

## **EFFECT OF CARBONATION ON THE CHLORIDE DIFFUSION OF MORTAR SPECIMENS EXPOSED TO CYCLIC WETTING AND DRYING**

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### **Abstract**

Carbonation and chloride ingress are the two main causes of corrosion in reinforced concrete structures. Despite the combined action of these mechanisms being a reality, there is little research on the effect of carbonation on the chloride diffusion in concrete. This work intends to study the influence of carbonation on chloride diffusion of mortar specimens. Cubic mortar specimens were cast with 0.55 of water-cement ratio. After curing, the specimens were subjected to 56 days of wetting and drying cycles. Half the samples were immersed for a day in a 3.5% NaCl solution and then placed for 6 days in a carbonation chamber (4%CO<sub>2</sub>); the other half were also kept a day in 3.5% NaCl, but after were kept during 6 days in laboratory environment. Afterwards, the depth of chlorides and CO<sub>2</sub> penetration was evaluated. Complementary tests were also carried out, such as rapid chloride migration coefficient and water capillary absorption. The results show that carbonation has a direct influence on chloride penetration, decreasing it. The noncarbonated samples showed a profile with higher amount of chloride than carbonated ones. This fact can be related to the refinement of the mortar pores caused by carbonation and observed in water absorption tests.

**Key words:** chloride, carbonation, mortar, deterioration, combined degradation, accelerated testing

## 1 INTRODUCTION

A significant number of concrete structures around the world have been damaged by steel reinforcement corrosion. Among the factors that contribute to this kind of damage is the aggressiveness of chloride ions, one of the main reasons for damage to ordinary concrete structures due to reinforcement corrosion [1].

According to Tilly and Jacobs [2] simultaneous chloride ingress and carbonation account for over 50% percent of the deterioration of concrete structures . It is well known that the corrosive effect of chlorides is based on their ability to destroy the electrochemical condition of the passive state of reinforcement even under the existing alkalinity in the concrete. Regarding carbonation, the corrosive effect is based on the decreasing trend of alkalinity of pore liquid in concrete and the resulting depassivation of steel, enabling the conditions for corrosion to occur.

The service life of concrete structures is directly affected by its durability. Thus, several authors have been studying the action of chloride ions and carbonation [3,4]. However, previous studies were confined mostly to the deterioration of concrete structures under a single deteriorating factor, although the real environment is actually a combination of factors [5].

Recently research has begun to look into the effect of combined degradation mechanisms. Chloride ingress and carbonation are among the most studied, however, there is no consensus on the effect of the combined action of chlorides and carbonation on the durability of concrete. In their experimental research, Chengfang *et al.* [6], for example, conclude that after carbonation, chloride diffusivity coefficient increases and it will increase with the increasing of carbonation time. On the other hand, Backus *et al.* [7], for example, say that the combination of carbonation with the entrance of chloride can act reducing the penetration of chlorides in concrete. However, the lack of standardization about the various parameters involved in the tests such as type of test, temperature, humidity and concentration of NaCl and CO<sub>2</sub> makes it difficult to compare results.

This experimental work studies the influence of carbonation on chloride transport into mortar specimens using accelerated tests.

## 2 EXPERIMENTAL WORK

Cubic mortar specimens with 50x50x50mm<sup>3</sup> were cast with ordinary Portland cement (CEM I 42.5) based mortar manufactured with 0.55 of water-cement ratio and a common river sand. Chemical composition of cement is presented in Table 1. Mortar mixture and its main characteristics in fresh (flow table test) and hardened state (compressive strength at 28 and 90 days, open porosity and permeability to oxygen and water) are presented in Table 2.

Table 1: Chemical composition of cement

Composition	Portland cement
SiO <sub>2</sub>	19.65
Al <sub>2</sub> O <sub>3</sub>	4.28
FeO <sub>3</sub>	3.35
CaO	61.35
MgO	1.7
SO <sub>3</sub>	3.36
K <sub>2</sub> O	0.89
Na <sub>2</sub> O	0.19
Insoluble residue – IR	1.7
Loss on ignition – Li	2.82

Table 2: Mixture and properties of mortar

Material and Properties	Mortar
Cement: sand (kg)	1:3
Flow value (mm)	190
Compressive strength (MPa) – 28/ 90 days	37.6/ 39.1
Open porosity (%)	21.4
Oxygen permeability (K) - 10 <sup>-16</sup> m <sup>2</sup>	0.797
Water permeability (K <sub>w</sub> ) - m <sup>2</sup>	11.16

After casted, these specimens were covered with a plastic sheet and they were stored, for one day, in a controller chamber (21°C and 98%RH). After, they were removed from the moulds and they were cured for 28 days in wet a chamber (21°C and 100%RH). Completed curing the specimens one had painted five of their six faces with an epoxy resin (3 days). Finished painting the specimens were drying more 4 days in laboratory environment (20°C and 60%RH).

After this, in order to simulate the combined action of chloride ions and carbonation, the specimens were subjected to wetting and drying cycles during 56 days. Half of the specimens were immersed for a day in 3.5% NaCl solution and then placed for 6 days in a carbonation chamber (20°C, 55%RH and 4%CO<sub>2</sub>); the other half specimens was used as reference specimens and were also kept a day in 3.5% NaCl solution, but after were stored 6 days in laboratory environment, Figure 1.



Figure 1: (A) Immersion in NaCl (B) Drying in carbonation chamber (C) Drying in laboratory environment

After the exposure period, the depth of penetration of chlorides and  $\text{CO}_2$  was determined. To evaluate the chloride penetration, a chloride profile was determined with 5mm interval until 30mm depth. Total chloride content was measured using the Volhard's method [8]. To evaluate the carbonation depth, the specimens were splitted along the direction of penetration of  $\text{CO}_2$ . Afterward, the fractured surfaces were sprayed with phenolphthalein and then the carbonation depth was measured [9]. Complementary tests were also carried out such as rapid chloride migration coefficient [10] and water capillary absorption [11].

### 3 RESULTS

#### 3.1 Wetting and drying cycles

Figure 2 shows chloride profiles obtained after wetting and drying cycles. Each point depicted represents the average value determined from three samples for each depth.

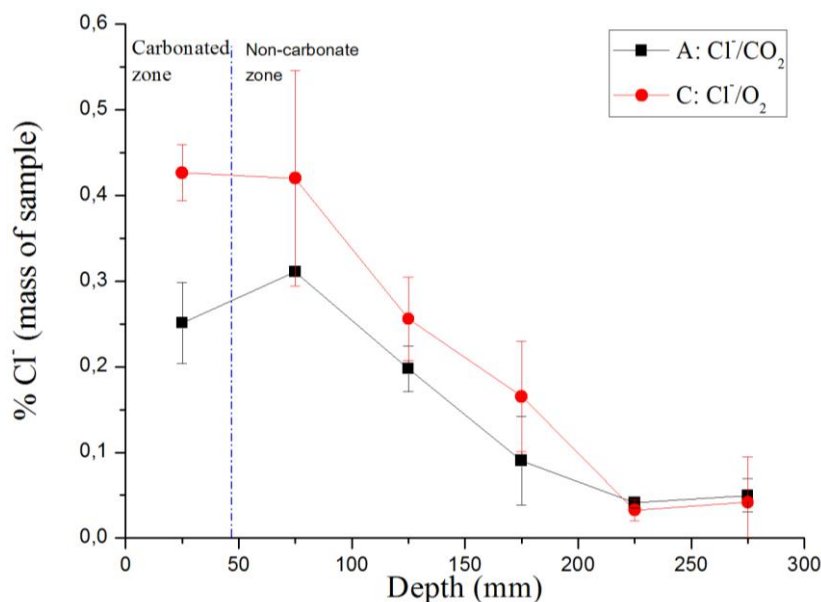


Figure 2: Chloride profiles

The chloride penetration achieved for specimens under combined cycles (A) was lesser than the one achieved for specimens under chloride cycles (C). This condition is more pronounced on the surface but it is maintained along almost entire profile.

The carbonation average depth obtained to specimens under combined cycles was  $4.9 \pm 0.04$  mm .

### 3.2 Complementary tests

The rapid chloride migration coefficient was evaluated for two types of specimens: specimens that were kept 56 days in a carbonation chamber only and specimens that were stored 56 days in a laboratory environment. The rapid chloride migration coefficient obtained for carbonated specimens was  $8.35 \times 10^{-12} \text{m}^2/\text{s}$ , while the reference specimens showed higher results,  $15.15 \times 10^{-12} \text{m}^2/\text{s}$  .

Figure 3 shows capillary absorption results obtained after the wetting and drying cycles. Each point depicted represents the average value from three specimens. The capillary absorption obtained for specimens under combined cycles (A) is bigger than results obtained for specimens under chloride cycles (C).

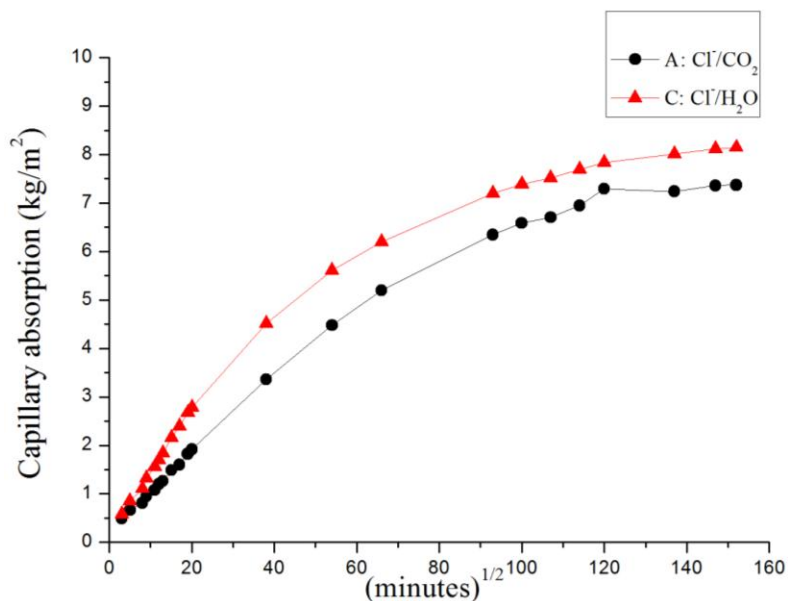


Figure 3: Water capillarity absorption to the situations studied

## 4 DISUSSION

### 4.1 Influence of carbonation on chloride penetration

The carbonation can reduce the capacity of chloride binding [12] and, consequently, lead to an increase in the rate of chloride ion ingress. On the other hand, carbonation can reduce the porosity and, consequently, lead to a decrease in the rate of chloride ion ingress. The chloride profiles presented in Figure 2 clearly show the influence of carbonation on chloride penetration for the studied mixtures. In this case, the carbonation acts by reducing the amount

of total chlorides present throughout the depth of the specimens. This fact can be related to the refinement of the pores of the mortar caused by carbonation.

There is a consensus that carbonation promotes microstructural changes that culminate in the densification of pores. The carbonation reaction gives rise to calcium carbonate ( $\text{CaCO}_3$ ) which solubility is limited and, therefore, tends to precipitate during the carbonation process [13]. This precipitation, at an initial phase, results in the aforementioned pore densification and consequent reduction of permeability.

The reduction in the permeability of the carbonated mortar can be confirmed by the water capillarity absorption results showed in Figure 3. It is possible to observe that the situation where the specimens were subjected to carbonation are those with a smaller water capillary absorption.

It is important to note that capillary absorption plays a key role in the wetting and drying cycles. In these tests, the chloride profile is generally different from those in which the relative humidity is approximately constant, Figure 4. For wetting and drying cycles there is a tendency to the formation of chloride's peaks in the region close to the surface. These peaks are typical formations of the cycles of gain and loss of moisture in the material surface [14].

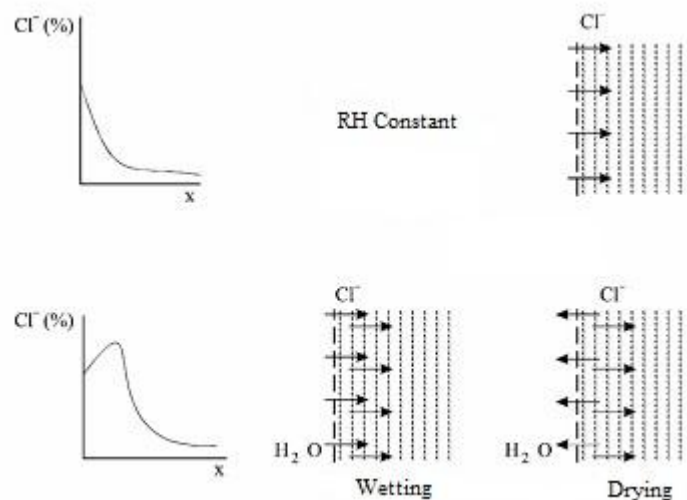


Figure 4: Simulation of chloride penetration in an environment with constant relative humidity (RH) and environment subjected to wetting and drying [13]

The chloride profiles presented in Figure 2 show the expected configuration for this type of test. However, the difference between the porosity of carbonated and non-carbonated zones may have increased the chloride peak observed in the combined cycle profile (A). The formation of a peak of chlorides after the carbonation front had already been verified by Lee and Yon [4] in a study on the deterioration of concrete, taking into account the combined action of carbonation and chloride ions. The skin effect cited by Andrade *et al.* [15] may explain this behaviour.

Concrete skin is considered the closest zone to the surface of concrete cover of reinforcements. It usually has a different composition than the internal concrete due to wall-effect phenomena or segregation of aggregates. In addition, environmental actions induce a gradient of moisture along the cover depth. These circumstances sometimes produce an irregular chloride profile in the cover, which either exhibits a maximum of chloride content some millimetres inside the outer surface or sometimes shows an anomalously high chloride concentration right at the concrete surface [15].

The extension of the test period and thus the carbonation depth may help to indicate the actual cause of the formation of the chloride peak observed in the combined cycle profile (A).

Concerning migration test, the results obtained emphasize the idea that the carbonation acts reducing the penetration of chlorides in mortars subjected to the combined action of the carbonation and chloride.

#### **4.2 Test time and humidity control**

Based on the obtained depth of carbonation, the carbonation front needs a longer test time to accentuate its depth and to clearly interact with the mechanism of chloride penetration. The time influence on the wetting and drying cycles was also observed by Lee *et al.* [5] for specimens subjected to the combination of carbonation and chlorides. Furthermore, the prolongation of the test time can cause leaching of precipitated calcium carbonate which may lead to open pores and cause an opposite effect.

Another important factor for the development of the carbonation front is moisture control. The drying time used (6 days) may have not been sufficient to provide a moisture content that could allow a maximum advance of the carbonation front inside the specimens. The presence of epoxy resin on five of the six faces of the specimens may have contributed to this behaviour.

## **5 CONCLUSION**

Based on the obtained results one may conclude that the chloride profiles presented clearly show the influence of carbonation on chloride penetration for the studied mixtures. In this case, carbonation reduces the amount of total chlorides present throughout the depth of the specimens. The diffusion coefficients of chloride obtained by the migration test corroborate this idea as they present lower values for carbonated samples than for those non-carbonated.

These statements, together, suggest that the passage of chloride ions through the carbonated zone of studied mortars becomes more difficult. Once the carbonated zone is on the surface of the specimen, it seems that this difficulty leads to a decrease of the initial amount of chlorides which can penetrate the sample and consequently reach its interior. However, more prolonged exposures can cause leaching of precipitated calcium carbonate which may lead to an increase in open pores and can cause an opposite effect.

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PROCEEDINGS  
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# XIII | DBMC

XIII International Conference on Durability  
of Building Materials and Components

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# Preface

*The ability to predict the service life—the durability—of a new building material is being ardently sought after in many parts of the world. Widespread efforts are correspondingly being made to increase the durability of materials already in wide use. Any estimate of the economic losses resulting from failures in durability lead inevitably to dollar figures so astronomically large that they are usually regarded with incredulity.*

R. F. Legget  
Proc. of the 1st DBMC  
Ottawa, 21-23 Aug. 1978.

Some 35 years after the 1<sup>st</sup> DBMC (Ottawa, 1978), sponsored by ASTM, NIST and NRC, durability is an even more important subject. At that time, an accurate service life estimate was considered a condition to life cycle cost (LCC) analysis. Nowadays, service life is also crucial information for the environmental life cycle analysis (LCA). The relevance of durability became widely recognized among scientists: in 1992 the World Business Council for Sustainable Development coined the term “eco-efficiency”, referring to a higher production with fewer environmental and economic resources and less environmental impact. The increase of durability (or service life) of products is one of the critical tools to improve eco-efficiency.

In that period the research community, which was organized into CIB and RILEM working groups, developed a methodology to plan and predict the service life of buildings and constructed assets at the design stage, which was consolidated in the ISO 15686-x standards. As a result of these scientific advances, the degradation factors and mechanisms of most materials and their assemblies are now much better comprehended. Information Technology advances facilitate to mapping the relevant environmental variables by using GIS platforms. Dose-response functions are available, making possible the estimation of degradation rates by combining environmental variables with material characteristics. Even effects of climate change on service life have been a matter of discussion. Altogether, it is an impressive achievement, recorded in the previous 12 DBMC conference. However, despite this scientific progress, the use of these tools in day-to-day production processes, including materials and components R&D, architecture and civil engineering design, and environmental and economic life cycle analysis is still very limited in most countries. Today’s research challenge is not only to increase the knowledge in the field, but also to make this knowledge readily available to the society, by developing more user friendly tools and better educate engineers and architects on the use of these tools.

The 13<sup>th</sup> edition of the DBMC conference was sponsored by ASTM, NIST, Rilem, CIB and University of Porto. It was joint organized by Polytechnic School of University of São Paulo and Secovi-SP, the most important Brazilian real estate industry’s association, an union that simbolizes our commitment to transfer the knowledge to the society. The support of Saint Gobain Group, Grace Construction Products, Votorantim Cimentos, Gerdau, Fapesp, CNPq and Capes made the conference possible.

We expect that these proceedings will help professionals and academy to incorporate service life planning concepts in their day-to-day decision-making processes.

São Paulo, 25-Aug-2014  
Marco Quattrone  
Vanderley M. John  
Editors

## CONTENTS

ACCELERATED AGING TEST TO STUDY SELF CLEANING PROPERTIES OF PHOTOCATALYTIC POST TREATMENTS	10
EVAULATION OF A MODULAR FRAMEWORK FOR PERFORMANCE-BASED DURABILITY ENGINEERING THROUGH ASSESSMENT OF A COASTAL REINFORCED CONCRETE STRUCTURE	18
PREDICTION OF SEALANT MODULUS CHANGE DUE TO OUTDOOR WEATHERING	26
DURABILITY OF COOL AND CONVENTIONAL COLORED COATINGS AFTER SIX MONTHS EXPOSURE AT DIFFERENT ENVIRONMENTS	33
IMPACT OF DIFFERENT EUROPEAN LOCAL CLIMATE CONDITIONS ON THE MOISTURE PERFORMANCE OF WOODEN COMMODITIES	41
NO CONCRETE IS SUSTAINABLE WITHOUT BEING DURABLE!	49
TOWARDS PERFORMANCE BASED DURABILITY STANDARDS FOR WOOD IN CONSTRUCTION – PART 1: DELIVERING CUSTOMER SERVICE LIFE NEEDS	57
TOWARDS PERFORMANCE BASED DURABILITY STANDARDS FOR WOOD IN CONSTRUCTION – PART 2: CONSIDERING MOISTURE RISKS IN WOODEN COMPONENTS	65
REPEATED AUTOGENOUS HEALING IN CEMENTITIOUS COMPOSITES WITH MICROFIBRES AND SUPERABSORBENT POLYMERS	73
STRUCTURAL RECYCLED CONCRETE SCIENCE AND MARKET: A SUCCESS STORY	81
IMPROVING THE RESILIENCE AND DURABILITY OF NEW ZEALAND'S BUILDINGS	89
RE-ASSESSMENT OF NEW ZEALAND'S ATMOSPHERIC CORROSIVITY	97
COMPARING THE INFLUENCE OF FLY ASH ON THE DURABILITY PROPERTIES OF PORTLAND CEMENT-BASED AND ALKALI ACTIVATED SLAG-BASED CONCRETES	105
ASSESSMENT OF THE HYDRATION CONSTANTS OF POST BLENDED BLAST FURNACE SLAG CEMENTS	113
HYGROTHERMAL CONSEQUENCES OF APPLYING EXTERIOR AIR BARRIER SYSTEMS IN HIGHLY INSULATED WOOD-FRAME WALLS: A PARAMETER ANALYSIS	121
THE DURABILITY OF BASALT FIBRES REINFORCED POLYMER (BFRP)	129
THE INFLUENCE OF CARBONATION ON DEICER SCALING RESISTANCE OF BLAST FURNACE SLAG CONCRETE (BFSC)	138
DISSOLUTION KINETICS EXPERIMENTS AS A METHOD TO ASSESS THE REACTIVITY OF SUPPLEMENTARY CEMENTITIOUS MATERIALS (SCMS): THE EFFECT OF SOLUTION COMPOSITION AND SCM STRUCTURE	147

DURABILITY OF STRUCTURAL STEEL INTRANSITIONAL FRAMED BUILDINGS	155
DEVELOPMENT OF EVALUATION METHOD BY SMALL CORE SPECIMEN FOR DETERIORATED FIBER CEMENT CORRUGATED SHEET AND SOME RESULTS OF ROOFS OVER STATION PLATFORMS	163
SERVICE LIFE PREDICTION AND EMBODY CO <sub>2</sub> OF WOODEN BUILDINGS	171
PERFORMANCE ANALYSIS AND DURABILITY OF ENVIRONMENTAL AND ENERGY SUSTAINABILITY INTERVENTIONS ON EXISTING BUILDINGS	179
FREEZE-THAW RESISTANCE OF ULTRA-HIGH PERFORMANCE CONCRETE IN NORMAL AND SEVERE CONDITIONS	187
GYPSUM EFFLORESCENCE ON CLAY BRICK MASONRY: FIELD SURVEY AND LITERATURE STUDY	196
DECAY SUSCEPTIBILITY OF RAIN EXPOSED WOOD JOINTS – COMPARISON OF TWO DECAY MODELS	204
THE EFFICIENCY OF AUTONOMOUS CRACK HEALING OF MORTAR IN CHLORIDE SOLUTIONS	212
DEGRADATION OF BRAZILIAN GYPSUM EXTERIOR WALLS DUE TO HYGROTHERMAL ACTION	220
A NEW PERFORMANCE TEST TO ASSESS THE POTENTIAL DELETERIOUS EFFECT OF SULPHIDE-BEARING AGGREGATES FOR CONCRETE	228
VACUUM INSULATION TECHNOLOGY FOR BUILDINGS – A COMPARATIVE LCA STUDY OF A SWEDISH MULTIFAMILY BUILDING	236
SYSTEM MODEL OF MOULD GROWTH ON BUILDING MATERIALS	244
ASSESSMENT OF DAMAGE DUE TO ALKALI-SILICA REACTION THROUGH MICROSCOPIC AND MECHANICAL TOOLS	250
AN IN-SITU AND LABORATORY STUDY OF THE EFFECT OF THE INTRINSIC PROPERTIES OF MORTARS ON THEIR POTENTIAL BIORECEPTIVITY	258
INFLUENCE OF MATERIAL CHARACTERISTICS ON ELECTROCHEMICAL REALKALISATION OF ARBONATED CONCRETES	266
ENVIRONMENTAL IMPACT ASSESSMENT OF FIRE RETARDANT TREATED WOOD USED IN EXTERIOR CLADDINGS	274
PERFORMANCE ANALYSIS OF WOOD SUBJECTED TO TREATMENTS MODIFIED WITH TITANIUM DIOXIDE AND CLAY NANOPARTICLES	281
PERFORMANCE OVER TIME OF ADHESIVE SYSTEMS FOR BONDING CERAMIC TILES ON FAÇADES – A CASE STUDY	289
HYGROTHERMAL PERFORMANCE OF A NATURALLY VENTILATED GYPSUM HOUSE – THE BRAZILIAN CLIMATE INFLUENCE	297
ACTUAL SERVICE LIFE PREDICTION OF BUILDING COMPONENTS	305

ALKALINE HYDROLYSIS OF POLYMERS IN CONTACT WITH CEMENT BASED MATERIALS	313
THE ENVIRONMENTAL BENEFITS OF SERVICE LIFE EXTENSION OF BUILDINGS COMPONENTS WITH TRANSFORMABLE DESIGN STRATEGIES	318
EXPERIMENTAL STUDY ON CARBONATION AND CHLORIDE MIGRATION COEFFICIENTS OF SELF-COMPACTING CONCRETE (SCC) WITH BINARY AND TERNARY MIXTURES OF FLY ASH (FA) AND LIMESTONE FILLER (LF)	326
RESISTANT-BASED MOISTURE CONTENT MEASUREMENTS ON WATER REPELLENT TREATED MORTAR AND BRICK	334
DURABILITY OF ROOF UNDERLAYS EXPOSED TO LONG TIME EXPOSURE UNDER IN-USE CONDITIONS	342
DRYING RATE AS A TRANSPORT INDEX FOR CONCRETE	351
DEVELOPMENT OF A METHODOLOGICAL PROCEDURE FOR THE DURABILITY EVALUATION OF EXISTING EXTERIOR FINISHINGS	359
STOCHASTIC, DETERMINISTIC, STATISTICAL AND ARTIFICIAL INTELLIGENCE BASED MODELS TO PREDICT THE SERVICE LIFE OF RENDERED FACADES	367
INFLUENCE OF LIMESTONE POWDER ON THE SETTING AND HYDRATION OF CEMENT PASTE IN THE FRESH STATE	375
IN-SITU MEASUREMENTS OF MOISTURE CONTENT AND TEMPERATURE IN WOODEN CONSTRUCTIONS	383
EXPERIMENTAL EVALUATION OF PERFORMANCES OF DIFFERENT SOLUTIONS OF FINISHING FOR MASONRY BUILDINGS FOR DETERMINING DURABILITY	391
A METHODOLOGY FOR EVALUATING MAINTAINABILITY AS A TOOL FOR DURABLE AND SUSTAINABLE BUILDINGS	399
INVESTIGATION OF THE EFFECTS OF LEACHING ON THE SCALING RESISTANCE OF CONCRETE	407
SERVICE LIFE OF INNOVATIVE SILOXANE COATING FORMULATIONS	415
EVALUATION OF RISING DAMP USING INFRARED THERMOGRAPHY	424
EFFECT OF CONFINEMENT AND CONCRETE STRENGTH UPON CRACK BEHAVIOR INDUCED BY CORROSION- PRODUCT EXPANSION	432
A NEW TEST METHOD TO EVALUATE THE RESISTANCE OF CEMENTITIOUS MATERIALS EXPOSED TO BIOGENIC ATTACK IN SEWER NETWORKS	440
DURABILITY OF QUATERNARY CEMENTS CONTAINING CALCAREOUS FLY ASH, SLAG AND LIMESTONE	449
RATING BUILDING ASSET DEGRADATION WITH EFFICIENCY INDEXES	457
DURABILITY OF FIBRE-CEMENT ROOFING CORRUGATED SHEETS	465

THERMAL SHOCK EFFECTS ON BONDING OF CEMENT MORTAR RENDERINGS	474
<b>EFFECT OF CARBONATION ON THE CHLORIDE DIFFUSION OF MORTAR SPECIMENS EXPOSED TO CYCLIC WETTING AND DRYING</b>	<b>482</b>
ACCELERATED CLIMATE AGEING OF WOOD SURFACE TREATMENTS AND THEIR EVALUATION BY FOURIER TRANSFORM INFRARED SPECTROSCOPY	490
NANORENDERS ON BUILDING FACADES: TECHNICAL, ECONOMIC AND ENVIRONMENTAL PERFORMANCE	498
AN INVESTIGATION OF THE EFFECTIVENESS AND DURABILITY DURABILITY CHARACTERISTIC OF BIOMASS ASHES AS POZZOLANIC MATERIALS	506
MULTI-PHYSICS MODELLING FOR DURABILITY EVALUATION OF ETICS	514
MICROSTRUCTURE DEVELOPMENT IN SLAG-CEMENT SYSTEMS	522
A NUMERICAL STUDY OF THE INFLUENCE OF THE HYDRAULIC INTERFACE CONTACT ON THE HYGRIC PERFORMANCE OF A MULTI-LAYERED SYSTEM	527
EVALUATION OF DAMAGE OF BUILDING FACADES IN BRASILIA	535
ACCELERATED AGEING AND CHARACTERIZATION OF EXPANDING WEATHER STRIPPING FOAM	543
DEVELOPMENT OF A 2D NUMERICAL MODEL FOR RAINWATER RUNOFF ON VERTICAL PLANES	551
AIRTIGHTNESS OF THE WINDOW–WALL INTERFACE IN WOOD-FRAME WALLS	559
A MODEL OF CARBONATION AND A TECHNIQUE TO DETERMINE THE DEGREE OF CARBONATION	570
NONDESTRUCTIVE DETERMINATION OF CHLORIDE ION USING AG/AGCL ELECTRODE PREPARED BY ELECTROCHEMICAL ANODIZATION	578
MODIFICATION OF CELLULOSE-CEMENT COMPOSITES BY ADMIXTURE OF SUGAR CANE BAGASSE ASH	586
ACCELERATED AGEING AND DURABILITY OF DOUBLE- GLAZED SEALED INSULATING WINDOW PANES	594
TORTUOSITY AND ELECTRICAL IMPEDANCE OF CONTAMINATED MORTARS WITH NITRATES OF Cr <sup>3+</sup> , Pb <sup>2+</sup> , AND Zn <sup>2+</sup> .	602
THE INFLUENCE OF WETTING PRIOR AND ADDITION OF FINES IN PRODUCED MORTARS WITH RECYCLED AGGREGATE	610
MOULD RISK ASSESSMENT FOR THERMAL BRIDGES: WHAT IS THE IMPACT OF THE MOULD PREDICTION MODEL?	616
A METHOD FOR BLIND VALIDATION OF HYGROTHERMAL CALCULATION TOOLS	624

METHODS FOR COMPENSATE LACK OF CLIMATE BONDARY DATA	632
FRAGILITY CURVES FOR THIN-WALLED COLD-FORMED STEEL WALL FRAMES WITH DIFFERENT SHEATHING SYSTEMS AFFECTED BY GROUND SETTLEMENTS DUE TO LAND SUBSIDENCE	640
SELF-HEALING OF SELF-COMPACTING CONCRETES MADE WITH BRAZILIAN BLAST FURNACE SLAG CEMENTS ACTIVATED BY CRYSTALLINE CATALYST	648
DURABILITY ASSESSMENT OF INNOVATIVE BUILDING PRODUCTS IN BRAZIL: EXAMPLE OF PVC COMPONENTS USED AS STAY-IN-PLACE FORMS AND FINISHING OF CONCRETE WALLS	656
SULFATE INGRESS AND MICROSTRUCTURAL CHANGES IN CEMENT PASTES AT EARLY AGE	665
DETERMINATION OF CRITERIA FOR THE EXPLORATION AND FOR OBTAINING INDICATORS IN EVOLUTIONAL ANALYSIS OF DEGRADATION IN URBAN FACADES	672
CORROSION RESISTANCE MEASUREMENTS AND THE INFLUENCE OF MATRIX ALKALINITY ON DURABILITY PERFORMANCE OF MEDIUM STRENGTH GEOPOLYMER CONCRETES	680
MAINTENANCE AND REFURBISHMENT; MARKET PRICE AND FIXED ASSETS VALUE	688
CHEMICAL TREATMENT WITH OXALIC ACID TO IMPROVE THE DURABILITY OF CEMENT-BASED MATERIALS IN ACID ENVIRONMENTS	695
SYNTHESIS, CHARACTERIZATION AND DURABILITY OF SELF-CLEANING ANTIREFLECTION COATINGS FOR GLAZING APPLICATIONS	704
EVALUATION OF CHLORIDE ION PENETRATION METHODS IN CONCRETE WITH RECYCLED CONCRETE AGGREGATE AND RICE HUSK ASH	712
PERFORMANCE OF THE COPPER TREATED, AND NATURALLY DURABLE WOOD IN LABORATORY AND OUTDOOR CONDITIONS	720
FROM LAB SCALE TO IN SITU APPLICATIONS _ THE ASCENT OF A BIOGENIC CARBONATE BASED SURFACE TREATMENT	728
MICROSCOPY AND COMPUTED MICRO-TOMOGRAPHY FOR EVALUATION OF MICROBIAL SELF-HEALING IN CONCRETE	736
MICROBIALLY INFLUENCED DEGRADATION OF CEMENT- BASED MATERIALS IN BIOGAS PRODUCTION ENVIRONMENTS	744
DURABILITY PROBLEMS WITH ZINC CLADDED ROOFS DUE TO IMPROPER UNDERLAYS	754
THE HIGH SCHOOL "GIUSTINO FORTUNATO" IN RIONERO IN VULTURE (POTENZA, ITALY): HOW TO REFURBISH AN HIGHER EDUCATION INSTITUTE MADE IN BRUTALIST ARCHITECTURE OF '80, LEADING IT TO BE A NZEB AND PRESERVING ITS FORMAL FEATURES.	762

DURABILITY AND MICROSTRUCTURE OF MORTAR WITH CALCINED MARL AS SUPPLEMENTARY CEMENTING MATERIAL	771
EMBODIED ENERGY AS A TOOL TO SUSTAINABLE STRATEGIC DECISIONS ON REFURBISHMENT	781
THE INFLUENCE OF THE SUBSTRATE CHARACTERISTICS ON THE BEHAVIOR OF THE MORTAR	789
DURABILITY OF PVC ROOFING AND WATERPROOFING MEMBRANES – CASE STUDIES AFTER 40+ YEARS OF EXPOSURE	797
PULL-OUT TESTS OF CAST-IN-PLACE ANCHOR STUDS WITH DIFFERENT EMBEDMENT DEPTHS	805
SERVICE LIFE ASSESSMENT OF CONCRETE STRUCTURES BASED ON SITE TESTING	813
ENVIRONMENTAL SUSTAINABILITY PERFORMANCE OF RESIDENTIAL BUILDINGS' THERMAL RETROFITS	821
SPECIFICATIONS FOR EXTERNAL RENDERS AND INTERNAL PLASTERS: A REVIEW ON DURABILITY ASSESSMENT	829
ANISOTROPIC EXPANSION OF ASR AFFECTED CONCRETE PRISM SPECIMENS	837
THE INFLUENCE OF CRACK WIDTH ON THE DURABILITY OF REINFORCED CONCRETE STRUCTURES	845
TIME-DEPENDENT PASSIVE INFRARED THERMOGRAPHIC INSPECTION OF FACADES	853
STUDY OF WOOD WASTE ASH AS A POZZOLANIC ADDITION	861
PROBABILISTIC MODELING DURABILITY OF WOODEN WINDOW FRAMES	869
DURABILITY OF JOINTED REINFORCED CONCRETE PAVEMENTS UNDER SEVER EXPOSURE CONDITIONS: A CASE STUDY IN IRAN	877
RESPONSE MONITORING OF NATIONAL HERITAGE FIVE STORY WOODEN PAGODA BY THE USE OF IMAGE PROCESSING.	885
OUTDOOR EXPOSURE TEST OF PHOTOCATALYST-COATED MATERIALS IN VARIOUS TEST LOCATIONS WITHIN ASIA	893
REHABILITATION OF A WASTEWATER TREATMENT PLANT	901
BACTERIOGENICALLY INDUCED SULFURIC ACID ATTACK ON CONCRETE IN AN AUSTRIAN SEWER SYSTEM	908
DURABILITY OF CEMENT-REDUCED ECO-FRIENDLY CONCRETES AGAINST SULFATE ATTACK	915
PHYSICAL AND CHEMICAL PROPERTIES OF CEMENTITIOUS MATERIALS UNDERGOING ACCELERATED DECALCIFICATION	922



EVOLUTION OF THE CEMENT MORTAR MICROSTRUCTURE CAUSED BY SALT CRYSTALLIZATION AND WATER FREEZING INVESTIGATED BY MULTICYCLE MERCURY INTRUSION POROSIMETRY	930
DURABILITY PERFORMANCE OF CONCRETE WITH GLASS POWDER AS SUPPLEMENTARY CEMENTITIOUS MATERIAL	938
USE OF 2-D NANO-PARTICLE TO ENHANCE THE CONCRETE RESISTANCE TO CHLORIDE INGRESS	946
CHARACTERISTICS OF IN-SERVICE, 30-YEAR-OLD EPDM ROOF MEMBRANE	954
EVALUATION OF CLUSTERED DISTRIBUTION OF SUPERABSORBENT POLYMERS AND ITS RELATION TO AUTOGENOUS SHRINKAGE BEHAVIOUR OF INTERNALLY CURED MORTARS	960
HAIL PROTECTED ETFE-ROOF LIGHTS FOR THE NEW ELEPHANT PARK BUILDING OF ZOO ZURICH	968
CHARACTERIZATION OF MORTARS WITH CERAMIC RESIDUES	976
STUDY OF THE DURABILITY OF MORTAR FOR STRUCTURAL CONCRETE MADE WITH FINE POWDER OF WHITE GLASS	984
ADHESIVE TAPES FOR BUILDING PURPOSES: FRAMEWORK FOR EVALUATION OF PERFORMANCE AND DURABILITY	994
INVESTIGATION METHODS OF THE RESISTANCE OF UHPC TO CHEMICAL ATTACK AND ITS CLASSIFICATION IN THE EXPOSURE CLASSES OF THE EUROPEAN STANDARD	1002
REDUCTION OF AUTOGENOUS SHRINKAGE IN OPC AND BFSC PASTES WITH INTERNAL CURING	1010
QUALITY CONTROL OF REINFORCED CONCRETE STRUCTURES FOR DURABILITY	1018
A NUMERICAL INVESTIGATION OF AGGREGATE SHAPE EFFECT ON THE DIFFUSIVITY OF MORTAR BASED ON ELLIPSOIDAL PARTICLE PACKING	1027
BIOGENIC CORROSION OF CONCRETE SEWER PIPES: A REVIEW OF THE PERFORMANCE OF CEMENTITIOUS MATERIALS	1035
THE EFFECTS OF VARIOUS MOULD RELEASE TECHNOLOGIES ON CONCRETE SURFACE FINISH	1043
INFLUENCE OF SERVICE LIFE ON BUILDING LCA	1053
CHARACTERIZATION THROUGH MERCURY POROSIMETRY OF TORTUOSITY FACTORS OF CONCRETES CONTAINING MINERAL ADDITIONS	1061
INFLUENCE OF REDISPERSIBLE POLYMER POWDERS ON DAMAGE MECHANISMS OF ETICS RENDER MORTARS	1069
TRANSPORT PROPERTIES OF RECYCLED FINE AGGREGATE CONCRETES	1077

EVOLUTION OF HYDRATION OF CEMENTS WITH COAL BOTTOM AND FLY ASH BY NON – DESTRUCTIVE TECHNIQUES AND POROSIMETRY	1085
MEASURINGWATERSORPTIONISOTHERMOFMORTAR CONTAINING CHLORIDE	1093
BACTERIAL CONCRETE: A BIOLOGICAL APPROACH TO REDUCED PERMEABILITY	1101
DEVELOPMENT AND STANDARDIZATION OF RAPID METHODS FOR ASSESSING THE FLUID PENETRATION RESISTANCE OF CONCRETE	1111
ASSESSMENT OF CHLORIDE INGRESS IN BLENDED CEMENT WITH COMBINED ADDITION OF CALCINED CLAYS AND LIMESTONE	1118
CHLORIDE PENETRATION DEPENDENCY ON FREEZE-THAW CYCLES	1126
COMBINING GEOGRAPHICAL INFORMATION SYSTEMS AND MULTI-SCALE MODELLING TO PREDICT SERVICE LIFE	1134
MULTI-OBJECTIVE DECISION-MAKING SUPPORTING SYSTEM OF MAINTENANCE STRATEGIES FOR DETERIORATING REINFORCED CONCRETE BUILDINGS	1142
SUSTAINING A HISTORIC HIGH-RISE STRUCTURE	1150
EFFECT OF CEMENT CONTENT ON CONCRETE DURABILITY	1158