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Editors

Calcined Clays for Sustainable Concrete

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Contents

Calcined Clay-Cement Stabilisation – Physicochemical Attributes and Stabilised Strengths of a-1-a and a-2-6 Soils 1
O.A. Adekitan and G.M. Ayininuola

Sulfate Resistance of Cement Mortar Containing Metakaolin 8
Abdelsalam M. Akasha and Jamal M. Abdullah

Use of R^3 Rapid Screening Test to Determine Reactivity and Chloride Binding Potential of Locally Available Kaolinite Clay . . . 15
M. Almarshoud, J.L. Saint Rome, and K.A. Riding

Assessment of Cuban Kaolinitic Clays as Source of Supplementary Cementitious Materials to Production of Cement Based on Clinker – Calcined Clay – Limestone 21
Roger S. Almenares Reyes, Adrián Alujas Díaz, Sergio Betancourt Rodríguez, Carlos Alberto Leyva Rodríguez, and José Fernando Martirena Hernández

Proposal of a Methodology for the Preliminary Assessment of Kaolinitic Clay Deposits as a Source of SCMs 29
Adrián Alujas Díaz, Roger S Almenares Reyes, Florencio Arcial Carratalá, and José F. Martirena Hernández

Hydration Study of Limestone Calcined Clay Cement (LC^3) Using Various Grades of Calcined Kaolinitic Clays 35
F. Avet and K. Scrivener

Reaction Degree of Metakaolin in Limestone Calcined Clay Cement (LC^3) 41
F. Avet and K. Scrivener

Durability of Steam Cured Pozzolanic Mortars at Atmospheric Pressure 46
Kübra Ekiz Barış and Leyla Tanaçan

Use of Low-Carbon Cement in the Preparation of Masonry Mortars for Building Restoration 54
D. Betancourt Cura, Y. Lima Triana, and F. Martirena Hernandez

Quantification of Pore Size Distribution Modification Due to Metakaolin Inclusion in Cement Based Systems 60
B. Bhattacharjee

Limestone Calcined Clay Cement: The Experience in India This Far 64
Shashank Bishnoi and Soumen Maity

Pilot Scale Production of Limestone Calcined Clay Cement 69
Shashank Bishnoi, Soumen Maity, Mukesh Kumar, S.K. Saxena, and S.K. Wali

The Special Case of North-Eastern India for the Production of LC^3 . . . 75
Shashank Bishnoi, Soumen Maity, S.P. Pandey, and P.K. Tripathy

Comparative Study of Compressive Creep Behavior of Concrete with Metakaolin or Silica Fume 80
R. Bucher, H. Cagnon, T. Vidal, and M. Cyr

Effect of Carbonate Minerals and Calcination of Carbonatites and Kamafugites on Their Pozzolanic Performance and Early Age Concrete Properties 86
A. Buregyeya, Y. Ballim, S. Nwaubani, A.G. Kerali, and M. Otieno

Assessment of the Pozzolanic Reactivity of Calcined Kaolinitic Clays by a Rapid Alkaline Solubility Test 98
E. Cabrera, R. Almenares, and A. Alujas

Sustainability of Cuban Construction Supply Chain by Means of LC^3 Cement: Case Studies in Villa Clara Province 105
Yudiesky Cancio Díaz, Inocencio Raúl Sánchez Machado, José Fernando Martirena Hernández, and Guillaume Habert

Degradation of Calcined Clay-Limestone Cementitious Composites Under Sulfate Attack 110
Cheng Yu, Peng Yuan, Xin Yu, and Jiaping Liu

Sulfate and Alkali-Silica Performance of Blended Cements Containing Illitic Calcined Clays 117
Gisela Cordoba, Agustin Rossetti, Dario Falcone, and E.F. Irassar

Use of Ceramic Waste as a Pozzolanic Addition on Cement 124
Rayda Crespo Castillo

Development of the Microstructure in LC^3 Systems and Its Effect on Concrete Properties 131
Yuvaraj Dhandapani, K. Vignesh, Thangadurai Raja, and Manu Santhanam

Carbonation of Concrete with Low Carbon Cement LC3 Exposed to Different Environmental Conditions 141
Ernesto Díaz, Raúl González, Dayran Rocha, Adrian Alujas, and Fernando Martirena

Evaluation of Compressive Strength and Microstructure of Cement Pastes Containing Different Qualities of Metakaolin 147
N. Dumani and J. Mapiravana

Influence of Initial Water Curing on Strength and Microstructure Development of Blended Cements 155
A.C. Emmanuel, H. Talluru, S. Krishnan, and S. Bishnoi

Initial Performance Evaluation of Calcined Clay Based Ternary Blended Cement Under Various Climatic Conditions in India 160
A.C. Emmanuel, G. Mishra, and S. Bishnoi

Alkali Silica Reaction and Sulfate Attack: Expansion of Limestone Calcined Clay Cement 165
A. Favier and K. Scrivener

The Effect of Limestone on the Performance of Ternary Blended Cement LC3: Limestone, Calcined Clays and Cement 170
Aurélie Favier, Franco Zunino, Ioannis Katrantzis, and Karen Scrivener

Influence of Clay Type on Performance of Calcined Clay – Limestone Portland Cements 176
S. Ferreiro, D. Herfort, and J.S. Damtoft

Metakaolin-Based Geopolymers for Nuclear Waste Encapsulation 183
D.A. Geddes, X. Ke, S.A. Bernal, M. Hayes, and J.L. Provis

The Influence of Recycled Concrete and Clay Brick Particles on the Strength and Porosity of Cement-Based Pastes 189
T.M. Grabois, G.C. Cordeiro, and R.D. Toledo Filho

The Effect of Kaolinite Content of China Clay on the Reactivity of Limestone Calcined Clay Cement 195
P.K. Haldar, S. Mithia, K. Mukherjee, N.R. Dhabarde, E. Bansal, P. Phulwari, A. Kumar, S. Kesh, and S. Maity

The Effect of Alkali on the Properties of Limestone Calcined Clay Cement (LC^3) 200
W. Hanpongpun and K. Scrivener

On the Reactivity of Calcined Clays from Lower Lusatia for the Production of Durable Concrete Structures 205
Klaus-Juergen Huenger, Robert Gerasch, Ingolf Sander, and Maria Brigzinsky

Compressive Strength Improvements of Cement-Based Composites Achieved with Additional Milling of Metakaolin 212
Biljana Ilić, Aleksandra Mitrović, Vlastimir Radonjanin, Mirjana Malešev, and Miodrag Zdujić

Properties of the Cement-Based Composites with High Content of Metakaolin 219
Biljana Ilić, Vlastimir Radonjanin, Mirjana Malešev, Miodrag Zdujić, and Aleksandra Mitrović

Calcined Clays – Performance as Composite Material 226
Christian Kalb

Structural Ordering of Aged and Hydrothermally Cured Metakaolin Based Potassium Geopolymers 232
Xinyuan Ke, John L. Provis, and Susan A. Bernal

Carbonation of Limestone Calcined Clay Cement Concrete 238
M.S.H. Khan, Q.D. Nguyen, and A. Castel

Grinding of Calcined Clays and Its Effects on Cement Properties 244
W. Kluge and B.O. Assmann

Hydration and Mechanical Properties of Limestone Calcined Clay Cement Produced with Marble Dust 249
Sreejith Krishnan, Arun C. Emmanuel, Swadesh Kumar Kanaujia, and Shashank Bishnoi

Performance-Based Design Procedure Applied to the Selection of Low-CO_2 Binder Systems Including Calcined Clay 254
Wilson R. Leal da Silva, Lars N. Thrane, Thomas L. Svensson, Sergio Ferreiro, Duncan Herfort, Claus Pade, and Jesper S. Damtoft

Thermal Processing of Calcined Clay 262
J. Lemke and C. Berger

Thermal Transformation of Illitic-Chlorite Clay and Its Pozzolanic Activity 266
Roxana Lemma, Cristina C. Castellano, Viviana L. Bonavetti, Monica A. Trezza, Viviana F. Rahhal, and Edgardo F. Irassar

In-situ Observation of Dissolution Behavior of Carbonatite in Water Glass Solution 273
Jing Li, Jianqin Lin, Qijun Yu, Jie Hu, and Suhong Yin

Analysis of the Mixing Performance Containing the LC3 as Agglomerant with Different Types of Calcined Clay 279
D. Lins, J. Rêgo, and E. Silva

Evaluation of Calcined Clays from Boyaca-Colombia Containing Alunite as Supplementary Cementitious Materials 286
Ariam Lozano Perez and Mathieu Antoni

Improvement of the Environmental Energy Sustainability in the Production of Cement Portland with Addition of Thermally Activated Clays 293
I.L. Machado, H.I. Moya, S.B. Sánchez, and F. Martirena

Resource Mapping of China Clay for LC^3 Application in India 299
A. Soumen Maity and B. Santanu Mithia

Chloride Transport Behavior of LC^3 Binders 306
H. Maraghechi, F. Avet, and K. Scrivener

Blended Cements with Calcined Illitic Clay: Workability and Hydration 310
Guillermina Marchetti, Jaroslav Pokorny, Alejandra Tironi, Mónica A. Trezza, Viviana F. Rahhal, Zbyšek Pavlík, Robert Černý, and Edgardo F. Irassar

Low Carbon Cement LC^3 in Cuba: Ways to Achieve a Sustainable Growth of Cement Production in Emerging Economies 318
Fernando Martirena and Karen Scrivener

Studies on the Influence of Limestone-Calcined Clay Blend on the Hydration of Cement 322
G. Mishra, A. Emmanuel, and S. Bishnoi

Thermal Resistivity of Chemically Activated Calcined Clays-Based Cements 327
Marangu J. Mwiti, Thiong'o J. Karanja, and Wachira J. Muthengia

Promising Early Age Evaluations of Fly Ash - Calcined Marl - OPC Ternary Cement 334
Serina Ng and Tone Østnor

Applicability of Lime Reactivity Strength Potential Test for the Reactivity Study of Limestone Calcined Clay Cement 339
Anuj Parashar, Vineet Shah, and Shashank Bishnoi

Limestone and Calcined Clay Blended Cement Used as Low-Cost Binder to Reduce Heat Production and Potential for Delayed Ettringite Formation 346
Jerry M. Paris and Christopher C. Ferraro

Hydrate Phase Assemblages in Calcium Sulfoaluminate – Metakaolin – Limestone Blends 352
M. Pedersen, B. Lothenbac, F. Winnefeld, and J. Skibsted

Influence Grinding Procedure, Limestone Content and PSD of Components on Properties of Clinker-Calcined Clay-Limestone Cements Produced by Intergrinding 358
A. Pérez, A. Favier, K. Scrivener, and F. Martirena

Durability of Concretes Made with Calcined Clay Composite Cements 366
Roland Pierkes, Simone E. Schulze, and Jörg Rickert

Alkali-Activation of Calcined Clays – Past, Present and Future 372
John L. Provis

Influence of a Calcined Clay and the Temperature on the Hydration of an Oilwell Cement 377
Juan Alberto Ribalta, Adrián Alujas Díaz, and José Fernando Martirena

Influence of the Kind of Mineral Addition and the Seawater on the Hydration of a Portland Cement 384
Juan Alberto Ribalta, Leidys Laura Pérez, and Adrián Alujas Díaz

Standardization Strategy of Low Carbon Cement in Cuba. Case Study for "Siguaney" Cement Factory 391
D. Rocha, R. Almenares, S. Sanchez, A. Alujas, and F. Martirena

Antibacterial Metakaolin-Based Geopolymer Cement 398
Jose-Carlos Rubio-Avalos

Identification of Reactive Sites in Calcined Kaolinite and Montmorillonite from a Combination of Chemical Methods and Solid-State NMR Spectroscopy 404
C. Ruiz-Santaquiteria and J. Skibsted

The Decay of the Historical Site of Malecon in Havana, Cuba: Salt Crystallization Damage at Repair Interfaces 409
A.M. Aguilar Sanchez, F. Caruso, F. Girardet, F. Martirena, T. Wangler, and R.J. Flatt

Introducing Low Carbon Cement in Cuba - A Life Cycle Sustainability Assessment Study 415
S. Sánchez Berriel, Y. Ruiz, I.R. Sánchez, J.F. Martirena, E. Rosa, and G. Habert

Sulphate Optimization of Binders with Calcined Clay Using Isothermal Calorimetry 422
P. Sandberg and S. Bishnoi

Reaction Kinetics of Basic Clay Components Present in Natural Mixed Clays 427
S. Scherb, N. Beuntner, and K.-C. Thienel

Colloid-Chemical Investigation of the Interaction Between PCE Superplasticizers and a Calcined Mixed Layer Clay 434
M. Schmid, N. Beuntner, K.-Ch. Thienel, and J. Plank

Prediction of Carbonation Depth in Blended Systems 440
V. Shah and S. Bishnoi

Autogenous Shrinkage and Creep of Limestone and Calcined Clay Based Binders 447
J. Ston, A. Hilaire, and K. Scrivener

Hydration of Blended Cement with Halloysite Calcined Clay 455
Alejandra Tironi, Fernanda Cravero, Alberto N. Scian, and Edgardo F. Irassar

Progress of Limestone Calcined Clay Cement in China 461
Sui Tongbo, Bin Wang, Yuliang Cai, and Shengliang Tang

Thermal Activation of Two Complex Clays (Kaolinite-Pyrophillite-Illite) from Tandilia System, Buenos Aires, Argentina 469
M.A. Trezza, A. Tironi, and E.F. Irassar

Assessment of Calcined Clays According to the Main Criterions of Concrete Durability 475
A. Trümer and H.-M Ludwig

Application of Industrially Produced LC^3 to Pavements, AAC Blocks and Other Products 482
S.K. Wali, S.K. Saxena, Mukesh Kumar, Soumen Maity, and Shashank Bishnoi

Machine Learning Approaches to Admixture Design for Clay-Based Cements 488
N.R. Washburn, A. Menon, C.M. Childs, B. Poczos, and K.E. Kurtis

Micro-Chemo-Mechanical Characterization of a Limestone-Calcinated-Clay Cement Paste by Statistical Nanoindentation and Quantitative SEM-EDS 494
William Wilson, Luca Sorelli, Sreejith Krishnan, Shashank Bishnoi, and Arezki Tagnit-Hamou

Addressing Key Challenges in MK-PLC Blends at Early Ages: Workability, Slump Retention, and Heat of Hydration 500
B.H. Zaribaf and K.E. Kurtis

Assessing the Effect of Calcite Impurities in Clay on Optimal Dehydroxylation Parameters for Enhanced Reactivity 507
F. Zunino and K. Scrivener

Reactivity and Performance of Limestone Calcined-Clay Cement (LC^3) Cured at Low Temperature 514
F. Zunino and K. Scrivener

Durability of Steam Cured Pozzolanic Mortars at Atmospheric Pressure

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Abstract. In the present study, the durability properties of Earth of Datça--hydrated lime mortar (ED)M under steam curing conditions at atmospheric pressure was inspected and compared with relatively well-known "horasan" mortars consisting of hydrated lime and fired clay as artificial pozzolan. Resistance of the lime-pozzolan mortars to wetting and drying, salt crystallization, freeze-thaw cycling and ageing by SO_2 action tests was assessed. Tests results showed that both mortars produced under steam curing at atmospheric pressure can be used as load-bearing building bricks. The better behaviour of the ED(M) under ageing tests compared to Fired Clay-hydrated lime mortar (FC)M is in conformity with its efficient physical and mechanical characteristics.

1 Introduction

Pozzolans are silicate or alumino-silicate compositions which by themselves possess no binding capacity, but when finely grinded react with calcium hydroxide and water at ordinary temperature to form calcium silicate hydrates or calcium aluminate hydrates [1]. Pozzolanic mortars can be used for the structures exposed to an excessive amount of moisture and liquid water such as public baths, aqueducts, and bridges due to their strength and durability [2]. Their durability can be affected by various deterioration factors such as wet and dry cycles, rain exposure and related leaching, freezing and thawing cycles, exposure to pollutants, and sun's rays. To what extent mortars are influenced by these deterioration factors depends on the type and properties of the binder and the aggregate, the binder/aggregate ratio, the amount of mixing water, the permeability of mortar, the capillarity coefficient, water intake, the presence of admixtures and the curing conditions [3]. The existence of water is necessary for several deterioration processes [4].

In the previous study, physical and mechanical characteristics of pozzolanic lime binder consisting of hydrated lime and (ED), under steam curing conditions at atmospheric pressure at 70 °C for 6 h was investigated [5]. Test results showed that the binder might be used for the production of materials similar to that of fired clay and calcium silicate bricks. In the present study, durability properties of (ED)M under the same curing conditions was investigated and compared with relatively well-known "horasan" mortars consisting of fired clay and hydrated lime (FC)M.

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2 Materials and Methods

Quicklime supplied from its producer was slaked to obtain hydrated lime putty and stored in the moist state by covering it for minimum 15 days. ED obtained from the volcanic rocks of Datça Peninsula [6] was used as the natural pozzolan. FC supplied from a local pottery factory was used as artificial pozzolan. The maximum grain size of the pozzolans was determined as 90 µm. Semi quantitative element analysis (XRF) results of the hydrated lime and pozzolans are given in Table 1. Rılem Cembureau graded standard sand was used as aggregate.

Table 1. Chemical analysis of the Hydrated lime, Earth of Datça (ED), and Fired Clay (FC).

Component	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	CO_2	Na_2O	K_2O	TiO_2	P_2O_5	MnO	Cl	Cr_2O_3	SO_3	Total
Lime (%)	1	-	-	1	85	5	-	-	-	-	-	-	-	0.6	92.6
ED (%)	72.07	13.06	1.39	0.44	2.34	-	3.27	4.60	0.20	0.05	0.06	0.10	-	-	99.73
FC (%)	45.21	14.95	14.56	4.52	9.32	-	2.52	2.47	2.12	0.27	0.21	0.05	0.37	0.48	99.68

The pozzolanic activity of (ED)M and (FC)M was examined according to [7]. The mixture proportion of lime:pozzolan:sand is 1:2.5:9, by weight. The mixtures were prepared at water-to-solid ratio of 0.16, by weight. The mixtures were cast in prismatic 40 × 40 × 160 mm moulds. The freshly moulded specimens, pre-cured at 20 ± 2 °C and 95 ± 5% RH for 36 h in the moulds by keeping them inside the polyethylene cover. Then they were removed from the moulds and PE and cured at 20 ± 2 °C and 65 ± 5% RH for 24 h. After pre-curing, the specimens were cured at 70°C under saturated steam in a closed container for 6 h [5]. The specimens were tested for specific density, unit weight [8], capillary water absorption [9], water absorption under

Table 2. Properties of (ED)M and (FC)M before and after durability tests

Properties	Before durability tests									
	Wetting-drying cycles		Salt crystallisation		Freeze-thaw cycles		Ageing by SO_2 action			
ED	FC	ED	FC	ED	FC	ED	FC	ED	FC	
Pozzolanic Act. test (MPa) Compressive str./ Flexural str.	6.12/1.43	4.10/1.03								
Weight variation (%)	-	-	+0.34	+0.54	+4.48	+5.90	−3.59	−9.04	+0.25	+0.46
Unit weight (g/cm^3)	1.81	1.78	1.86	1.84	-	-	-	-	-	-
Capillarity coefficient (cm^2/s)	0.09	0.11	-	-	-	-	-	-	-	-
Water absorption (%)	8.11	9.28	-	-	-	-	-	-	-	-
Apparent porosity (%)	14.71	17.15	13.68	15.43	-	-	-	-	-	-
Porosity (%)	22.98	32.36	-	-	-	-	-	-	-	-
Ultrasound velocity (km/s)	1.88	1.81	2.18	2.27	3.35	3.74	-	-	-	-
Compressive strength (MPa)	10.74	6.10	13.00	9.88	12.78	7.56	-	-	-	-
Flexural strength (MPa)	1.49	1.15	2.28	2.60	1.81	1.50	-	-	-	-
Modulus of elasticity (MPa)	6390	6020	8940	9480	-	-	-	-	-	-
Visual inspection scale	-	-	0	0	0	0	3	3	0	0
Specific density ED: 2.35 g/cm^3; FC: 2.63 g/cm^3; Hydrated lime: 2.15 g/cm^3										

atmospheric pressure [10], ultrasound velocity to determine Young's dynamic modulus [11] and flexural and compressive strengths [12]. Mortars' resistance to wetting and drying [13], salt crystallization [14], freeze-thaw cycling [15], and ageing by SO_2 action [16] were assessed to determine the effect of accelerated aging. Following the durability tests, specimens were visually inspected, compared with the reference specimens and their appearance was scored according to the scale given in [15]. The test results are the average of six specimens and given in Table 2.

3 Results and Discussion

3.1 Physical and Mechanical Properties

Results obtained by XRF and pozzolanic activity tests, for both (ED)M and (FC)M, fit well with the requirements of TS 25 [7]. After they were cured at low temperature steam curing their compressive strengths increase 1.75 and 1.45 times and flexural strengths increase 1.04 and 1.12 times respectively for (ED)M and (FC)M than TS 25. This improvement observed also in their physical properties seems low temperature steam curing is the suitable curing condition for them to enhance the formation of CSH gel produced from the pozzolanic reactions [5]. The CSH gel leads to infilling pores, thus, increases pore refinement, reduces total porosity and the amount and connectivity of capillary pores within the mortar [17]. On the other hand (ED)M has more reactivity towards lime regarding the development of successfully hydration reactions; hence, measured compressive and flexural strengths of (ED)M are 1.76 and 1.29 times higher than (FC)M. Parallel to these results, other properties of (ED)M like porosity, apparent porosity, which indicates the existence of exposed pores, and water absorption were respectively found 0.71, 0.86 and 0.87 times lower than (FC)M as expected. Ultrasound velocity, unit weight and capillarity coefficient values of the both mortars are close to each other, which may indicate they have similar pore structure.

3.2 Influence of Wetting-Drying Cycles

After 20 cycles of wetting-drying, the increase of the mass variation in (ED)M and (FC)M was found very low, 0.34% and 0.54%, respectively, and can be considered negligible. All the physical and mechanical properties of both type of the mortars improved after the test. This increment is probably assigned to the fact that there is a pozzolanic reaction enhancement by the application of successive wetting and drying cycles. 70 °C drying at the wetting-drying test is equal to the curing conditions applied in this study and besides; it is well known that pozzolanic mortars prone to water. The ratio of this improvement was 1.34 times higher for (FC)M than (ED)M. The higher CaO content of FC pozzolan may lead to the higher ratio of $Ca(OH)_2$ formation in the solution which may continue its pozzolanic reaction to form CSH gels during the wetting-drying cycles. At the end of the test, there were no flaking, cracking, or any other alteration observed on the specimens which can be classified as '0' according to the numerical scale of visual inspection (Fig. 1).

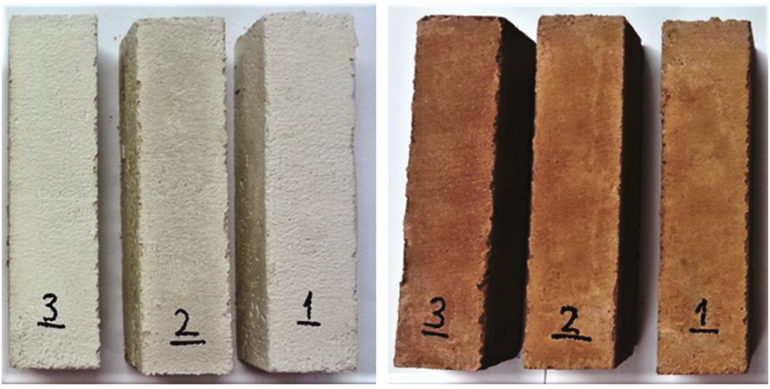


Fig. 1. The specimens after wetting-drying cycles: (ED)M on the left; (FC)M on the right.

3.3 Influence of Salt Crystallization

The salt decay susceptibility is described by the salt uptake and the mass variations for specimens [18]. Mass uptakes of (ED)M and (FC)M gradually increased towards 15th cycles due to the absorption of salt from solution inside the pores accessible to the mortar. Total connected porosity and the microporosity of pores smaller than 5 μm in radius optimally facilitate salt uptake [19]. The quantity of the salt uptake of the (FC)M was 1.32 times higher than that of (ED)M, because of its 1.40 times higher total porosity and 1.14 times higher water absorption ratio than the former one. Proximate improvements were recorded in mechanical properties of the both mortars. High mass uptake of the salts and their subsequent crystallization within the pores of the mortar during the drying stage may partially infill the pores of the material. This increase may also be due to the reaction of Na_2SO_4 with $Ca(OH)_2$ to form gypsum and ettringite which fill in the micro-pores leading to a denser structure [20]. Likewise, ultrasound velocities of the (ED)M and (FC)M increased 1.78 and 2 times respectively. On the other hand, according to the visual inspection of the mortars, neither disintegration into fragments, nor significant surface changes such as hairline cracking or chipping were noted that



Fig. 2. (a) The specimens after salt crystallization test; (b) The section of the mortars:

may occur due to crystal pressure over the pore surface and hence, classified as '0' in the scale (Fig. 2a). After salt crystallization test, relatively darker colour was observed at the exterior side of the sections of (FC)M broken in the flexural tests (Fig. 2b).

Based on this observation, after the test, the specimens were soaked in deionized water for 24 h and were left to dry at constant ambient temperature. While the surface of the (ED)M did not display any visual alteration, efflorescence was seen on surface of the (FC)M due to its higher salt content (Fig. 3).

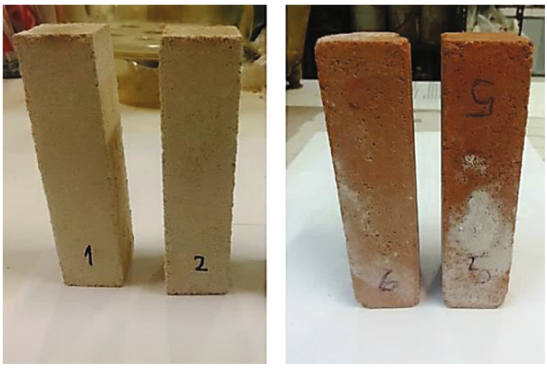


Fig. 3. The specimens after dried at constant ambient temperature: (ED)M on the left; (FC)M on the right.

3.4 Influence of Freeze-thaw

Although saturation coefficients (ST) of both mortars are lower than 80% both mortars loss appreciable amount of material after the freeze-thaw test. Freeze-thaw test of the (FC)M could not be continued further after the 9th cycle because of the formation of the large cracks and dissolution. On the other hand, any substantial differences were noted at the (ED)M after the 9th cycle. The total mass loss of (FC)M and (ED)M was 9.04%



Fig. 4. (ED)M after 20 freeze-thaw cycles (on the left); (FC)M after 9 freeze-thaw cycles (on the right).

(after 9th cycle) and 3.59% (after 20th cycle), respectively. (FC)M was more susceptible to freeze-thaw degradation due to its higher total porosity with small pore sizes, higher water absorption ratio and lower tensile strength. Excessive amount of $Ca(OH)_2$ content stem from the pozzolan type and probably incomplete pozzolanic reaction may accelerate the disintegration of the (FC)M. Visual analysis scale of the (ED)M was determined as '3' after 20 cycles because of the several cracks larger than 0.1 mm width and surface scaling (Fig. 4).

3.5 Influence of Ageing by SO_2 Action

SO_2, NO_x and CO_2 are the atmospherical pollutants and among these SO_2 is the most dangerous one [21]. SO_2 and NO_2 react with water to form HNO_3 and the H_2SO_4 acids. Acidic waters in contact with $CaCO_3$ of the mortars and concretes give place to the formation of calcium nitrates, sulphates, bicarbonates, etc. Following formula shows the conversion of $CaCO_3$ to gypsum with its reaction with sulphuric acid. Gypsum formation is responsible for the development of cracks and spalling because of its expansion during crystallization.

$$H_2O + H_2SO_4 + CaCO_3 \rightarrow CaSO_4, 2H_2O + CO_2 \uparrow \quad (1)$$

After 21 days of exposure to the 5% SO2 concentration, the appearance of the mortars was inspected and in no case were chipping, hairline cracking, scaling or cracks noted (Fig. 5). The increase in mass variation of (ED)M and (FC)M is very low and can be considered negligible. According to the visual analysis scale, '0' could be attained. This may be attributed to the steam curing conditions of the mortars in which pozzolanic reaction is dominant than carbonation reaction.

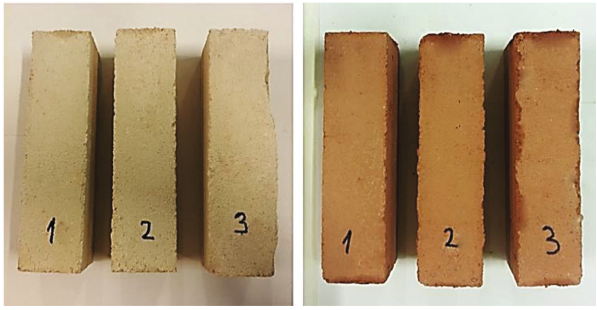


Fig. 5. The specimens after ageing by SO2 action test: (ED)M on the left; (FC)M on the right.

4 Conclusions

- According to the results of the pozzolanic activity, ED seemed to be the more reactive triggering a superior development of the hydraulic compounds.
- Steam curing at atmospheric pressure benefits hydration reactions between lime and both types of pozzolans. A special importance should be given to the curing conditions of the mortars.
- This study reveals the importance of pozzolanic activity and its influence on the mortars durability. The higher the pozzolanic activity the higher the amount of hydrated calcium silicates and aluminates; thus, more resistance to durability tests.
- In the wetting-drying test, wetting in the water and drying at 70 °C may provide additional curing effect on the mortars which result in any mass loss or visual degradation but strength gain.
- Either (ED)M or (FC)M are found resistant to the deterioration of salt crystallization due to their denser structure and strength gains after salt uptake. On the other hand higher porosity of FC mortar keeps higher salt intake which deposits efflorescence on the surface of the mortar carried with water.
- Ageing under freeze-thaw cycles is found as the most aggressive test. On the other hand, (FC)M has lower resistance to freeze-thaw effect due to its lower mechanical strength, higher total porosity with small pore size. Composition of the pozzolan is also an important parameter regarding frost resistance.
- In terms of exposure to SO_2 concentration, any surface level deterioration and changes in volume or expansion on the mortars was observed. This indicates that both mortars may keep their stability against atmospheric pollutants in time.
- Both mortars of (ED)M and (FC)M produced under steam curing at atmospheric pressure can be used as load-bearing building bricks. The better behaviour of (ED)M under ageing tests compared to (FC)M is in conformity with the efficient physical and mechanical characteristics of the (ED)M.

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