect on cement strength after 2 days and specifically at 28 and 180 days. The blended cements demand significantly more water than the relatively pure cement [13]. With the addition of MK to concrete, the pozzolanic reaction is accelerated between 7 and 28 days, accompanied by a steep decrease of $Ca(OH)_2$ content [13]. Finally, it is concluded that a 10 wt.% metakaolin content seems to be, generally, more favorable than 20 wt.% [13].

This study evaluates the effects of the addition of metakaolin to concrete on capillary absorption and depth of carbonation. Two types of concrete were used, CPV and CPIII cement, with different particle size, and two types of aggregates were utilized, generating a study which was not found in literature. This study, as already mentioned, has academic and technological importance, and contributes to the development of a durable and environmentally friendly product.

2. MATERIALS AND METHODS

The capillary absorption and depth of carbonation were measured in concrete with a compression strength of 30MPa and 40MPa, which are widely used in civil works of medium and large size.

The samples were differentiated by the type of coarse aggregate (crushed rock 0 and 1) and the presence of metakaolin addition, plus two types of cement (CPIII and CPV), as shown in Table 1.

Badogiannis *et al* [13] and Batis *et al* [15] showed that an optimal content of metakaolin addition was 10 wt.% substituting the mass of cement. In this study, the content of 8 wt.% MK addition was used according to literature [13,15]. This content is usually adopted in industrial practice.

Table 1: Chemical composition of concrete samples.

SAMPLE	COMPRESSION STRENGTH (MPA)	CEMENT	TRACE	WATER/BINDER RATIO
1	30	CPIII	1:2.72:2.69 cement:sand:crushed rock 1	0.50
2	30	CPIII	1:0.08:2.72:2.69 cement:metakaolin:sand:crushed rock 1	0.48
4	30	CPV	1:2.72:2,69 cement:sand:crushed rock 1	0.40
5	30	CPV	1:0,08:2,72:2,69 cement:metakaolin:sand:crushed rock 1	0.47
7	40	CPIII	1:2.48:2.3:0.3 cement:sand:crushed rock 1;crushed rock 0	0.48
8	40	CPIII	1:0.08:2.48:2.72:0.3 cement:metakaolin:sand:crushed rock 1;crushed rock 0	0.47
10	40	CPV	1:2.48:2.3:0.3 cement:sand:crushed rock 1;crushed rock 0	0.48
11	40	CPV	1:0.08:2.48:2.72:0.3 cement:metakaolin:sand:crushed rock 1;crushed rock 0	0.40

The CPIII cement has an addition of blast furnace slag and presents its compression strength at 28 days. The CPV cement is used for high compression strength mainly for concrete requiring rapid unmolding, and initial resistance at approximately 7 days. The technical characteristics of the cement used are shown in Table 2, supplied by the manufacturer. The high performance plasticizer additive Viscocrete Sika was used. The additive acts by different mechanisms, through the effects of surface absorption and steric separation of the cement particles and in the cement hydration process. It acts as a water reducer, increasing compression strength, and reducing the rate of carbonation.

Table 2: Physical and chemical properties of CPIII and CPV cement.

PHYSICAL TESTS	CPIII-32 cement	CPV -ARI
Concrete Inicial Setting Time (h:min)	≥1	≥1
Concrete Final Setting Time (h:min)	≤10	≤10
Fineness of the 200≠ sieve (%)	≤12.0	≤6.0
Le Chatelier Soundness (mm)	≤5.0	≤5.0
Compression Strength (MPa) 1 day		≥14
Compression Strength (MPa) 3 days	10	≥24
Compression Strength (MPa) 7 days	≥20	≥34
Compression Strength (MPa) 28 days	≥32	
CHEMICAL TESTS		
Ignition loss (%)	≤4.5	≤4.5
Insoluble residue	≤1.5	≤1.0
SO_3 (%)	≤4.0	≤3.5
MgO (%)		≤6.5

For the capillary absorption test, the samples were oven dried and weighed to obtain the initial weight. After this procedure, they were immersed in containers with blades 3 cm of water for periods of 1, 2, 3 and 24 hours. After each period, the samples were dried and weighed, and these values were used to obtain the curve:

$Abs = A / Bx$ (1)

where Abs is the value of capillary absorption equation, A and B are constants, and x is the value of compression strength. This equation is known as the Abrams equation.

The carbonation test was carried out after curing the specimens in a moist chamber. The samples were placed in an incubator to standardize the humidity for two hours and placed in the carbonation chamber for 30 days with 5% CO_2 at 23±2°C chamber. After this period, the specimens were cut in half, sprayed with a solution of phenolphthalein and the images were registered. The images were used in a design program, AutoCAD, where three measurements were taken on the four sides of the samples and put in a spreadsheet for removal of medium and the value of the depth of carbonation was determined for each sample. The method used is similar to that used in a previous research [27].

Figure 1 represents the main drawing program with measurements of a trait. The measurement (a) was taken 3 cm from the top of the specimen, the measurement (b) was taken in the middle of the specimen, and the measurement (c) was taken 3 cm from the bottom of the sample. The total number of measurements was six in each sample. The tests were performed in triplicate.

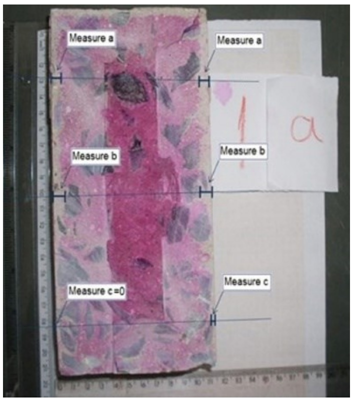

Figure 1: Measurement of carbonation depth.

3. RESULTS

Table 3 and Figures 2 to 5 show the results of capillary absorption for the samples studied.

Table 3: Capillary absorption and carbonation depth results.

TRACE	CAPILLARY ABSORPTION (G/M²)	CARBONATION DEPTH AFTER 30 DAYS (MM)
01	3204.45±160.20	0.44±0.14
02	3451.57±138.04	1.14±0.20
04	2776.62±83.28	0.08±0.01
05	2137.76±42.74	0.32±0.25
07	4777.62±95.54	0.16±0.19
08	6536.90±326.80	0.58±0.34
10	5334.78±213.36	0.06±0.04
11	2697.86±53.94	0.00±0.00

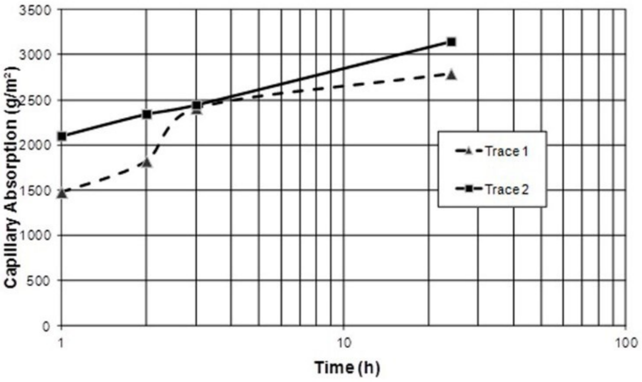

Figure 2: Capillary Absorption in function of time for traces 1 and 2.

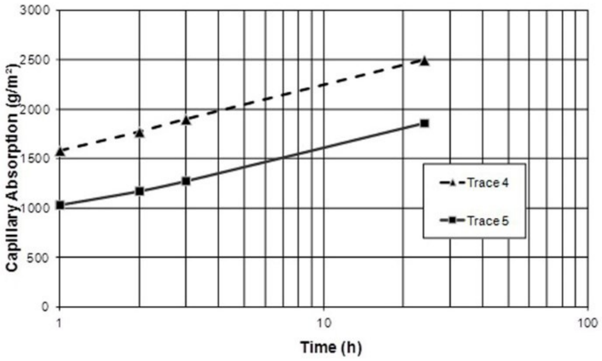

Figure 3: Capillary absorption versus time for traces 4 and 5.

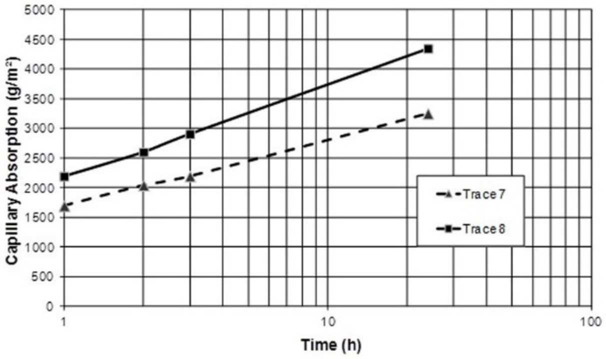

Figure 4: Capillary absorption versus time for traces 7 and 8.

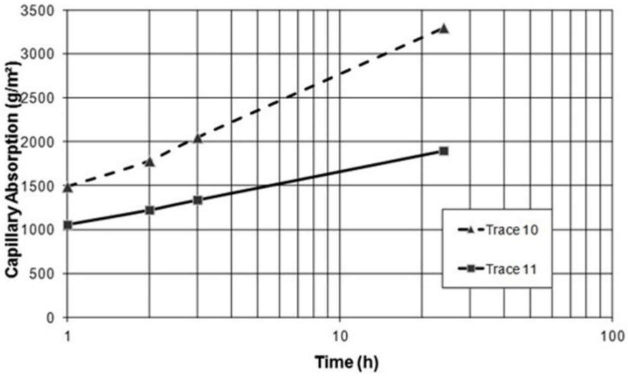

Figure 5: Capillary absorption versus time for traces 10 and 11.

Comparing samples 1 and 2, prepared with the same cement (CPIII) and crushed stone 1, sample 2 with MK addition showed a higher capillary absorption. For samples prepared with CPIII cement and crushed stone 0 and 1, sample 8 with MK addition also showed a higher capillary absorption.

In the case of Trace 1, without metakaolin addition, a higher rate of capillary absorption was identified between two and three hours of testing. After 3 hours of testing, the capillary absorption of the samples with and without metakaolin was the same. For times above 3 hours, the capillary absorption of the samples without metakaolin increased at a lower rate than the samples with MK. After 20 hours of testing, the capillary absorption of the concrete with MK addition was higher than the absorption of concrete without metakaolin.

For the samples manufactured with CPV cement, the sample without MK addition showed a higher capillary absorption, for the samples prepared with crushed stone 1, and with both crushed stones.

Table 3 and figures 6 and 7 show results of carbonation depth.

Figure 6: Carbonation depth of traces 1 (CPIII cement without MK), 2 (CPIII cement with MK), 4 (CPV cement without MK), and 5 (CPV cement with MK).

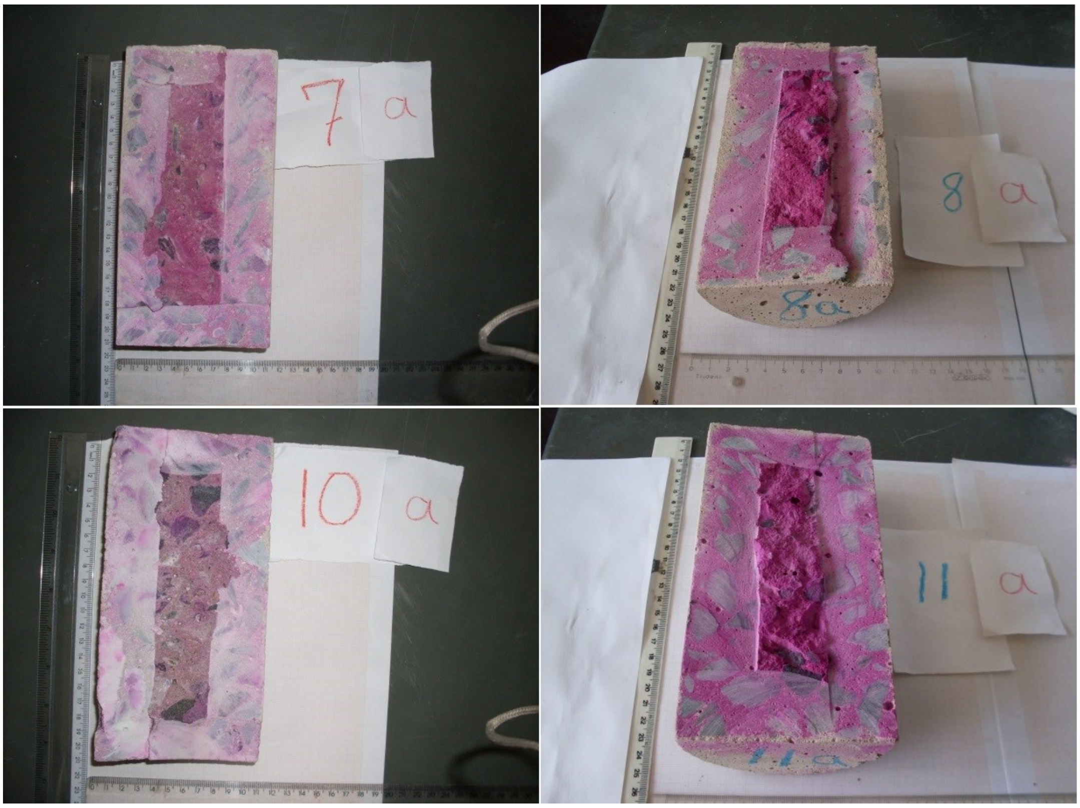

Figure 7: Carbonation depth of traces with two aggregates: 7 (CPIII cement, without MK), 8 (CPIII cement with MK), 10 (CPV cement without MK), and 11 (CPV cement with MK).

The highest carbonation depth was identified for the sample with CPIII cement, two types of aggregate, with MK addition. Changing only the type of cement, the use of cement CPV reduced the depth of carbonation due to the finer grinding of CPV cement.

The lowest value of carbonation depth was observed for samples 10 and 11, manufactured with CPV cement and two aggregates.

4. DISCUSSION

For samples with CPIII cement, the addition of metakaolin refines the pore system of concrete, leading to a decreased mean pore size and improved uniformity of the pore size distribution [12]. The refinement of pores generates an increase of capillary force, increasing the capillary absorption, as observed.

The CPV cement has the peculiarity to achieve high resistance in the first days of application. The development of high early strength is achieved by using the finer grinding of the cement (Table 2), so that in reaction with water, it acquires a higher resistance with a higher speed. The clinker is the same used for the manufacture of conventional cement, but remains in the mill for a longer time. The lower particle size of the CPV cement than the conventional cement generates a lower pore size, and the effect of MK of pore refinement become less significant. The addition of MK may even have clogged the fine pores of the concrete and caused the reduction of capillary absorption. Therefore, the cement CPV with MK addition showed a reduction in capillary absorption and is less susceptible to penetration of aggressive agents. Capillary absorption is directly related to the pore refinement, which occurs in samples of CPV cement. In addition, the pozzolanic reactions in CPV cement samples with MK addition occur faster than in samples of CPIII cement generating a higher compactness and hindering capillary absorption. The SiO_2 of MK reacts with the calcium hydroxide of cement generating a calcium silicate hydrate, stratlingite (C_2ASH_8), and tetra calcium aluminium hydrate (C_4AH_{13}) the main phases formed at ambient temperature. These hydraulic products alter the pore structure of the lime and cement pastes and greatly improving its resistance to the transport of water [28].

MK addition increased the carbonation depth. MK is a material produced by calcination of kaolinitic clays or special clays (kaolin, high purity), and induces pozzolanic reactions in concrete. These reactions induce the reduction of the content of $Ca(OH)_2$ in the pore solution of the concrete, causing it to need a smaller quantity of CO_2 to react with $Ca(OH)_2$ producing carbonates. Thus, the rate of carbonation increases due to the lower amount of $Ca(OH)_2$ to react with the CO_2 diffusing in the pores. The concrete with pozzolan additions are more susceptible to the effects of reinforcement corrosion than concrete made with cement without mineral addition [28].

High resistance concrete showed a higher resistance to external attack such as carbonation process due to the lower particle size of CPV cement than the CPIII cement. The samples with two types of aggregates (0 and 1) showed a lower carbonation depth, once the presence of both aggregates reduced the porous size or increased compaction, enhancing the concrete quality.

5. CONCLUSIONS

For samples prepared with CPIII cement with one or two aggregates, the metakaolin addition increased the capillary absorption. For the samples manufactured with CPV cement, with a lower particle size, the sample without MK addition showed a higher capillary absorption, for the samples prepared with crushed stone 1, and with both crushed stones.

The lowest value of carbonation depth was observed for samples manufactured with CPV cement and two aggregates. The highest carbonation depth was observed for the sample prepared with CPIII cement, crushed stone 1, and MK addition.

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