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Non-destructive evaluation of internal sulphate attack in cement-based materials applying non-linear ultrasonic techniques

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Outline

1. **Introduction**
2. **Materials and methods**
3. **Results and discussion**
4. **Conclusions**

Introduction

Sulphate attack

- It is one of the most harmful aggressive attack to which cement-based materials can be exposed
- Complex mechanism → Involves different chemical reactions between components of cement paste microstructure and sulphate ions.
- Expansive products are formed:
  - Volumetric strains → Microcracking and loss of pore refinement
  - Loss of strength and durability in the cement-based materials

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Non-destructive techniques

- Their application for characterizing the microstructure and properties of cement-based materials has become an important research field
- Useful for following the development of deleterious processes produced during the attack of aggressive substances to these cement-based materials.
- Non-linear ultrasonic (NLU) → Detecting cracks due to steel corrosion in reinforced concrete structures:
  - DOI: 10.1186/s40069-020-00432-x
  - DOI: 10.3390/MA12050813

Objetives

To study the possibility of using NLU techniques

↓

Non-destructive evaluation

↓

Initial stages of internal sulphate attack in cement-based materials

(until 100 hardening days)

Materials and methods

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Samples preparation

- Materials (cement pastes):
  - Reference specimens → CEM I 42,5 R (100%)
  - Sulphate specimens → CEM I 42,5 R (90.8%) + gypsum (9.2%) → To reach in the mixture a SO_3 context of 7% in mass according to ASTM Standard C452-02
  - Water to cement ratio = 0.5
  - Setting → UNE-EN 196-3
- Prismatic samples:
  - 25 mm x 25 mm x 285 mm.
- Storage in optimum laboratory condition

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Experimental techniques

- Mercury intrusion porosimetry
- Length change due to expansion
- Non-linear ultrasonic technique
- Linear ultrasonic pulse velocity

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Mercury intrusion porosimetry

- Poremaster-60 GT porosimeter
- Total porosity
- Pore size distributions
- Percentage of Hg retained at the end of the test
- Pieces taken from prismatic specimens

Length change due to expansion

- ASTM Standard C452-02

Linear ultrasonic pulse velocity

- Standard UNE-EN 12504-4
- Proceq Pundit Lab equipment

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Non-linear ultrasonic technique

- Two transducers → Simultaneously supply two pure tones (f_0 = 20 kHz and f_1 = 200 kHz)
- High-frequency probe signal at an amplitude of 5 V
- Low-frequency pump and acquisition of the frequency modulated signal → 16-bit ADC resolution I/O device NI-USB 6361 (sampling frequency 2 MHz)
- Pump wave signal → Amplifier FS WMA-100 and then transmitted through a Langevin transducer
- Input voltage → 140 V
- Transducers IDK09 → Emitting and receiving the high-frequency signal
- Parameter DIFA was studied in this work

Results and discussion

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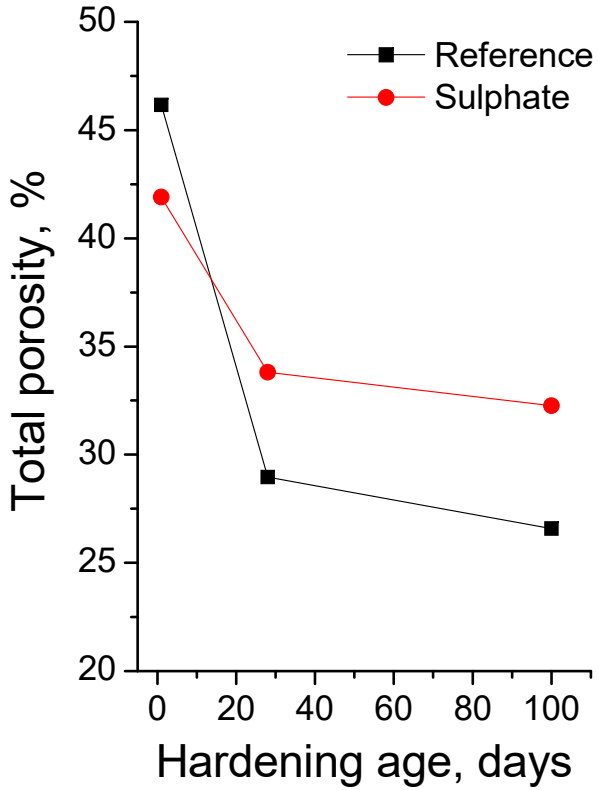
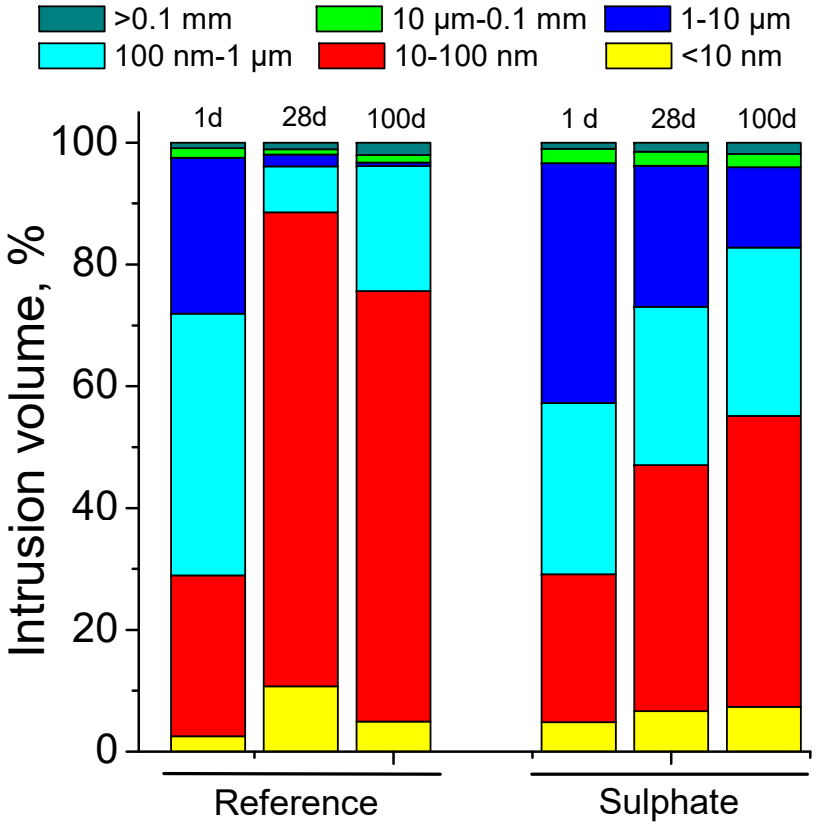
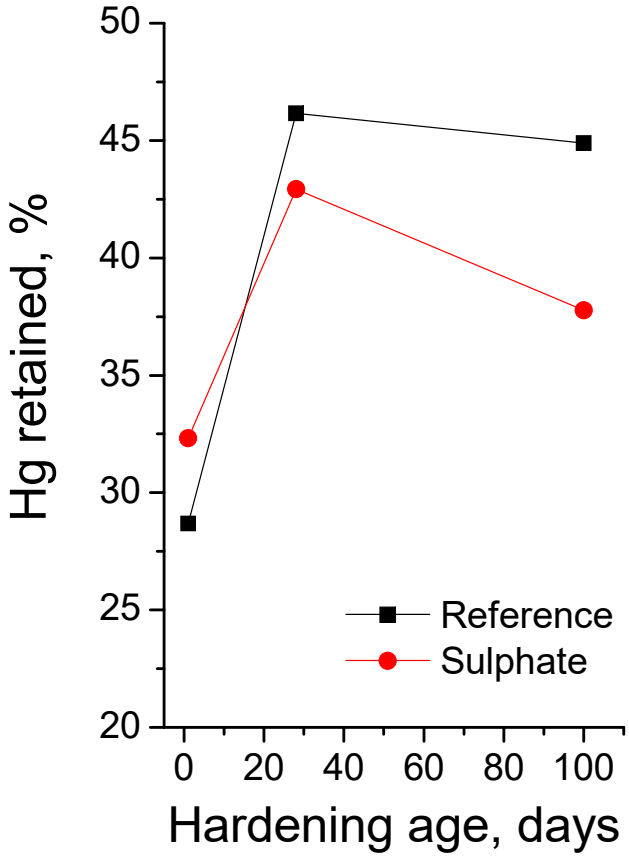
Results and discussion

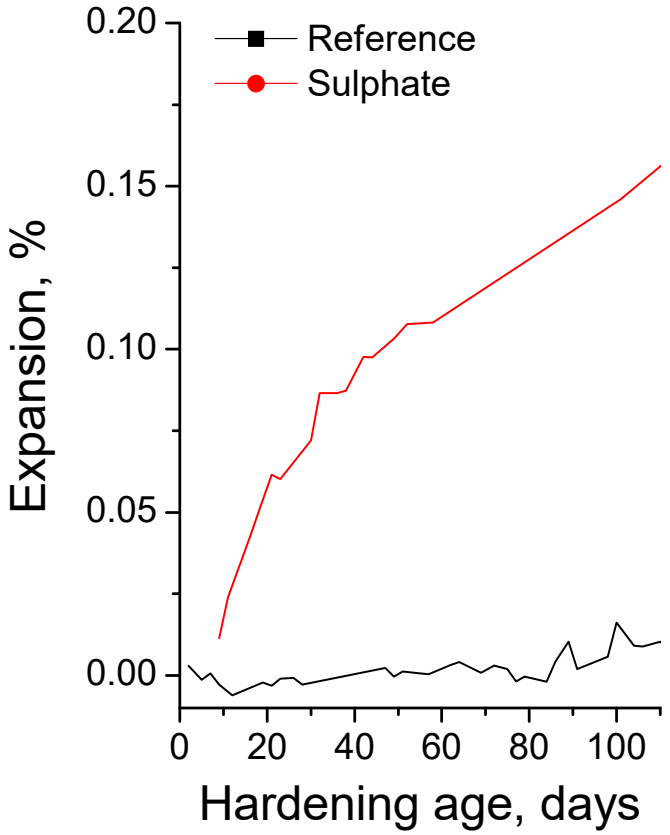




- Lowest porosity and highest pore refinemement → Reference
- Progressive pore refinement with age → Sulphate specimens
- **Combined effects of clinker hydration and initial sulphate attack**

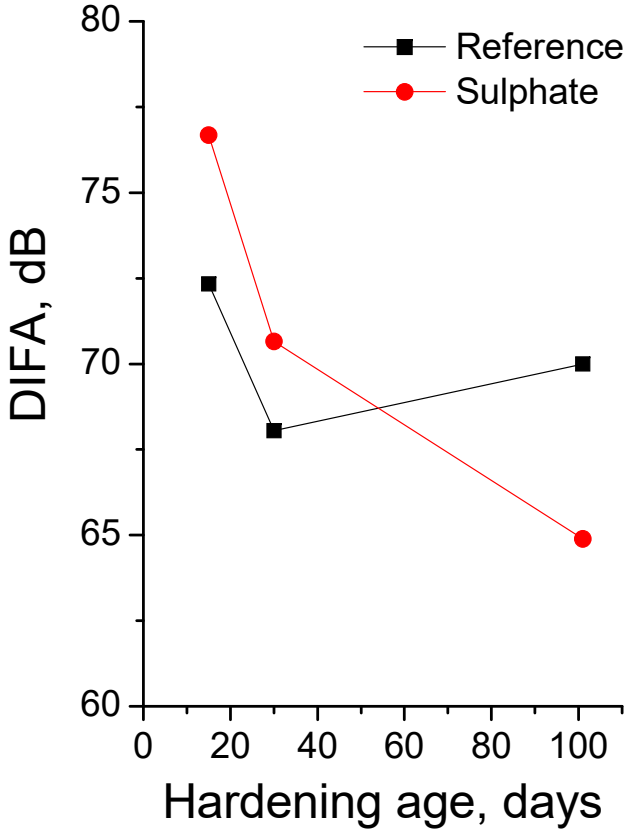
Results and discussion

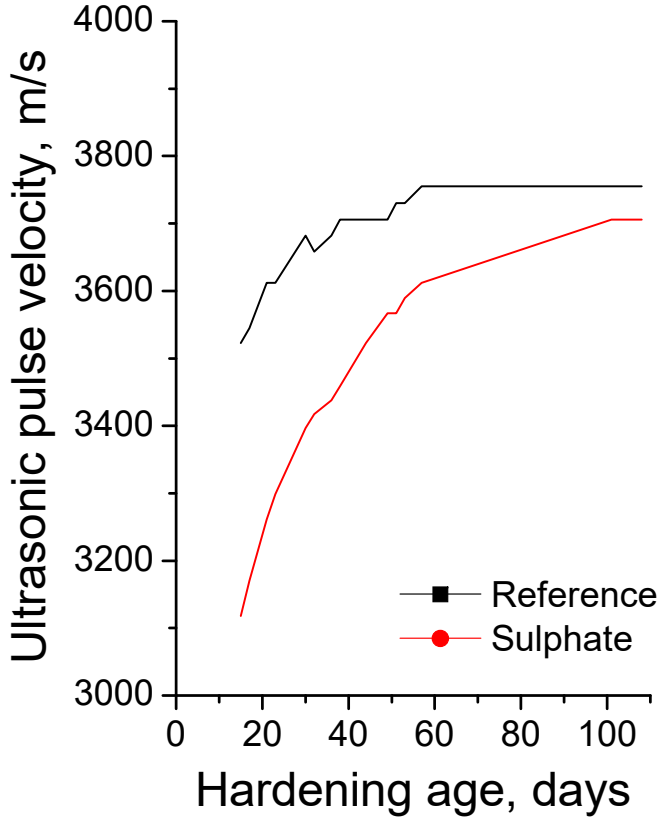




- Hg retained → Overall in keeping with pore size distributions
- Expansion for pastes subject to internal sulphate attack
- **Expansion would reveal possible microcracking development**

Results and discussion





- DIFA → System gradually turned less linear for sulphate pastes
- UPV → Coincided with PIM and not reveal effects of attack
- **NLU technique could be useful for providing information about the deleterious processes due to internal sulphate attack.**

Conclusions

Conclusions

- Reference pastes showed an important pore refinement at 28 hardening days → Microstructure development due to the hydration of clinker
- Pore size distribution of cements pastes exposed to internal sulphate attack gradually became more refined, but lower refinement compared to reference pastes → Simultaneous development of clinker hydration and sulphate attack (silting of pores and later microcracking)
- Continuous expansion for pastes subject to internal sulphate attack → Possible presence of pores already silted in which a possible formation of microcracks due to expansion could be produced.

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Conclusions

- The tendencies observed for the NLU parameter DIFA were overall in keeping with the results of the length change due to expansion.

- Linear ultrasonic pulse velocity → Progressive reduction of voids with age in the studied samples → No effects of sulphate attack in this parameter.

- In view of these preliminary results → NLU technique could be useful for providing information about the deleterious processes due to internal sulphate attack in cement-based materials → Further research would be needed for confirming this.

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