

# **Materials Science and Intelligent Technologies Applications**

Edited by  
Khanittha Wongseedakaew and Qi Luo



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# **Materials Science and Intelligent Technologies Applications**

Selected, peer reviewed papers from the  
2014 3<sup>rd</sup> International Conference on  
Key Engineering Materials and Computer Science  
(KEMCS 2014),  
August 5-6, 2014, Singapore

*Edited by*

**Khanittha Wongseedakaew and Qi Luo**



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# Preface

2014 3rd International Conference on Key Engineering Materials and Computer Science (KEMCS 2014) will be held on August 5-6, 2014, Singapore. KEMCS 2014 is an integrated conference concentrating its focus upon Key Engineering Materials and Computer Science. The conference promises to be both stimulating and informative with a wonderful array of keynote and invited speakers Prof. Gerald Schaefer from Loughborough University, UK

Materials science is an interdisciplinary field applying the properties of matter to various areas of science and engineering. This scientific field investigates the relationship between the structure of materials at atomic or molecular scales and their macroscopic properties. It incorporates elements of applied physics and chemistry. With significant media attention focused on Nanoscience and nanotechnology in recent years, materials science has been propelled to the forefront at many universities. It is also an important part of forensic engineering and failure analysis. Materials science also deals with fundamental properties and characteristics of materials.

In order to meet high standard of “Advanced Material Research, the Organization committee has made their efforts to do the following things,

Firstly, we start to call for papers in January 10, 2014. During the past 4 months, we have been received 380 papers, but poor quality paper has been refused after reviewing by anonymous referee experts.

Secondly, all the papers have gone through a peer-review process for their originality and quality. The topics covered in this book include:

Chapter 1: Materials Science and Materials Engineering

Chapter 2: Artificial Intelligence and Data Mining, Data, Image and Signal Processing, Intelligent Automation and Control

Chapter 3: Computer Science and Information Technologies

Chapter 4: Electrical and Magneto electric Applications

Chapter 5: Advanced Technologies in Social, Education, Economics, Statistics and Management Applications

In the proceeding, you can learn much more knowledge about Key Engineering Materials and Computer Science of researchers all around the world.

I am thankful to colleagues for their contributions to KEMCS 2014. I should also like to thank the members of the Organizing Committee and the corporate sponsors for their efforts and contributions towards making the conference a success. Special thanks go to TTP Publisher Our sincerely acknowledgements are also expressed to the people who contribute to the proceedings and conference. Moreover, my thanks are also due to the sponsors for providing much help for the conference.

Finally, I hope the attendees benefit from the conference, and have a happy and meaningful journey in Singapore.

Khanittha Wongseedakaew and Qi Luo

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# Table of Contents

<b>Preface</b>	v
<b>Organization Committee</b>	vi

## Chapter 1: Materials Science and Materials Engineering

<b>Modifications in Water Sorption Isotherms of Cement Mortars Caused by Carbonation: Effects of Cycles</b> S.T. Pham.....	3
<b>NEPE Propellant Ignition and Combustion under Laser Irradiation</b> H.M. Wang, X. Chen and C. Zhao.....	10
<b>Simplified Design Method of GRP Pipes in Different Design Service Life</b> J.Z. Chen and S.R. Zhu.....	15
<b>Optimization of Preparation and Characterization of the Plasmid DNA from Newcastle Disease Virus Encapsulated in Chitosan Nanoparticles</b> C. Shi, Y.W. Sun, G.Y. Rong, Y. Zhang and K. Zhao.....	19
<b>A Bending Sequence Planning Algorithm Based on Multiple-Constraint Model</b> Z.Y. Zhao, L.C. Zhang and Y.S. Shi.....	26
<b>Effects of the Stress Concentration and Plane Stress on the Essential Work of Fracture (EWF) Method</b> C. Zhao, X. Chen, C.S. Zhou and H.M. Wang.....	32
<b>The Fabrication and the Properties of Gradient Porous Titanium by Centrifugal Deposition and Sintering</b> G.J. Cao, W.J. Xu, Y.C. Feng and W.Y. Tang.....	38
<b>Acid Fracture Conductivity Behavior of Tahe Carbonate: High Closure Stress, Long-Term Conductivity, and Composite Conductivity</b> J.N. Mou, M.T. Yao and K.X. Zheng.....	44
<b>Effective Atomic Displacements in <math>\alpha</math>-Zirconium under Neutron Irradiation</b> V. Svetukhin and M. Tikhonchev.....	52
<b>Ultrasonic Cavitation Based Processing of Metal Matrix Nanocomposites: An Overview</b> S. Sardar, S.K. Karmakar and D. Das.....	58
<b>Numerical Simulation of Foam Structure Formation and Destruction Process Using Phase-Field Model</b> T. Uehara.....	65
<b>Analysis on Magnetoelectric Responses of Porous Polypropylene and Polyvinyl Chloride after High-Voltage Corona Discharge</b> J.W. Zhang and R. Belouadah.....	70
<b>Electroforming Solution Component Performance Impact Study of the Electroformed Layer</b> X. Zhang, B.L. Li, D.X. Kong and H. Jiang.....	75

## **Dynamic Recrystallization Microstructure Grain Size Prediction of 7075 Aluminum Alloy Piston Prepared by Isothermal Extrusion**

Y. Wu, W.L. Chen, Y. Gao and R. Zhou ..... 81

## **Chapter 2: Artificial Intelligence and Data Mining, Data, Image and Signal Processing, Intelligent Automation and Control**

### **Automatic Badminton Action Recognition Using RGB-D Sensor**

H.Y. Ting, K.S. Sim and F.S. Abas ..... 89

### **Kinect-Based Badminton Action Analysis System**

H.Y. Ting, K.S. Sim and F.S. Abas ..... 94

### **A Review of Hot Topic Detection and Tracking Technology**

G.X. Xu, Y. Hai, Y.X. Feng, S.Z. Xie and J.L. Zhang ..... 100

### **Study on the Improvement of TFIDF Algorithm in Data Mining**

H.F. Sun and W. Hou ..... 106

### **A New Protocol for Secure Distributed Multiplication of Two Polynomial Shared Values**

X.N. Hao, X.M. Wang and L.Q. Deng ..... 110

### **Human Posture Recognition Based on DAG-SVMS**

S.M. Wang, Y. Gao and L. Luo ..... 117

### **The Design and Implementation of Astronomical Multi-Catalog Storage and Cross-Match Based on Hadoop**

X.X. Zhang and J.C. Pan ..... 121

### **The Multi-Scale Hough Transform Lane Detection Method Based on the Algorithm of Otsu and Canny**

Y. Chai, S.J. Wei and X.C. Li ..... 126

### **Design of Heart Sound Analyzer**

L. Zhang ..... 131

### **The Application of Wavelet Denoise in Sampled Grating Comb Filter**

D.D. Zhu and H.F. Wang ..... 135

### **A Real-Time Image Mosaic Algorithm Based on Förstner Operator**

Y.L. Liu, L.Y. Gu and J.J. Li ..... 139

### **Simulation for SAR Feature Image of Planar Structures**

Q.C. Tao ..... 145

### **The Design of Static Image Compression System**

L. Zhang ..... 150

### **A Study on Velocity Decay of $\phi 6\text{mm}$ Tungsten Ball Fragment**

C.L. Yi, D.R. Kong, Y.Z. Li, L.P. Li, J.Q. Zhang and J. Wang ..... 154

### **Autonomous Underground Azimuth Measuring Drive System Based on MEMS Gyroscope**

Y. Liu, X.C. Xu, Q. Liu and T.H. Ji ..... 159

### **Development of Driver Model for Lane Keeping Manoeuvre**

P.P. Em, K.S. Sim and E.K. Wong ..... 165

<b>Solving the Problem of UAV Air Combat Game Based on Differential Variational Inequality and D-Gap Function</b>	
G.Y. Xu, P. Li and B. Zhou .....	172
<b>Study on Diffusion Simulation and Visualization of Hazardous Chemicals's Disclosure Based on 3D GIS</b>	
T.G. Shi, F. Ding, Y. Wan, Y. Lei and W.H. Song .....	178
<b>A New Model-Free Predictive Control Method Using Input and Output Data</b>	
S. Yamamoto.....	182
<b>Influence of Engine Cabin Simplification on Aerodynamic Characteristics of Car</b>	
X.J. Hu and J. Chang.....	188

### **Chapter 3: Computer Science and Information Technologies**

<b>Design and Implementation of JMF-Based VOD System</b>	
N. Qiu and H.C. Du.....	197
<b>Research and Implementation of Internet Topology Based on BRITE</b>	
C. Liu, J.C. Pan and G.Z. Ge .....	203
<b>Research and Implementation of Internet Topology Based on AB Model</b>	
G.Z. Ge, J.C. Pan and C. Liu .....	207
<b>Using FPGA to Accelerate Deduplication on High-Performance SSD</b>	
Z.G. Chen, N. Xiao, F. Liu, Y.X. Xing and Z. Sun .....	212
<b>Research and Prototype Implementation of Network Group Sentiment Analysis Based on Topic</b>	
Y.H. He, Y.R. Yang, Y.Z. Qian and J. Li.....	218
<b>"The Last Kilometer" Network Optimization Research of E-Commerce</b>	
Y.F. Zhuang and Z.W. Wang.....	224
<b>Optimization Study of Personalized Information Recommendation Model Based on Tensor Decomposition</b>	
H.F. Sun and X.D. Liu .....	228
<b>A Modified Back Propagation Algorithm of Neural Network with Global Optimization</b>	
J.J. Li and Z. Cui.....	232
<b>Study on the System Simulation Model of Personalized Information Recommendation</b>	
H.F. Sun and F. Wang.....	239

### **Chapter 4: Electrical and Magnetoelectric Applications**

<b>Investigation of Surface Potential Decay Behavior of Insulator Polymers after High-Voltage Poling</b>	
J.W. Zhang and R. Belouadah .....	245



<b>Analysis on Magnetoelectric Effect Induced by Torsion Moment in Self-Powered Piezoelectric Material</b>	
J.W. Zhang and R. Belouadah .....	249
<b>A Prototype of Magnetic Actuator Based on Generalized Magnetoelectric Phenomena: Experiments and Analysis</b>	
J.W. Zhang and R. Belouadah .....	253
 <b>Chapter 5: Advanced Technologies in Social, Education, Economics, Statistics and Management Applications</b>	
<b>The Influence on the Consumption Level of Tibetans</b>	
S.T. Liu, H. Liu and G.L. Long .....	261
<b>Vocational Education of Girls in Rural Areas Analysis - Based in Hebei</b>	
H. Liu, S.T. Liu and G.L. Long .....	266
<b>Research on Collaborative Design System of Green Building Information Model</b>	
X.S. Luo, M. Xu and C. Gan .....	272
<b>The Study of the Framework of an Intelligent Decision Supporting System for Haze Condition</b>	
D.D. Wang, X.H. Yang and Q. Kan.....	277
<b>A Study on Teaching Method of Cultural Cognition Courses Based on Network Teaching Platform - Take a Survey of Japan as an Example</b>	
X.J. Shi and L.J. Wang .....	282
<b>The Study on the Bank Crisis Pre-Warning System Based on Balanced Scorecard</b>	
W. Wang .....	286
<b>On Internet-Based Translation Teaching Mode Reform and Practical Translation Talents Cultivation</b>	
D.F. Ge and X.H. Yu .....	290
<b>The Research of the Interactive Translation Teaching Method Based on the Network - Taking the Reform of Translation Teaching in NCIST as the Example</b>	
X.H. Yu and L.M. Hou .....	296
<b>Computer Technology in Tropical Areas of China Elementary Student Body Function Research and Application Research of Health Interventions</b>	
Q.H. Lin, W.T. Hao, Y.L. Zhang and Y. Jiang.....	300
<b>Functional Action Screening (FMS) Girls Pilates for Ordinary Colleges and Universities Study the Guidance of Design</b>	
C.L. Xi, W.Z. Li and Y.H. Cao.....	303
<b>The Application of Lean Management in Power Engineering Projects</b>	
W. Li and M.Y. Ma.....	309
<b>Keyword Index</b> .....	317
<b>Author Index</b> .....	321

## **CHAPTER 1:**

# **Materials Science and Materials Engineering**



# Modifications in Water Sorption Isotherms of Cement Mortars Caused by Carbonation: Effects of Cycles

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Laboratory of Civil Engineering and Mechanical Engineering, National Institute of Applied Sciences, 35000 Rennes, France

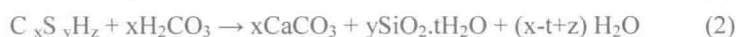
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**Keywords:** Water adsorption; Carbonation; Pore size distribution; Humidification drying cycles

**Abstract.** This work aimed to examine the influence of carbonation on the water sorption isotherms of cement materials. Two types of normalized CEM I and CEM II Portland cement mortars were carbonated at 20°C, 65% relative humidity and 20% of CO<sub>2</sub> concentration for 32 days. The pore size distributions determined from the water sorption showed a reduction in microporosity and a slight increase in the mesoporosity. The pores clogging due to formation of calcium carbonate was highlighted by the reduction of the quantity of adsorbed water and the decrease in the hysteresis isotherms. We also studied the coupling effect between carbonation and humidification-drying cycles. The results of this study also indicated that the humidification-drying cycles coincide only from the second cycle because of a difficult evacuation of water during desorption, even at low humidity.

## Introduction

Carbon dioxide from the air can react with the portlandite Ca(OH)<sub>2</sub> and the calcium silicate hydrate C-S-H in concrete to form calcium carbonate CaCO<sub>3</sub>. This process is called carbonation, which is a natural aging process for all cement materials. This transformation is accompanied by a decrease in pH. The principle reactions are:



The progress of these carbonation reactions causes modifications in the microstructure, which is highlighted by various parameters such as variations in water content. The carbonation reactions of portlandite and C-S-H indicate that the process releases part of the water of hydrate structures. Therefore, the carbonation at first induces a local accumulation of moisture in the pores. Pihlajavaara [1] observed an increase in the quantity of evaporable water content in carbonated materials by drying at 105°C. This released water can certainly participate in the composition of the interstitial solution and contribute to the transport of corrosive agents.

This excess water will be evacuated after a certain time and the material will be in equilibrium with environmental moisture. Houst [2] and Johannesson and Utgenannt [3] observed that, at a given relative humidity, the carbonated mortar contains less water than the non-carbonated one. This decrease in water content is probably due to the reduction in porosity as a result of carbonation. Thus, Meyer [4] found that carbonated concretes dried faster than non-carbonated ones.

Some authors present the water activity by the saturation level and they also find that, for a relative humidity equal to 65%, the saturation ratio of non carbonated material is higher than that of the carbonated material, which is coherent with the reduction of porosity after carbonation [5], [6].

However, the concrete structures in-situ is also subjected to humidification-drying cycles, which results in a much more complicated water activity. This has motivated us to perform this work in order to approach the laboratory results to in-situ results. In this work, the water activity of non-carbonated and well-carbonated cement mortars were firstly studied using water vapor adsorption. Then we examined the effects of humidification-drying cycles on the water activity. The pore size

distributions were also calculated from water vapor adsorption desorption isotherms in order to study the micro structural changes caused by carbonation.

### Materials and methods

**Mortar.** The cements used in this study were:

CEM I 52.5 N PM-ES-CP2

CEM II / B-M (LL-V) 32.5 R

These cements were fabricated by Lafarge company in accordance with European norm EN 197-1 "Cement – Part 1: Composition, specifications and conformity criteria for common cements".

The CEM I and CEM II mortars were prepared by mixing respectively the cements CEM I and CEM II with French normalised sand certified in accordance with norm EN 196-1 and ISO 679:2009. The water/cement and sand/cement ratios were 0.5 and 3, respectively. At the end of the mixing, the mortar was placed in cylindrical moulds ( $\varnothing = 40$  mm,  $h = 60$  mm) and stored in a humid chamber in order to avoid precocious desiccation in the vicinity of the surface exposed to drying. The samples were demoulded after 24 hours and then cured in water for 90 days in a humid chamber (20°C, 100% relative humidity). The cure in water prevents the natural carbonation during storage.

Bier et al. [7] observed the creation of macroporosity after carbonation of a mortar, which was not rich in portlandite and contained fly ash. The CEM II is poor in portlandite in comparison with CEM I, and therefore the CEM II was also chosen for this study because we want to ensure significant changes not only in the micro porosity but also in the domains of meso and macro pores.

**Pre-treatment.** We employed a method of pre-treatment basing on the work of Thierry [8] and Parrot [9]. The specimens are dried at 105°C until constant weight. Finally, the specimens are placed for 7 days in a climatic chamber at 65% relative humidity. The advantages of this method are to homogenise the internal humidity in the samples and to prevent the natural carbonation during the pre-treatment process.

**Carbonation test.** To implement the test, the specimens were subjected to CO<sub>2</sub> in an environmentally controlled chamber at 20°C, 65% relative humidity and 20% CO<sub>2</sub> concentration for 32 days to ensure a complete carbonation. The carbonation device consists of a climatic chamber Vötsch VP1300 connected to a CO<sub>2</sub> tank (Figure 1). The concentration of CO<sub>2</sub> in the chamber is controlled by an automatic CO<sub>2</sub> regulator.

Samples collected in carbonated and non-carbonated specimens were then grinded into powders to 80 µm using a mortar – pestle and a sieve, and subsequently used for water adsorption.

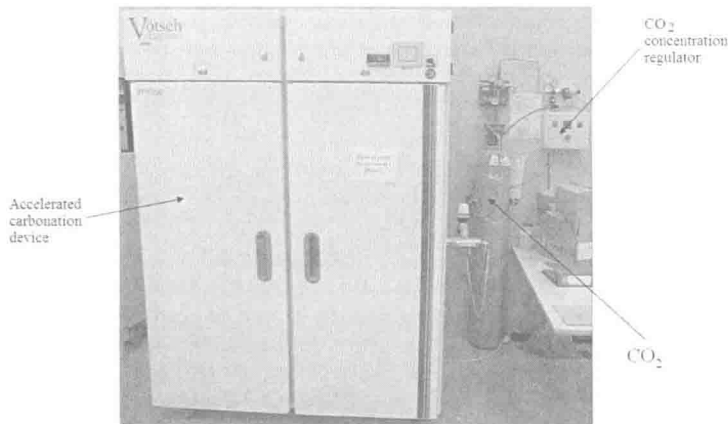


Fig.1 Accelerated carbonation test device

**Water adsorption.** It is well known that cement paste has a multiscale structure that spreads from the nano- and microstructure to the mesoscopic scale and then to the macroscopic scale, which is visible to the naked eye. The nano- and microscopic scales consist of sheets of C-S-H. These sheets associated with packets are called grains and constitute a second level of the observation, which is the mesoscopic scale.

We examined the water vapor adsorption on grinding powders originating from the test samples to study the microscopic scale. For this test, we used a humidification regulator connected to the microbalance of the thermal analysis Mettler Toledo machine (Figure 2). The regulator controlled automatically the temperature at 20°C and changed the relative humidity every one hour.

The sorption steps are: 5%-10%-20%-30%-40%-50%-60%-70%-80%-90%-95%.

The desorption steps are : 95%-90%-80%-70%-60%-50%-40%-30%-20%-10%.

Change in mass of the sample was automatically followed by the microbalance (Figure 3). This method is also known as the dynamic adsorption.

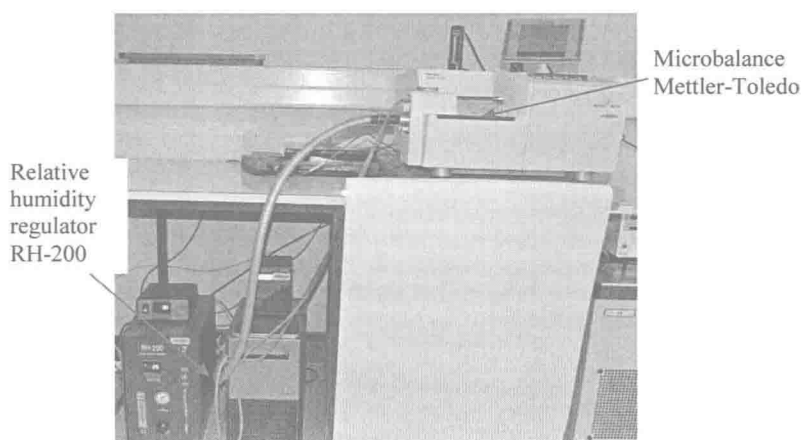


Fig.2 Water sorption device

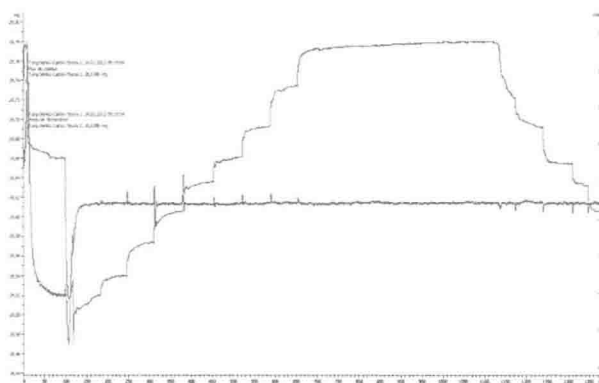


Fig.3 Example of a dynamic sorption test

## Results and discussion

The tests were carried out on the powders obtained by grinding samples to 80  $\mu\text{m}$ . In this case, the macroporosity is virtually eliminated and the adsorption takes place essentially in the micropores and mesopores. To study the influence of humidification-drying cycles on the isotherms, we stop the desorption at 10% relative humidity and then proceed the second sorption cycle. The results are presented in Figure 4 for CEM I mortar and in Figure 5 for CEM II mortar. Firstly, we note that the

sorption branch of the second cycle is different from that of the first cycle. Secondly, the desorption of the second cycle coincides with the desorption of the first cycle. From this moment, for any cycle, the adsorption branches are coincident with the desorption branches.

As the material was dried at 105°C to constant weight before testing, water molecules in inter layers of C-S-H were indeed evacuated during this deep drying. Then, during the first adsorption cycle, the water molecules returned to the internal structure of the C-S-H. Desorption at 10% relative humidity could not exclude these internal water molecules of C-S-H. This explains why the cycles coincide only from the second adsorption.

Changes in isotherms curves are similar for both types of mortar and can be summarized as follows:

On the curve of the non-carbonated sample, water is continuously adsorbed until 90% relative humidity. On the contrary, this water is desorbed with difficulty during the desorption step. Only at relative humidity below 20% that much of the water is desorbed. This results in a significant hysteresis of the isotherms.

On the curve relative to the carbonated sample, a hysteresis is also observed but less marked than that of the non-carbonated sample. This is because the carbonation develops from the inner wall of the pores and eventually tends to homogenize the pore radius whose initial morphology is ink-bottle type (Figure 6).

Comparing the two types of sample (carbonated and non-carbonated), we note that the isotherms of carbonated samples are generally below the isotherms corresponding to the non-carbonated samples. Thus, at the same relative humidity, the carbonated mortars adsorbed less water than the non-carbonated ones. This is explained by the gradual clogging of the pores by the products of carbonation ( $\text{CaCO}_3$ ).

We present in Figures 7 and 8 the evolutions of the pore size distributions curves recorded from the reference samples (non-carbonated) and those obtained after carbonation. We observe for both types of mortar a reduction in the microporosity ( $r < 2 \text{ nm}$ ) and a small variation in the mesoporosity between 2 and 4 nm. The relative changes in porous volume are almost zero beyond 5 nm. The water vapor adsorption desorption isotherms seem therefore poorly adapted to effectively characterize the pores size distribution beyond 5 nm. This is because the steam condensation occurs rapidly in these mesopores.

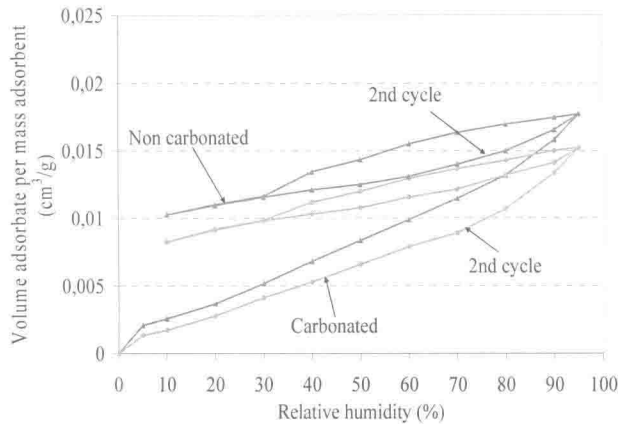


Fig.4 Cycles of water adsorption-desorption isotherms of CEM I mortars

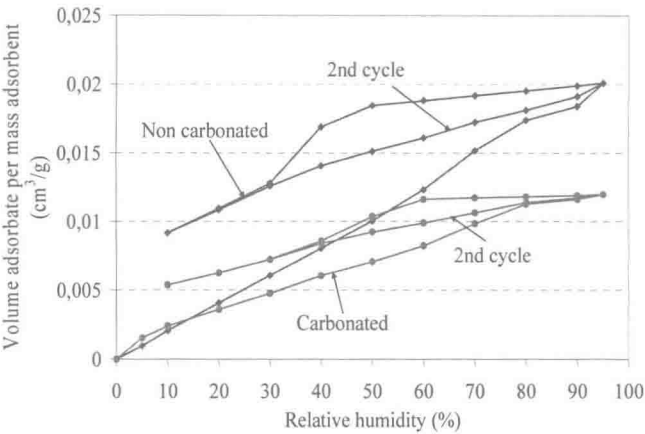


Fig.5 Cycles of water adsorption-desorption isotherms of CEM II mortars

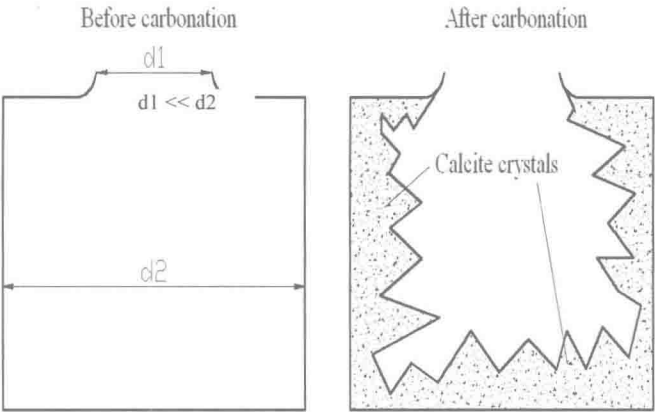


Fig.6 Schematic of ink-bottle pores before and after carbonation.

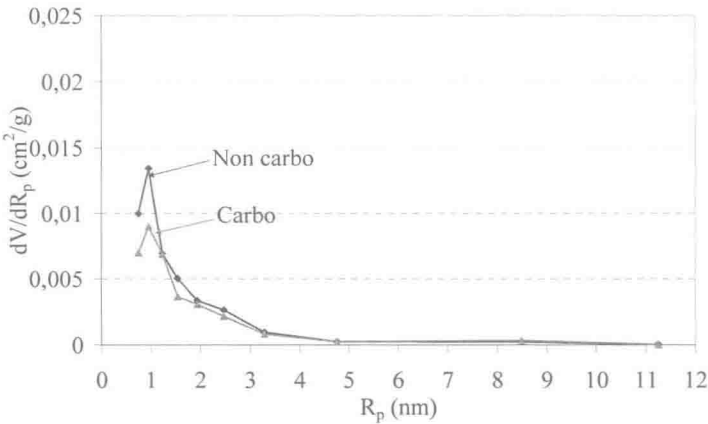


Fig.7 Pores size distribution of CEM I mortar.



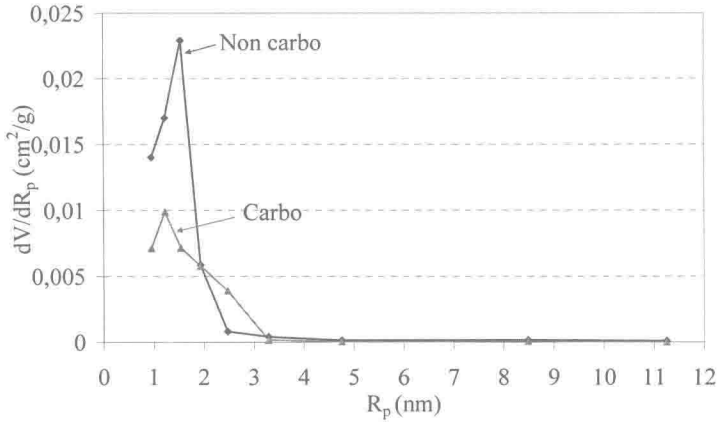


Fig.8 Pores size distribution of CEM II mortar.

## Conclusions

This work was devoted to the study of changes in water adsorption desorption isotherms and in pores size distribution caused by carbonation of Portland cement mortars. The water adsorption results indicated that:

Firstly, there is a decrease in the volume of micropores with radius smaller than 2 nm, which is due to the shrinkage in the C-S-H structure.

Secondly, there is a small increase in the volume of mesopores with radius over 2 nm, which highlighted the cracks caused by the carbonation shrinkage.

Thirdly, carbonated mortars adsorb less water than non-carbonated ones because of pores clogging due to calcium carbonate crystals formed during carbonation. This also induced a homogenization in internal radius of ink-bottle pores, which was highlighted by the reduction in the hysteresis of the isotherms.

Finally, the results indicated that the water molecules evacuated during drying at 105°C return into the C-S-H microstructure during adsorption and can not be desorbed afterward at low relative humidity even under 10%. This explains why the humidification-drying cycles coincide only from the second cycle.

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