

Smart Pavement Materials with Integrated Functionalities: Sensing, Healing, Thermal Responsiveness, and Photocatalysis

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the degree of

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CERTIFICATE OF ORIGINAL AUTHORSHIP

I, Zhizhong Deng, declare that this thesis is submitted in fulfilment of the requirements for the award of Doctor of Philosophy, in the School of Civil and Environmental Engineering Technology at the University of Technology Sydney.

This thesis is wholly my own work unless otherwise referenced or acknowledged. In addition, I certify that all information sources and literature used are indicated in the thesis.

This document has not been submitted for qualifications at any other academic institution.

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List of Abbreviations

SHM	Structural Health Monitoring
TD	Traffic Detection
CB	Carbon Black
NCB	Nano-carbon black
CF	Carbon Fibre
CNT	Carbon Nanotube
MWCNT	Multi-walled Carbon Nanotube
SW	Seawater
UHI	Urban Heat Island
TC	Thermochromic
TM	Thermochromic material
PC	Photocatalytic
CFRP	Carbon fibre reinforced polymer
FRCPs	Fibre-reinforced concrete pavements
LG-FBG	Long-gauge fibre Bragg grating
EAFS	Electric arc furnace slag
GNP	Graphene nanoplate
WIM	Weigh-in-motion
ERT	Electrical resistance tomography
FBG	Fibre Bragg grating
TGA	Thermogravimetric Analysis
NMR	Nuclear Magnetic Resonance
MRK	MAS ROTOR Kit
SEM	Scanning Electron Microscopy
DTC	Derivative Thermogravimetric Curve
CH	Calcium Hydroxide
IC	Isothermal Calorimetry
ICP	Inductively Coupled Plasma

ACIS	AC Impedance Spectroscopy
AC	Alternating current
DC	Direct current
GP	General Purpose
C-S-H	Calcium Silicate Hydrate
EDS	Energy Dispersive X-ray Spectrometry
VPV	Volume of Permeable Voids
RCPT	Rapid Chloride Permeability Test
C-A-S-H	Calcium Alumino Silicate Hydrate
ML	Machine Learning
ANN	Artificial Neural Network
ANFIS	Adaptive Neuro-fuzzy Inference System
DIC	Digital Image Correlation
CT	X-ray Computed Tomography
EqDiameter	Equivalent Diameter
WD	Wet-dry cyclic self-healing curing
MSH	Magnesium Silicate Hydrate
FTIR	Fourier Transform Infrared Spectroscopy
CA	Crystalline Admixture
UV	UV-Vis Spectroscopy
RS	Raman Spectroscopy
ICG	Ion Chromatography
ITZ	Interfacial Transition Zone

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Abstract

Cement–asphalt pavements are widely used in modern transport infrastructure. Yet, their serviceability and durability are often compromised by surface cracking caused by heavy traffic loads, temperature fluctuations, and environmental degradation, particularly from urban vehicle emissions. This thesis addresses these challenges by developing and analysing multifunctional pavement materials that integrate four core functions: (1) self-sensing, (2) self-healing, (3) temperature regulation through thermochromic materials, and (4) photocatalytic air purification.

The self-sensing function supports both Structural Health Monitoring (SHM) and Traffic Detection (TD). Conventional cementitious sensors typically incorporate conductive fillers such as carbon black (CB), carbon fibre (CF), or carbon nanotubes (CNTs) to alter electrical conductivity. However, their performance is often inconsistent and difficult to control. To overcome these limitations, this thesis systematically investigates the influence of pore structure, humidity, and ion concentration on piezoresistive behaviour.

Additionally, the integration of self-healing and self-sensing functions was explored to enable real-time monitoring of micro-crack repair in cementitious composites, thereby enhancing long-term durability. The role of ion concentration in healing efficiency was also examined, offering new insights into optimising self-healing processes.

Thermochromic (TC) materials were incorporated to mitigate pavement temperature fluctuations. While previous studies largely considered individual functions in isolation, this research demonstrates the cooperative effects of combining TC coatings with self-sensing composites. The results show that thermochromic coatings significantly reduce

humidity loss, thereby enhancing piezoresistive performance while simultaneously lowering surface temperature.

The photocatalytic function was investigated as a means of reducing traffic-related air pollutants. Although electron–hole recombination typically limits photocatalytic efficiency, this thesis demonstrates that multi-walled carbon nanotubes (MWCNTs), when integrated as conductive fillers, act as effective electron acceptors. This mechanism improves charge separation and enhances photocatalytic activity. The synergy between self-sensing and photocatalytic functions was experimentally validated, underscoring the application potential of such multifunctional composites.

This thesis develops and evaluates multifunctional pavement materials that integrate self-sensing, self-healing, thermochromic, and photocatalytic properties. Novel cementitious and asphalt-based composites incorporating carbon nanotubes and seawater were designed to enhance sustainability and resilience. Experimental programs investigated mechanical, electrical, and functional behaviours using advanced techniques, including CT scanning, NMR, and spectroscopy. The results demonstrate that carbon-based additives enable reliable piezoresistive sensing, seawater enhances chloride-binding capacity, and thermochromic coatings regulate surface temperature. Collectively, these findings establish new design strategies for smart pavements that improve structural monitoring, durability, and environmental performance.

Chapter 1: Introduction

1.1 Background

Modern transportation systems are primarily constructed using two types of pavements: concrete and asphalt. Asphalt pavements, owing to their relatively low elastic modulus, are particularly susceptible to thermally induced strain during summer. This necessitates thicker asphalt layers to withstand high service temperatures (Denneman, 2013; Denneman & Lam, 2015). Moreover, the design and testing temperatures commonly used for asphalt pavements do not always reflect actual field conditions (Austroads, 2012). Establishing a consistent relationship between traffic conditions and pavement responses also remains challenging due to insufficient performance data (Yousefdoost et al., 2018). These limitations underscore the urgent need for long-term monitoring of pavement responses and real-time traffic detection.

Concrete, in contrast, is globally valued for its durability, cost-effectiveness, and ease of construction. Variants such as recycled concrete, ultra-high-performance concrete, and lightweight concrete have been developed to meet specific engineering requirements. Nevertheless, concrete is subject to deterioration, deformation, and cracking, which compromise its serviceability and durability. Environmental fluctuations and mechanical loading accelerate this degradation, leading to structural deficiencies, economic losses, and safety risks. In response, researchers have proposed advanced alternatives to traditional materials. Among these, self-sensing concrete has attracted particular attention as an intelligent material capable of automatically monitoring structural and traffic conditions in real time. Its integration into infrastructure enhances functionality and supports the broader development of smart structures (ASTM C642).

Self-healing concrete has also emerged as a promising solution to improve structural durability by sealing cracks. Autogenous healing, which occurs naturally in moist environments, enables the precipitation of calcium carbonate to close fine cracks (De Rooij et al., 2013). However, the process is poorly understood and largely ineffective for larger cracks (Edvardsen, 1999). To extend healing capacity, researchers have investigated fibre reinforcement to control crack width and the incorporation of healing agents within the concrete matrix (Yang et al., 2009). These approaches have substantially enhanced performance, making engineered self-healing systems a well-accepted research direction.

Temperature regulation represents another critical consideration in pavement design. Dark pavements, particularly asphalt, absorb and store large amounts of solar energy, exacerbating the Urban Heat Island (UHI) effect (Kertesz & Sansalone, 2014). Previous studies demonstrated that reflective and blocking coatings can effectively reduce surface heat absorption (ITO, 2006).

In addition, vehicle emissions significantly contribute to atmospheric pollution, driving ozone depletion, the greenhouse effect, and acid rain (Perez-Nicolas et al., 2017). Photocatalytic (PC) technology, primarily based on titanium dioxide, has emerged as an effective strategy for mitigating vehicular air pollution (Jimenez-Relinque et al., 2015). The PC technology utilised titanium dioxide (TiO_2) as the major material, as has been researched in the past decade (Poon & Cheung, 2007). Applied to both concrete and asphalt pavements, TiO_2 -based photocatalysts have demonstrated measurable air-cleaning potential.

Despite progress in understanding these individual functional mechanisms, research into the integration of multiple functionalities within a unified pavement material system

remains limited. In many cases, the incorporation of conductive fillers for self-sensing may adversely affect mechanical durability, thermal behaviour, or photocatalytic efficiency, highlighting potential trade-offs among different functional requirements. Conversely, certain mechanisms may exhibit cooperative interactions, such as enhanced electron transport pathways that simultaneously influence sensing and photocatalytic performance. However, the complexity associated with balancing structural integrity, environmental durability, and multifunctional performance presents significant unresolved challenges. Therefore, a systematic investigation into the cooperative and competitive effects among self-sensing, self-healing, thermal responsiveness, and photocatalysis is essential for advancing next-generation smart pavement technologies.

1.2 Research Problem Statement

While considerable advances have been made in developing functional pavement materials, current approaches remain largely single-function oriented, lacking a unified framework for integrating multiple performance-enhancing mechanisms. The absence of systematic investigations into the cooperative and competitive interactions among functional components presents a fundamental barrier to the realisation of next-generation smart infrastructure materials. This thesis addresses this critical research problem by exploring the design principles and performance implications of multifunctional pavement systems that integrate sensing, environmental responsiveness, and durability enhancement within a coherent material framework.

1.3 Research objectives

Previous research on self-sensing sensors has focused predominantly on conductive fillers in cementitious composites, with limited attention given to asphalt-based systems. Study (Dong, Li, Lu, et al., 2019) examined factors such as humidity, internal structure, and ion concentration in pore solutions, highlighting their influence on self-sensing performance.

However, the integration of self-sensing with advanced digital models, such as machine learning, has scarcely been explored. Similarly, while self-healing, thermochromic, and photocatalytic functions have each been studied individually, their cooperative behaviour within multifunctional composites remains poorly understood.

This research aims to bridge these knowledge gaps by conducting a systematic investigation into the mechanisms and synergies of multifunctional pavement materials.

The specific objectives are as follows:

- (1) To review the research progress of those four functions in previous studies and identify the limitations of the application potential of multifunctional composites.
- (2) To investigate factors such as humidity, ion concentration, and pore structure on the piezoresistive performance of cement/asphalt-based self-sensing sensors.
- (3) Mixing with seawater (SW) and analysing the SW influence on self-sensing and self-healing performance. Moreover, to understand the co-operation between self-sensing and self-healing functions.
- (4) To develop self-sensing and thermal responsiveness composites and identify the application potential of self-sensing and thermochromic (TC) multifunctional materials.
- (5) To evaluate the photocatalytic performance of the photocatalyst and analyse the photocatalytic material performance after mixing with conductive fillers. Also, to analyse the application potential of specimens that combine self-sensing and photocatalytic functions.
- (6) Experimental investigations are complemented by MATLAB-based algorithm to support the interpretation of multifunctional material behaviour.

1.4 Outline of Thesis

Chapter 1 introduces the research background and outlines the motivation for developing multifunctional pavement materials. It defines the research objectives and presents the overall structure of the thesis, establishing the framework for the subsequent investigations (based on Publication 1).

Chapter 2 reviews the state of the art in conductive fillers, self-healing agents, thermochromic materials, and photocatalysts, with a focus on their applications in pavement engineering. It highlights that existing studies predominantly investigate these functionalities in isolation, with limited understanding of their interactions within a multifunctional system. These limitations define the key research gaps addressed in this thesis and provide the foundation for the subsequent experimental investigations (based on Publication 1).

Chapter 3 This chapter investigates the piezoresistive and mechanical properties of self-sensing cementitious sensors incorporating bitumen emulsion. It addresses the challenge of achieving reliable sensing performance without compromising structural integrity in conductive cementitious systems. The findings contribute to the development of stable self-sensing materials and provide a foundation for subsequent studies on multifunctional pavement systems (based on Publication 2).

Chapter 4 investigates seawater-mixed cementitious sensors, focusing on the influence of ion concentration on piezoresistive behaviour and the resistance of bitumen–cement composites to chloride diffusion. It addresses the interaction between electrical performance and durability in conductive cementitious systems. The findings provide important insights into the design of multifunctional materials for harsh environmental conditions (based on Publications 4 and 5).

Chapter 5 explores the relationship between strain and piezoresistive response in asphalt-based self-sensing sensors. It further incorporates machine learning algorithms to predict sensor behaviour, linking experimental observations with data-driven modelling. The findings enhance the understanding and predictive capability of self-sensing pavement systems (based on Publication 3).

Chapter 6 evaluates the influence of pore structure on asphalt-based self-sensing composites incorporating carbon fibres. It examines how microstructural characteristics affect the formation and stability of conductive networks and, consequently, piezoresistive behaviour. The findings provide fundamental insights into the structure–property relationship of self-sensing materials.

Chapter 7 investigates the cooperative effects of self-sensing and self-healing functions in seawater-mixed cementitious composites. It examines the interaction between functional mechanisms and their combined influence on material performance under aggressive environments. The findings provide important insights into the integration of multiple functionalities in smart pavement materials (based on Publication 7).

Chapter 8 examines the integration of thermochromic coatings with self-sensing cementitious composites, including the influence of seawater on piezoresistive behaviour. It explores the combined effects of thermal responsiveness and sensing functionality under varying environmental conditions. The findings further advance the understanding of multifunctional integration in smart pavement materials (based on Publication 6).

Chapter 9 assesses the synergy between self-sensing and photocatalytic functions and evaluates their application potential in pavement materials. It examines the interaction between sensing capability and environmental functionality, highlighting their combined

performance. The findings further demonstrate the feasibility and benefits of integrating multiple functions in smart pavement systems (based on Publication 8).

Chapter 10 summarises the main contributions of this research and highlights how the individual studies collectively advance the understanding of multifunctional pavement materials. It also provides recommendations for future research, including potential directions for further integration and optimisation of functional performance.

Chapter 2: Literature review

2.1 Introduction

The concept of smart pavement has been proposed to enhance the functionality and durability of transportation infrastructure (Han et al., 2017). As illustrated in Fig. 2.1, smart pavements integrate four advanced functions, self-healing, self-sensing, thermochromic responsiveness, and photocatalysis. While certain functional cementitious composites have already been evaluated, many innovative functions remain underexplored (Segundo et al., 2021). Recent studies have examined the role of nanotechnology in pavement engineering, the performance of flexible and rigid pavements, and the mechanical behaviour of road infrastructure (Faruk et al., 2014; Steyn, 2011; Steyn, 2009; Steyn, 2008). Increasingly, functions such as photocatalysis, thermochromism, self-healing, and wettability control have attracted significant research attention (Homem et al., 2017; Kempieński et al., 2017; Nascimento et al., 2012; Rocha et al., 2019; Rocha Segundo et al., 2018; Tadanaga et al., 2000; Xu et al., 2018; Henglong Zhang et al., 2021).

The objective of this chapter is to critically review the development of these four functions and to evaluate their potential applications in pavement engineering.

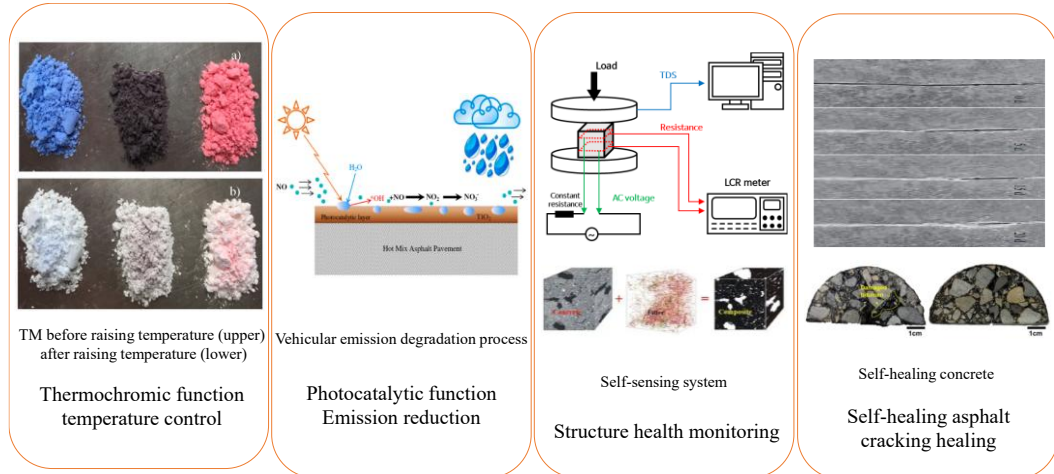


Fig. 2.1 Relationship between the smart pavement and functions (TM represents thermochromic material).

2.2 Self-healing agents and mechanisms

Cracking is widely recognised as an indicator of structural distress, typically caused by mechanical loading and environmental exposure. Such degradation reduces infrastructure durability and substantially increases maintenance costs (Ameri et al., 2011; Li et al., 2016). Self-healing function is one option to address the cracking issue. Previous studies of self-healing materials are summarised and discussed below. Section 2.2 will discuss different self-healing agents and healing methods. Section 2.7.1 will introduce the self-healing performance and application of the self-healing function.

2.2.1 Bio-based self-healing agent

Among the most widely studied self-healing agents are bacteria-based systems, which exhibit a remarkable capacity to repair relatively large cracks, particularly in lightweight composites (Luo et al., 2015). Lightweight aggregates have been shown to facilitate bacterial transport, thereby enhancing the durability and healing performance of lightweight aggregate concrete structures (Tziviloglou et al., 2016). Given that the internal environment of concrete is inherently alkaline (Erşan et al., 2015; Siddique & Chahal, 2011), the bacterial strains employed must be able to withstand such conditions

during experimental applications. Upon crack formation, bacteria become activated by exposure to oxygen, subsequently precipitating calcium carbonate that seals the fissures (Vijay et al., 2017). Once healing is complete, the bacteria revert to a dormant state. Previous studies have demonstrated that cementitious materials can encapsulate bacterial spores—highly resistant structures formed under adverse conditions—thereby increasing bacterial density compared with uncoated spores. Nevertheless, despite this protective mechanism, bacterial viability continues to decline over time within the cementitious matrix (D.J., 2021). Overall, the incorporation of bacterial healing agents has been shown to significantly enhance the self-healing efficiency of cementitious composites.

2.2.2 Polymeric healing agent capsules

Polymeric capsules have been extensively employed as self-healing carriers in cementitious composites, where they serve to bond and repair cracks. Among the various designs, spherical microcapsules and cylindrical fibres are the most widely investigated forms (Dong et al., 2016; Hilloulin et al., 2015; Pang & Bond, 2005). Recent research has primarily focused on refining organic spherical microcapsules, while other studies have examined cylindrical capsules as reservoirs for healing agents. Beyond these two types, a range of alternative encapsulation materials has also been explored for self-healing applications (Araújo et al., 2018; Lv et al., 2016; X. Wang et al., 2017). In particular, poly(methyl methacrylate) capsules fabricated from glass were investigated as a promising system for enhancing healing performance (Araújo et al., 2018).

2.2.3 Self-healing in asphalt concrete

Asphalt pavements are among the most widely used materials in transportation infrastructure. However, climate change and increasing traffic loads have made cracking the primary factor contributing to their loss of durability (Li et al., 2016). The resulting need for frequent maintenance not only elevates costs but also intensifies greenhouse gas

emissions associated with asphalt production and application (Rubio et al., 2012). To address these challenges and extend service life, the integration of self-healing functions has gained significant attention (Li et al., 2016; Tabaković & Schlangen, 2015). Although asphalt possesses an inherent ability to repair microcracks, its healing capacity is insufficient for long-term performance (Li et al., 2021). Accordingly, several conventional strategies have been developed to enhance asphalt's self-healing potential. Rejuvenation, for example, has proven effective when combined with vascular fibres (Anupam et al., 2022). Other approaches include the use of induction heating and microwave energy to accelerate crack closure (Norambuena-Contreras & Garcia, 2016). More recently, additives such as nanomaterials, shape-memory polymers, and ionomers have been investigated to further improve thermally activated self-healing efficiency (Chen et al., 2015; Ganjei & Aflaki, 2016; Ma et al., 2017).

2.3 Conductive fillers in self-sensing composites

Concrete composites can be applied under diverse conditions, including varying humidity and temperature regimes, as well as in environments characterised by heavy rainfall and marine exposure. Their durability, however, is strongly influenced by external loads and long-term aging. Reducing cement consumption is particularly significant, as it directly lowers carbon dioxide emissions (Moser et al., 2012). Substantial research efforts have therefore been devoted to improving the durability and serviceability of cementitious composites through alternative approaches. Self-sensing cementitious materials, first introduced by Chen and Chung (Chen et al., 1993; Song et al., 2024), represent one such advancement. These systems rely on conductive fillers to impart electrical conductivity to the cementitious matrix. Commonly employed fillers include CNTs, steel fibres, CF, and CB, all of which enhance the functional and mechanical performance of the composites (Chu et al., 2016; Chung, 2004; Han et al., 2014; Rana et al., 2016; Wen &

Chung, 2001; Xie et al., 1996). Compared with traditional mixtures, self-sensing cementitious composites generally exhibit superior mechanical properties, largely attributed to the incorporation of nanomaterials such as CNTs, CNFs, and CB (Bautista-Gutierrez et al., 2019; Han et al., 2014). Given the breadth of prior research, the present section will focus on three of the most extensively studied conductive fillers, CB, CF, and CNT, since other fillers have been comprehensively reviewed elsewhere.

2.3.1 Carbon fibre

Because of their relatively low cost and satisfactory conductivity, CFs have been widely employed to enhance the self-sensing performance of cementitious composites (Bontea et al., 2000; Chacko et al., 2007; Wen & Chung, 2006; Wen & Chung, 2007). Their effectiveness in self-sensing applications has been examined in numerous studies. For instance, Azhari and Banthia (2012) compared the sensing behaviour of cement-based composites incorporating 15% CFs with those containing a hybrid system of 1% multi-walled carbon nanotubes (MWCNTs) and 15% CFs. According to Fig. 2.2 (Azhari & Banthia, 2012), the results demonstrated that CFs exhibit a strong and reliable response to variations in compressive loading.

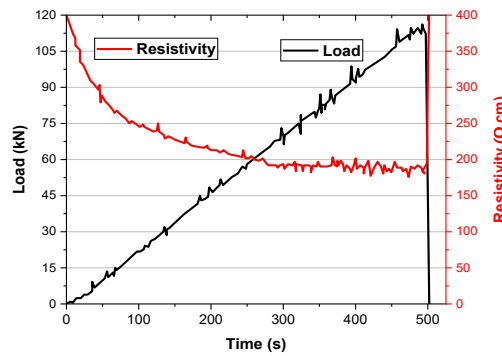
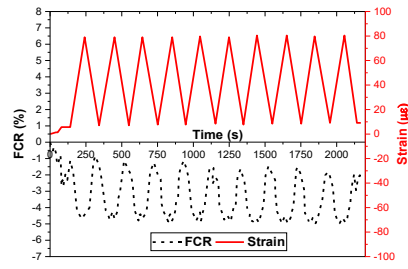
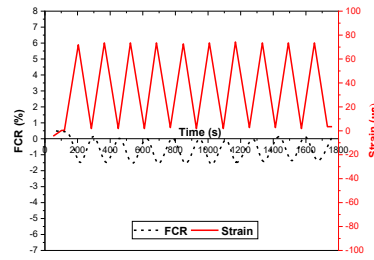


Fig. 2.2 Self-sensing performance of cementitious composites mixed with CF (Azhari & Banthia, 2012).



(a) With noise



(b) Without noise

Fig. 2.3 Relationship between Fractional change of resistance (FCR), strain, and time of the virgin and recycled carbon fibre composites (Belli et al., 2018).

However, because the experiment incorporated MWCNTs, it was difficult to isolate the specific contribution of CFs to the self-sensing behaviour. In a subsequent study, virgin and recycled CFs were incorporated into cement mortars to evaluate their piezoresistive performance. As shown in Fig. 2.3, the results revealed that data noise exerted a dominant and undesirable influence on the sensing performance of both fibre types (Belli et al., 2018). Feng & Ou (2018) investigated the self-sensing capacity of carbon fibre reinforced polymers (CFRPs). Their findings indicated that while CFRPs can enhance the structural sensing capability, crack formation may reduce the measurable energy response. Overall, these studies demonstrate that both noise interference and crack morphology substantially affect self-sensing performance. Consequently, future research should integrate healing mechanisms and employ noise-isolation environments to improve the reliability of experimental measurements.

Segura et al. (Segura et al., 2019) investigated ultra-high-performance concrete (UHPC) incorporating CFs as self-sensing sensors, demonstrating that CFs significantly enhance the piezoresistive response of cementitious composites. Their findings also indicated that CFs improve the percolation threshold of self-sensing systems; however, when the CF content fell below the threshold limit, piezoresistive performance was markedly reduced. A comparable study by (Wang et al., 2020) measured the piezoresistive behaviour of CFRPs using LG-FBG technology. The results showed that incorporating CFRPs not only improves load-bearing capacity but also strengthens structural compatibility. Collectively, these studies suggest that CFRPs can enhance both the mechanical properties and piezoresistive behaviour of cementitious self-sensing mixtures. More recently, Allam et al. (Allam et al., 2022) examined the influence of CF dosage on the percolation threshold of self-sensing concrete. Their results confirmed that optimal sensing performance occurs when CF content falls within the percolation threshold, while excessive sand content was shown to reduce piezoresistivity.

2.3.2 Carbon black

CB, widely studied as a conductive filler, has been applied in both cement- and asphalt-based self-sensing composites. At appropriate dosages, CB not only enhances the electrical conductivity of cementitious systems but also promotes cement hydration reactions (Dong et al., 2020). For example, cementitious composites containing 4 wt% CB exhibited favourable mechanical properties, while 7 wt% CB provided an optimal balance between piezoresistive sensitivity and mechanical strength (André O. Monteiro et al., 2017). In addition, the incorporation of silica fume (SF) has been shown to further increase electrical conductivity, with suitable SF contents also improving piezoresistivity (Dong et al., 2020). More recently, cementitious mixtures containing nano-carbon black

(NCB) and slaked lime demonstrated not only reliable piezoresistive performance but also self-healing capacity (Dong et al., 2021).

2.3.3 Carbon nanotube

The properties of conventional concrete can be significantly enhanced by incorporating CNTs (Tian et al., 2019). As one-dimensional nanomaterials, CNTs markedly improve the piezoresistive behaviour of cementitious composites Li et al. (2022), and have therefore been extensively investigated as conductive fillers. Smaller CNT agglomerations create smoother boundary surfaces, and studies have shown that while sandwiched agglomerations exert minimal influence on mechanical performance, they can substantially enhance piezoresistivity (Dong et al., 2020). To further improve both piezoresistivity and compressive strength, Dong et al. (Dong et al., 2021) developed composites incorporating waste glass cullet with CNTs to establish conductive pathways. Multi-walled carbon nanotubes (MWCNTs) have attracted considerable attention due to their high electrical conductivity. Cementitious composites containing MWCNTs demonstrate not only superior piezoresistive sensitivity but also improved mechanical properties (Danoglidis et al., 2016). Furthermore, MWCNTs can alter the percolation thresholds of cement-based composites, thereby facilitating the design of highly efficient electromagnetic shielding materials (Luo et al., 2018; Singh et al., 2013). Overall, the integration of CNTs, particularly MWCNTs, enables the development of self-sensing cementitious sensors that combine high sensitivity to external loading with enhanced mechanical performance.

2.4 Photocatalyst

The rapid growth of urbanisation has driven an increasing demand for vehicles; however, the resulting exhaust emissions have adversely affected environmental quality. Nitrogen oxides (NO_x), a major component of vehicular pollution, accumulate at high

concentrations near road surfaces, and their reduction remains a significant challenge (Atkinson, 2000). Various approaches have been proposed to mitigate vehicle-related emissions (Menz & pollution, 2002; Pollution, 2010; Rhimi et al., 2025). The concept of photocatalysis (PC) was first introduced by the Japanese chemist Fujishima through pioneering energy-conversion experiments (Dylla et al., 2013). Among available photocatalysts, titanium dioxide (TiO_2) has been the most extensively studied, particularly in applications related to self-cleaning materials and environmental remediation (Fujishima & Zhang, 2006). Owing to its favourable properties and commercial availability, TiO_2 has been identified as one of the most effective photocatalysts (Cassar, 2004; Diamanti et al., 2008; Fujishima & Zhang, 2006; Toma et al., 2009; Yu & Brouwers, 2009). Its self-cleaning capability has already been exploited in practical applications, such as coatings for glass windows and building facades (Fujishima & Zhang, 2006).

2.5 Temperature-responsive materials

During hot seasons, the surface temperature of asphalt pavements can reach 48–67 °C due to their dark colouration. Such elevated temperatures accelerate the deterioration of asphalt infrastructure, thereby reducing its durability. Consequently, there is an urgent need to develop efficient technologies capable of lowering pavement surface temperatures (Santamouris et al., 2007; Tombrou et al., 2008). Several cooling strategies have been investigated, including methods that enhance pavement thermal inertia. However, these approaches often result in excessive heat loss during colder months, largely due to the inherent thermal properties of dark pavements (Jiang et al., 2019; Villena Del Carpio et al., 2016). Thus, it is essential to identify technologies that can simultaneously reduce surface temperatures in summer while mitigating thermal energy loss in winter. Thermochromic materials (TMs) have emerged as promising candidates

for this purpose, owing to their ability to respond dynamically to temperature fluctuations. These materials exhibit thermochromism, a phenomenon in which their colour changes reversibly with temperature (Kim et al., 2018). Previous studies have demonstrated that metallic oxides such as NiO_3 , Fe_2O_3 , and TiO_2 can function as temperature-responsive components within infrastructure systems, providing potential pathways for smart pavement applications (Day, 1968; Kirk, 1978).

2.6 Application of four functions

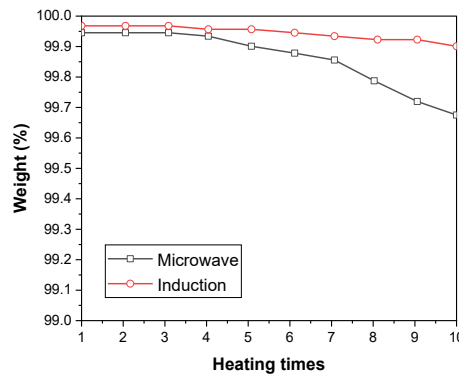
In modern transportation systems, pavements represent a fundamental infrastructure component. In recent years, the rapid increase in vehicle numbers has substantially raised traffic volume requirements. Consequently, pavements are now exposed to increasingly severe service conditions, driven by both heavier traffic loads and extreme weather events (Liang et al., 2015). At the same time, the growing demand for vehicles has intensified air pollution, with vehicular emissions exerting detrimental effects on environmental quality. As discussed in the preceding sections, the development of smart pavements offers a promising approach to address these challenges. Four key functionalities have been identified as central to this concept: self-healing (Xu et al., 2021), self-sensing (Borke Birgin et al., 2022), PC air-purification (Wang et al., 2016), and TC temperature-regulation (X. Zhang et al., 2022).

2.6.1 Application of self-healing function

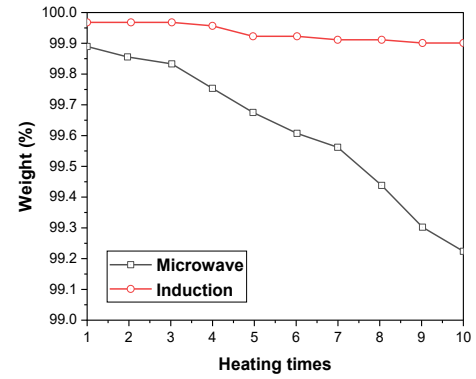
Microwave heating and induction heating are two widely investigated techniques for promoting heat-induced healing in asphalt composites (Anupam et al., 2022). Asphalt concrete admixtures have been employed to evaluate the performance of these methods (García et al., 2013). At elevated temperatures, the asphalt binder behaves as a Newtonian liquid while exhibiting viscoelastic properties (García-Morales et al., 2004; Lu & Isacsson, 1997). Experimental results indicated that heating to 100 °C achieved

approximately 60% crack recovery (Jiang et al., 2019; Santamouris et al., 2007; Tombrou et al., 2008), while subsequent studies reported healing rates of up to 80% at higher temperatures (García et al., 2013). Comparative studies have assessed the efficiency of induction and microwave heating in asphalt-based mixtures. In particular, mixtures containing fine and coarse aggregates combined with steel fibres demonstrated notable improvements in heating response (Norambuena-Contreras & Garcia, 2016). Using microwave heating, specimen temperatures reached 97 °C with 2% fibre content and increased to 135 °C with 8% fibre content, showing that fibres substantially enhance heat transfer and facilitate asphalt healing (Gallego et al., 2013). Overall, microwave heating has been shown to deliver superior performance compared with induction heating, as it selectively heats the asphalt binder rather than the aggregates (Norambuena-Contreras & Garcia, 2016). Moreover, as illustrated in Fig. 2.4, the incorporation of steel fibres markedly reduces particle loss during the heating process.

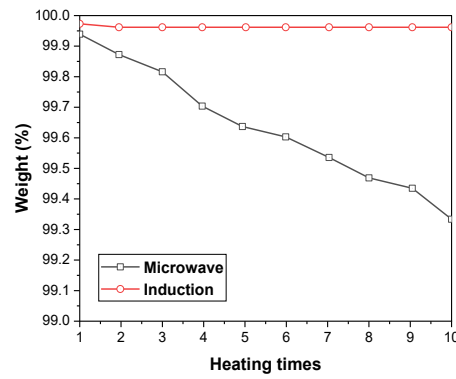
For effective healing, the temperature of asphalt mixtures must exceed 100 °C, as lower temperatures are insufficient to activate the heat-healing mechanism. However, under practical conditions, it is difficult to manually raise the pavement temperature to such levels, particularly in the absence of external confinement. Moreover, exposing asphalt pavements to elevated temperatures risks altering their geometry once the binder transitions into a Newtonian fluid state. Consequently, the direct application of heat-healing techniques to in-service pavements is impractical. Although previous studies have suggested that steel fibres can enhance heating efficiency, controlling the geometry of asphalt pavements under high-temperature conditions remains a critical challenge. Therefore, as outlined above, alternative strategies for achieving pavement healing under realistic field conditions must be developed.



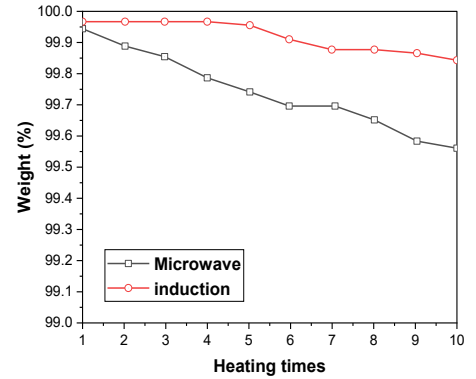
(a) 2% fibres



(b) 4% fibres



(c) 6% fibres



(d) 8% fibres

Fig. 2.4 Weight loss rate with different fibre contents (Norambuena-Contreras & Garcia, 2016).

The incorporation of healing agents represents a promising alternative for asphalt pavement self-healing. Agents with high concentrations of light components, such as oils, have been shown to promote the healing of asphalt mixtures (Anupam et al., 2022). Various additives, including vegetable oils, epoxy resins, and mineral oils, have been incorporated into asphalt binders to enhance their physical and mechanical properties (Al-Mansoori et al., 2017; Behnood, 2019; Liu, 2019; Yamaç et al., 2021). For instance, He et al. (He et al., 2021) investigated the use of aromatic oil and plant oil as healing agents in asphalt binders. Their results demonstrated that aromatic oil exhibited superior healing performance compared with plant oil, and that healing efficiency increased with rising temperature. Despite these encouraging findings, research on the long-term healing

performance of asphalt pavements remains limited. In particular, the maximum temperature considered in the study by He et al. (He et al., 2021) was 45 °C, whereas asphalt pavement surfaces can reach up to 67 °C during hot seasons (He et al., 2021; Santamouris et al., 2007; Tombrou et al., 2008). Future studies should therefore place greater emphasis on evaluating the influence of elevated service temperatures on the self-healing performance of asphalt pavements.

Porous asphalt concrete has been employed in previous studies to evaluate self-healing performance (Pamulapati et al., 2017). Steel fibres were shown to effectively facilitate specimens in reaching the target healing temperature, performing more efficiently than aluminium fibres. Using porous asphalt concrete, a recovery rate of up to 85% was achieved, significantly higher than that of specimens containing aluminium or steel fibres. Digital image analysis further confirmed superior healing efficiency in induced cracks (Pamulapati et al., 2017). Yoo et al. (Yoo et al., 2019) investigated the self-healing capacity of asphalt concrete modified with carbon-based materials, including CFs, CNTs, and GNFs. Their findings revealed that mixtures containing 0.5 wt% GNFs and CNTs achieved the highest levels of self-healing and flexural strength recovery. By contrast, CFs exhibited comparatively limited influence on the mechanical properties of the specimens.

Rejuvenators have also been applied to enhance the healing capacity of asphalt concrete. These agents restore and improve the intrinsic properties of asphalt, thereby yielding superior self-healing performance (Garcia et al., 2010; Zargar et al., 2012). Abadeen et al. (Abadeen et al., 2022) reported that the most effective healing period for rejuvenator-modified asphalt mixtures was approximately 120 hours.

2.6.2 Application of self-sensing function

To improve the functionality of cementitious pavements, several studies have incorporated self-sensing cementitious sensors into the pavement structure. Han et al. (Han et al., 2011) developed a self-sensing cementitious matrix, illustrated in Fig. 2.5, with the corresponding data acquisition system shown in Fig. 2.6. As presented in Fig. 2.7, the relationship between fractional change in resistance (FCR) and cyclic compressive stress revealed a temporal offset: the minimum FCR value occurred prior to the maximum compressive stress. This time lag suggests that the nickel-modified self-sensing cementitious composites did not provide fully accurate real-time stress monitoring. Moreover, the study did not account for the potential influence of environmental factors, such as weather conditions, on the piezoresistive response of the sensors.

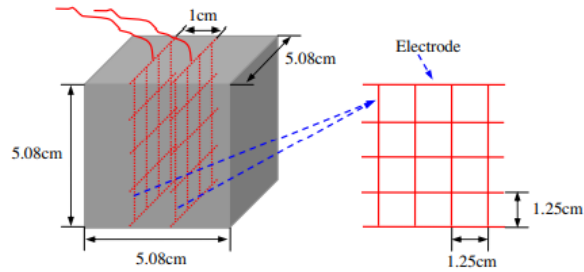


Fig. 2.5 Self-sensing cement-based sensors structure (Han et al., 2011).

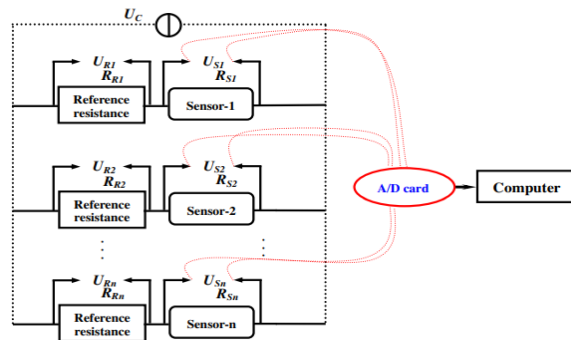


Fig. 2.6 Procedure of signal acquisition for the self-sensing pavement (Han et al., 2011).

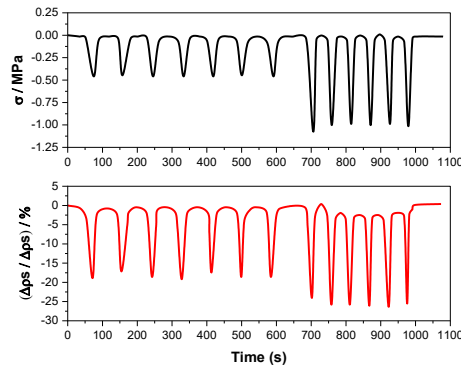


Fig. 2.7 Relationship between fractional change of resistivity and compressive force (Han et al., 2011).

Xin et al. (Xin et al., 2020) employed MWCNTs as conductive fillers to investigate the relationship between strain and piezoresistive behaviour using a two-electrode method. A high-sensitivity strain sensor was developed, and the experimental results demonstrated a clear correlation between changes in electrical resistance and strain. While the gauge factor of conventional sensors is typically around 2, the self-sensing sensors achieved a markedly higher value of 26.04. As illustrated in Fig. 2.8, the relationship between electrical resistance change and strain was approximately linear for strains below 1000 $\mu\epsilon$. Beyond this threshold, resistance decreased monotonically with further increases in strain. Composites combining MWCNTs with epoxy resin exhibited the highest sensitivity to strain fluctuations while maintaining compatibility with asphalt surfaces. These findings highlight the potential of MWCNT–epoxy composites for smart pavement systems; however, further validation studies are required to confirm their practical applicability.

In a separate study, (Kadhim et al., 2021) compared the piezoresistive responses of two types of fibre-reinforced concrete pavements (FRCPs). Their results showed that cracking led to an increase in resistivity. As shown in Fig. 2.9, the relationship between tensile

stress and resistivity demonstrated that FRCPs incorporating steel fibres exhibited piezoresistive behaviour, although the response was not consistently stable.

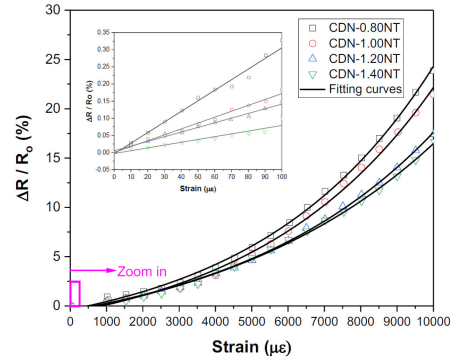
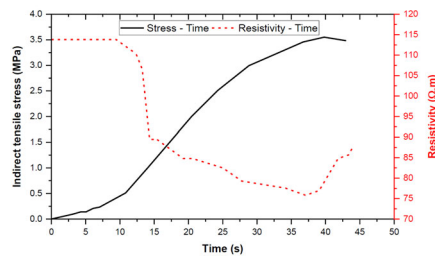
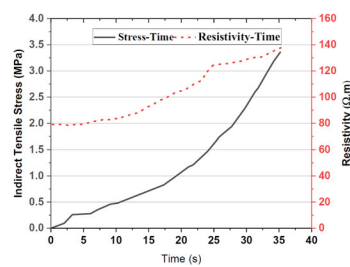


Fig. 2.8 Resistance response of composites in different CNT contents (Xin et al., 2020).



(a)



(b)

Fig. 2.9 Electrical resistivity of (a) steel and (b) carbon fibre-based composites (Kadhim et al., 2021).

Kadhim et al. (Kadhim et al., 2021) demonstrated that incorporating additional CFs not only improves the mechanical properties of fibre-reinforced concrete pavements (FRCPs) but also contributes to more stable piezoresistive behaviour. In another study, Gulisano et

al. (Gulisano et al., 2022a) investigated the use of electric arc furnace slag (EAFS) and graphene nanoplatelets (GNPs) to enhance the electrical conductivity of asphalt concrete. Their results revealed that mixtures containing 7% conductive fillers exhibited deterioration in piezoresistive performance, while mixtures with 3% and 5% filler contents performed even worse. As highlighted above, although numerous studies have explored the piezoresistivity of self-sensing composites with different conductive fillers, an optimal combination has yet to be identified. Further research is therefore required to systematically evaluate filler properties and establish strategies to improve the piezoresistive performance of both cement- and asphalt-based self-sensing sensors. To expand the application potential of these technologies in infrastructure, several innovative designs have been proposed. For example, Birgin et al. (Birgin et al., 2020) developed a novel weigh-in-motion (WIM) system, illustrated in Fig. 2.10, which demonstrates the practical integration of self-sensing materials into pavement monitoring systems.

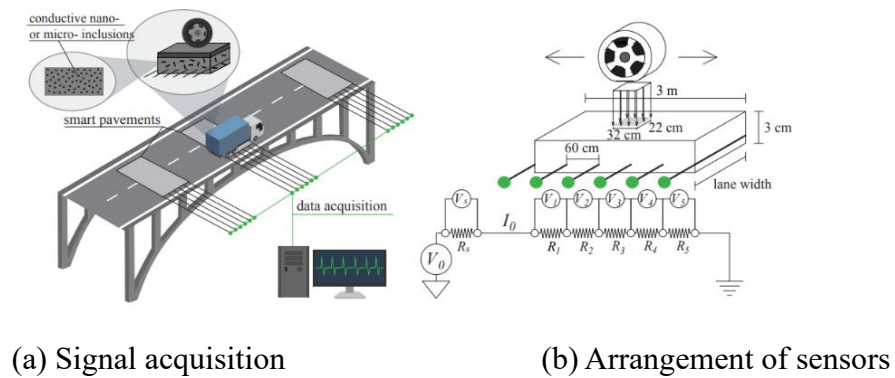


Fig. 2.10 WIM system in smart pavement (Birgin et al., 2020).

Although noise interference was present, the algorithm embedded in the WIM system demonstrated robust performance. The system was able to accurately evaluate vehicle weights as different types of vehicles passed over the self-sensing sensors. Nevertheless, further research is required to assess the long-term applicability of this WIM system for SHM in real-world conditions.

2.6.3 Application of thermochromic function

The skid resistance of asphalt pavements can be negatively influenced by heat-reflective coatings. To address this issue, approaches such as the incorporation of sand or other anti-skid materials have been proposed; however, previous studies have shown that sand may compromise the abrasion resistance of the coating (Cao et al., 2011; Nagare et al., 2025; Sha et al., 2017; Zheng et al., 2015). An alternative solution investigated by Jiang et al. (Jiang et al., 2019) was the use of a functional gradient multilayer structure (FGMS) coating. Their results demonstrated that FGMS coatings reduced the internal temperature of specimens by 12 °C and the surface temperature by 11 °C. Similarly, white coatings, owing to their light colour, achieved even greater reductions, lowering internal and surface temperatures by 14 °C and 13.5 °C, respectively. Yu et al. (Yu et al., 2020) employed TMs to evaluate temperature-responsive performance in asphalt pavements. Two asphalt types were examined in this study, SBS asphalt and #70 asphalt, through dynamic creep, rutting, and three-point bending tests, providing insights into the potential of TMs to improve both thermal and mechanical performance.

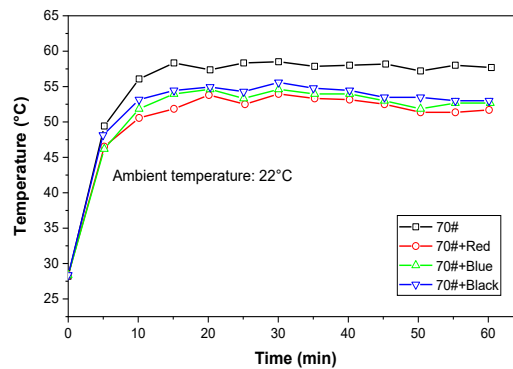


Fig. 2.11 Cooling performance of different thermochromic binders (Yu et al., 2020).

The incorporation of TMs into asphalt composites resulted in a measurable surface temperature reduction, with a maximum decrease of 5 °C, as illustrated in Fig. 2.11. In addition, TM-modified specimens exhibited a slower rate of heat loss, as shown in Fig. 2.12. Experimental results further revealed improvements in mechanical properties under

high-temperature resistance tests. Under cold-resistance testing, specimens with blue and black coatings demonstrated superior performance compared with the control group, whereas the red coating did not exhibit the same level of enhancement. However, asphalt composites with blue coatings were more prone to fatigue, while red coatings effectively mitigated fatigue effects (Yu et al., 2020). Alternative approaches, such as adding pigments to alter the colour of asphalt pavements, have also been investigated. Nonetheless, these methods have been associated with increased crack formation under low-temperature conditions (Anting et al., 2018; Kanerva et al., 1994; Pei-Wen et al., 2000; Synnefa et al., 2011).

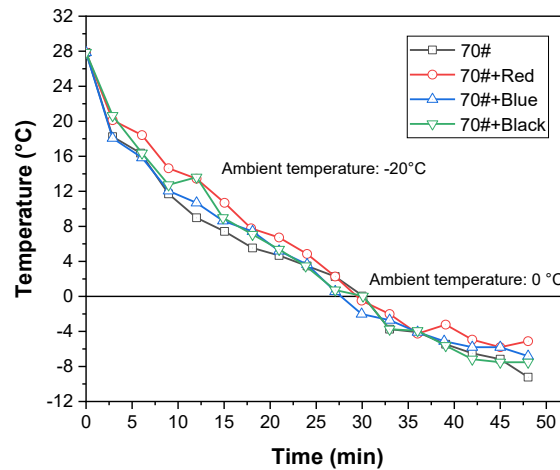


Fig. 2.12 Delayed cooling effect of different thermochromic binders (Yu et al., 2020). To further enhance temperature-control performance, TMs have been combined with nano-TiO₂ in varying proportions (Hu & Yu, 2020). The addition of nano-TiO₂ increased the heat reflectance rate of asphalt pavements by approximately 8–10%. However, rutting tests revealed that surface deformation reduced the solar reflectance rate. Outdoor solar heating experiments demonstrated that TM, nano-TiO₂, and reflective coatings could lower pavement surface temperatures by up to 15 °C during the daytime, while at night, the surface temperature increased by about 1 °C (Hu & Yu, 2020). Li et al. (Q. Li et al., 2020) reported that the most effective temperature regulation occurred at the top of the

middle asphalt layer and the bottom of the surface layer. Their findings further indicated that TMs could mitigate the urban heat island (UHI) effect. In addition, different TM colours exhibited variable temperature-control capacities, and prior studies have shown that their responsiveness can be further enhanced through the incorporation of nanomaterials. Future research should therefore explore the combination of TMs with alternative nanomaterials, such as MWCNTs and NCB, to optimise the thermal performance of asphalt composites.

2.6.4 Application of photocatalytic (PC) function

The first air-cleaning pavement was tested by Berthelot and colleagues at Louisiana State University (LSU) (Berthelot, 2011). For this experiment, a TiO_2 solution-distributing truck was designed to apply photocatalytic coatings onto the pavement surface (Osborn et al., 2012). As shown in Fig. 2.13, the results demonstrated that during the initial four days, the photocatalytic system achieved high levels of NO_x degradation. However, the performance declined noticeably after the fourth day of reaction, indicating a reduction in photocatalytic efficiency over time.

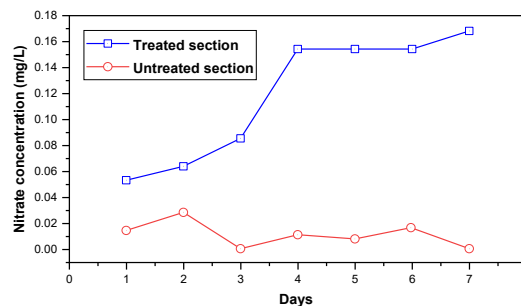


Fig. 2.13 Nitrate concentration changes after collection (Osborn et al., 2012).

Experimental results indicated that once nitrate accumulation reached high levels, the degradation efficiency of the PC coating became limited (Osborn et al., 2012). To further investigate this phenomenon, Dylla and colleagues (Dylla et al., 2013) integrated kinetic, field, and digital models to evaluate PC coating performance. Their kinetic analysis

revealed that NO mass transfer plays a critical role in governing the reaction rate of the photocatalytic process. According to equations (Dylla et al., 2013):



Evidence suggests that the rate of NO degradation may be influenced by environmental humidity. To examine this effect, Toro et al. (Toro et al., 2016) employed TiO₂ as a photocatalyst in both asphalt- and cement-based samples. Their results showed that NO degradation efficiency was relatively low in asphalt specimens and appeared independent of humidity. By contrast, cementitious samples exhibited higher NO removal efficiency under dry conditions, indicating that low humidity can enhance the performance of photocatalytic materials. However, the study also revealed that in cementitious specimens, the photocatalytic coating was less effective in reacting with CO.

To improve the NO absorption capacity of PC coatings, Fan et al. (Fan et al., 2018) investigated a novel carbon-modified TiO₂ coating for NO_x degradation. As shown in Fig. 2.14, the coating exhibited a significant reduction in NO_x concentration, demonstrating enhanced photocatalytic efficiency. Similarly, Chen et al. (Chen et al., 2021) employed N-doped TiO₂ in PC reactions and reported that the incorporation of silica sol yielded more stable nano-TiO₂ performance, further improving the durability and consistency of the photocatalytic response.

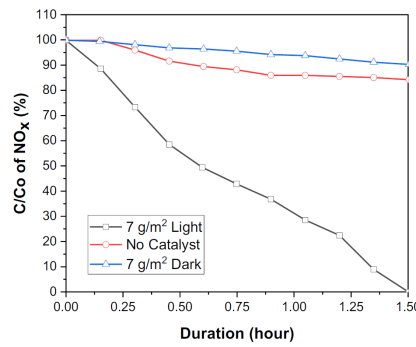


Fig. 2.14 Photocatalytic NO degradation validation (Fan et al., 2018).

N-doping has been shown to enhance the photocatalytic (PC) performance of TiO_2 (Chen et al., 2021). Nevertheless, further methodological advancements are needed to improve the absorption capacity of PC materials. Future research should place particular emphasis on the influence of environmental humidity, with the goal of identifying an optimal humidity range that maximises the performance of TiO_2 -based coatings.

2.6.5 ERT-based health monitoring

Airport pavements are designed to withstand extreme loading conditions; however, as aircraft weights continue to increase, the performance requirements of these pavements have also risen. Traditionally, pavement health monitoring has relied primarily on visual inspection (Cheng et al., 1999). Alternative approaches such as fibre Bragg grating (FBG) sensors, computer vision-based monitoring systems, and non-destructive evaluation techniques have been introduced (Kong & Li, 2018). Nevertheless, these methods are often limited by high costs and time inefficiency. To address these challenges, Gupta et al. (Gupta et al., 2021) proposed a more efficient approach that integrates electrical resistivity tomography (ERT) with self-sensing cementitious sensors. Their experimental results demonstrated not only the piezoresistive performance of the self-sensing composites but also the ability of ERT to successfully detect the geometry of subsurface cracks (Fig. 2.15). This finding highlights the potential of the method for identifying the location and shape of internal damage in pavement structures.

Despite these promising results, several shortcomings remain. The study did not account for long-term monitoring needs or the potential influence of weather on both the self-sensing cementitious specimens and the ERT detection system. Furthermore, coarse aggregates, due to their large particle size, were found to negatively affect piezoresistivity. Substituting coarse aggregates with fine aggregates or other additives may enhance the sensing capability without compromising the mechanical performance of the material. Overall, while further validation is required, the study confirmed that self-sensing concrete, in combination with ERT, holds significant potential for supporting the heavy loading demands of modern airport pavements.



Fig. 2.15 ERT results showing the shape of crack: (a) the shape of the crack, (b) ERT results of the cracking. (Gupta et al., 2021).

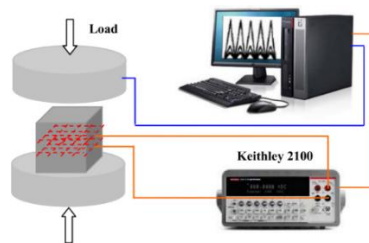


Fig. 2.16 Signal acquisition system of self-sensing concrete (Han et al., 2009).

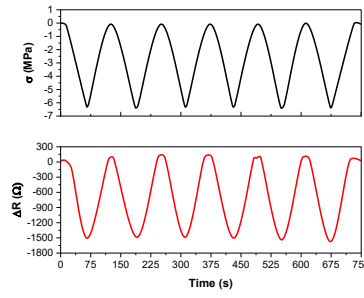


Fig. 2.17 Resistance changes synchronously as load change (Han et al., 2009).

2.6.6 Traffic and human motion detection

Various detection methods have been employed to facilitate the sharing of traffic-related information, such as vehicle counts (Faouzi & Klein, 2016). Conventional sensors, however, often face challenges including calibration difficulties, complex installation procedures, and high costs in both time and resources (Kell et al., 1990). Self-sensing cementitious sensors offer a promising alternative, as they can be readily integrated into infrastructure, are highly compatible with concrete-based structures, and have demonstrated strong application potential in prior studies (Han et al., 2009; Konstantoudos & Aza, 2014). MWCNTs were incorporated as conductive fillers in the self-sensing specimens, with the corresponding data acquisition system illustrated in Fig. 2.16. As illustrated in Fig. 2.17, these sensors exhibited stable responses under cyclic vehicular loading. Similarly, (Dong et al., 2022) evaluated vehicle speeds and pedestrian movements using self-sensing cementitious sensors through cyclic loading tests, further demonstrating that the approach can be extended to estimate wheel speeds. Collectively, these experimental results confirm the feasibility of employing self-sensing cementitious materials for traffic detection, highlighting their potential to significantly enhance traffic monitoring and management efficiency.

2.7 Summary

This chapter has reviewed four key functionalities of smart pavements: self-sensing, self-healing, photocatalysis (PC), and temperature responsiveness. While each of these functions has demonstrated significant potential, important limitations remain that must be addressed in future research.

The feasibility of self-sensing cement and asphalt-based sensors has been established in prior studies; however, most investigations have primarily focused on varying conductive fillers rather than on the detection performance associated with different vehicle types. To improve practical applicability, future research should consider cyclic compressive stresses representing a range of vehicle loads. Moreover, the stability of piezoresistive responses remains a concern. Internal factors such as water content, ionic conduction in the pore solution, and changes in pore structure, which influence the formation of new conductive pathways during loading, should be systematically examined in future studies.

The self-healing function has also shown promising results, yet its effectiveness is strongly influenced by environmental conditions. In particular, humidity plays a decisive role in healing performance, suggesting that wet–dry cyclic curing regimes should be incorporated into experimental designs. The role of free ions in the curing medium likewise warrants greater attention, as ionic interactions can significantly affect healing outcomes.

With respect to photocatalysis, prior research has confirmed the potential of PC coatings in pollutant degradation; however, their performance is limited by the low transfer rate of gaseous emissions and by environmental factors such as humidity. Future studies should therefore prioritise the development of modified or composite photocatalysts that can improve mass transfer and maintain efficiency under varying ambient conditions.

Finally, thermochromic and other temperature-responsive technologies have demonstrated their capacity to mitigate pavement overheating and reduce thermal fatigue. Yet, limited work has been undertaken to evaluate their long-term durability or their performance under diverse climatic conditions.

Overall, while the four functions have each shown considerable promise, very few studies have examined their integration within a single pavement system. Future investigations should focus on comprehensive evaluations of their compatibility and on pioneering research that explores the mechanisms underlying their cooperative and synergistic effects. Such efforts are essential to advance the development of multifunctional pavements capable of meeting the complex challenges of modern transportation infrastructure.

Chapter 3: Mechanical and piezoresistive properties of bitumen emulsion cementitious self-sensing sensors

This chapter presents a published study focusing on the piezoresistive and mechanical performance of self-sensing cementitious sensors incorporating bitumen emulsion. The study contributes to the overarching research objective by addressing key challenges associated with achieving reliable sensing functionality without compromising structural performance in pavement materials. By examining the formation and stability of conductive networks within composite systems, this work establishes essential knowledge for the broader integration of sensing capability within multifunctional pavement frameworks. The insights generated in this chapter provide a critical foundation for subsequent studies investigating additional functional mechanisms and their cooperative behaviour in smart infrastructure materials.

3.1 introduction

The combination of bitumen emulsion and cement produces an organic–inorganic composite. Such bitumen–cement mixtures are commonly employed in semi-flexible bases, cold recycling applications, and as cushion layers in ballastless tracks (Li et al., 2018; Zarei et al., 2020; Zhang et al., 2023). As cement hydration and bitumen agglomeration act as two distinct binding mechanisms, the composite benefits from the complementary properties of both materials. However, due to the prolonged hydration process, the mechanical behaviour of these mixtures undergoes significant changes over time. The compatibility of bitumen–cement mixtures plays a decisive role in determining their mechanical performance and workability (Tan et al., 2014). According to Ouyang & Meng (2022), the mixing stability of such composites is strongly influenced by viscosity, with high viscosity levels leading to reduced stability. The addition of water during

mixing lowers the viscosity of bitumen, thereby improving compatibility between bitumen and cement (Guo et al., 2022; Onuaguluchi & Banthia, 2016). Nonetheless, several studies have highlighted the retarding effects of bitumen emulsion on cement hydration. Wang et al. (Wang et al., 2015) demonstrated that bitumen emulsion delays the hydration process, while Tan et al. (Tan et al., 2013) showed that emulsifier dosage influences hydration kinetics, heat evolution, and setting behaviour through similar retardation effects. Qiang et al. (Qiang et al., 2011) further confirmed that the incorporation of bitumen emulsion can extend the dormancy period of cement hydration. Although the retardation effect of bitumen emulsion can influence cement hydration, the absorption rate between bitumen and cement can be improved through their interaction (Sun et al., 2019). Compared with conventional cement composites containing mineral fillers, bitumen–cement composites exhibit stronger stiffness interactions between hydrated cement and bitumen (Niazi & Jalili, 2009), thereby enhancing the mechanical characteristics of self-sensing systems. Hydration can also be promoted by the release of water during emulsification (Wang et al., 2013). In addition, cement can form stable electric double layers, which destabilise bitumen and subsequently reduce the electrical resistance of the composite (Lu et al., 2009; Tan et al., 2013). The incorporation of conductive fillers not only influences the electrical response of the material but is also closely related to its mechanical performance through the formation of internal conductive networks. Among various conductive materials, MWCNTs are widely adopted due to their high aspect ratio and ability to establish stable conductive pathways at relatively low dosages.

A range of conductive fillers has been explored for self-sensing applications, confirming the high potential of such systems in smart pavements (Deng et al., 2023). However, relatively few studies have specifically examined the self-sensing performance of

bitumen–cement composites. The incorporation of bitumen emulsion has the capacity to alter both the mechanical and piezoresistive properties of these composites. Accordingly, the objective of this study is to compare the mechanical behaviour and self-sensing performance of bitumen–cement-based sensors with those of conventional self-sensing sensors. Furthermore, this study considers the influence of water content on piezoresistive performance to provide a more comprehensive understanding of the behaviour of these composites.

3.2 Experimental program

3.2.1 Raw materials

The bitumen emulsion used in this study was supplied by Boral Asphalt, with its detailed properties provided by the manufacturer and summarised in Table 3.1. Multi-walled carbon nanotubes (MWCNTs) were selected as conductive fillers, while general-purpose cement conforming to AS 3972 (Standard, 2010), was employed as the primary binder. Specimen preparation began with the mixing of dry cement and fine aggregates in a Hobart mixer. Water containing dispersed MWCNTs was then added to the mixture, enabling the partial formation of internal conductive networks to enhance electrical conductivity. Multiple specimen groups with varying conductive filler contents were prepared for piezoresistivity investigations. In addition, different water–cement ratios (W/C) and bitumen–cement ratios (B/C) were adopted to evaluate compressive strength. The detailed mixture proportions of all specimen groups are presented in Table 3.2. Samples were cast in cubic moulds of $50 \times 50 \times 50 \text{ mm}^3$ and demoulded after 24 hours of curing. Cement served as the primary structural matrix, ensuring compatibility between the self-sensing composites and conventional concrete structures. Previous research has indicated that during the early hydration stage, hydration products can be encapsulated by a membrane formed by bitumen, thereby limiting hydration. However, with continued

curing, these products can penetrate the bitumen membrane, allowing hydration to proceed (Qiang et al., 2011). The MWCNTs used in this study have an average length of 3–12 μm , a diameter of 8–15 nm, and an aspect ratio of approximately $\geq 250 \frac{\text{m}^2}{\text{g}}$, with dosages ranging 0.05, 0.07, and 0.5 wt.% of cement. To investigate the mechanical behaviour of the self-sensing specimens incorporating bitumen emulsion, compressive strength tests were conducted at 7, 14, 28, 42, and 60 days. Thermogravimetric analysis (TGA), compression testing, and nuclear magnetic resonance (NMR) analysis were employed to characterise the materials.

Table 3.1 Properties of bitumen emulsion

	Bitumen	Water	Emulsifier	Boiling point	pH	Expected curing temp	Max temp (in use)
Bitumen emulsion	60%	40%	< 2%	100 °C	>10	20 °C	70 °C

Table 3.2 The specimen groups with different water-cement and bitumen cement ratios.

Group number	W/C ratio	B/C ratio
Group A	0.30	0.25
Group B	0.30	0.30
Group C	0.30	0.35
Group D	0.35	0.25
Group E	0.35	0.30
Group F	0.35	0.35
Group G	0.40	0.25

Group H	0.40	0.30
Group I	0.40	0.35

3.2.2 Preparation of specimens

For each specimen, 92 g of general-purpose cement and 137.5 g of fine aggregates were used. Initially, the dry cement and fine aggregates were manually blended in a pan mixer. The dry mixture was then subjected to oven-drying at 80 °C for 72 hours. The drying process was conducted to remove residual moisture from the materials and ensure consistent initial conditions, which is critical for reliable piezoresistive measurements. During this drying period, the required amount of MWCNTs was dispersed into the mixing water using an ultrasonicator. Because MWCNTs have a strong tendency to agglomerate due to van der Waals forces (Raki et al., 2010), ultrasonication was applied for 40 minutes for each batch to ensure adequate dispersion. The MWCNTs were first dispersed in water using Triton X-100 as a surfactant, at a MWCNT-to-surfactant mass ratio of 1:2. Ultrasonic dispersion was conducted in an ultrasonic bath (40 kHz) for 40 min to achieve a homogeneous and stable suspension. After drying, the components were mixed in a Hobart mixer. The raw materials were added in the following sequence: fine aggregates first, followed by mixing water containing dispersed MWCNTs and the bitumen emulsion, and finally cement. Mixing was continued for 1 minute to obtain a homogeneous mixture. The fresh mortar was then poured into cubic moulds ($50 \times 50 \times 50 \text{ mm}^3$). Four electrodes were inserted into each specimen before curing. The moulded samples were placed in a curing tank maintained at $20 \pm 1 \text{ °C}$ and 90% relative humidity for 24 hours before demoulding (Fang et al., 2015). Mechanical and electrical characterisation was subsequently carried out. Compressive strength was tested using UH 500 kN and AGX 50 kN machines, while electrical resistivity was measured via the embedded electrodes. Specimens were labelled as BCX and CCX, where BC and CC

denote mixtures prepared with and without bitumen emulsion, respectively, and X represents the proportion of MWCNTs incorporated. An overview of the experimental procedure is provided in Fig. 3.1.

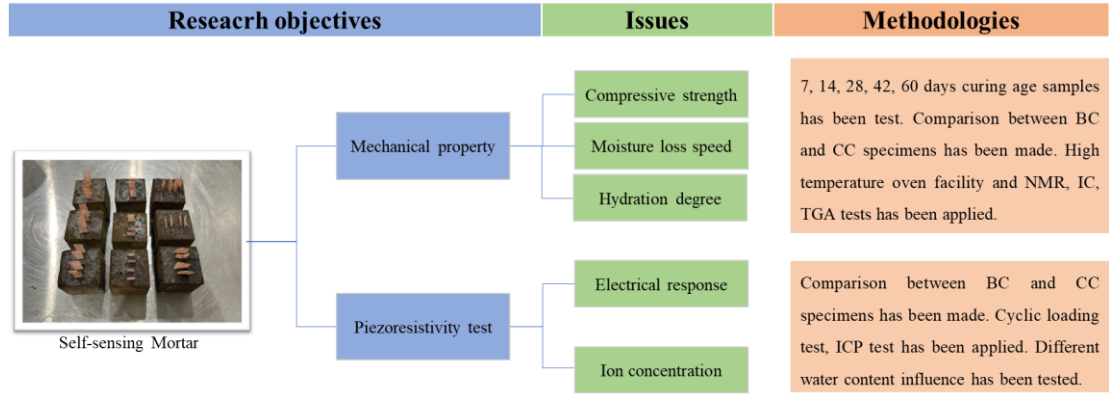


Fig. 3.1 Experiments flow chart.

3.2.3 Piezoresistivity test

The four-electrode method was employed to evaluate the piezoresistive performance of the composites, with copper mesh used as the electrodes. Cement-based self-sensing specimens without bitumen emulsion served as the control group. Two digital multimeters were utilised to record the electrical responses and assess the piezoresistive behaviour of the specimens. The following Eq. (1) illustrates the calculation method of resistivity change (Han et al., 2011):

$$\rho = \frac{V_1}{V_2} \times \rho_0 \quad (1)$$

where ρ is the resistivity of the specimen; V_1 is the voltage across the specimen; V_2 is the voltage across the additive resistor, and ρ_0 is the electrical resistance of the additive, which was 10,000 Ω . Fig. 3.2 illustrates the experimental circuit used for the piezoresistivity tests. A Solartron SI 1260 frequency response analyser was employed to conduct alternating current impedance spectroscopy (ACIS) in order to determine the electrical resistance of each specimen. An input frequency range of 1–10 MHz was

applied to record the impedance data, and measurements were obtained using the two-probe connection method (W. Li et al., 2024).

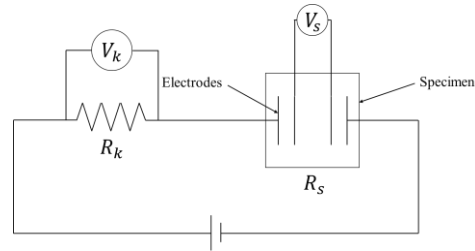


Fig. 3.2 Circuit of piezoresistivity test for four nodes specimens.

3.2.4 Compressive strength

The surface area of each specimen tested in compression was 50×50 mm. Bitumen–cement (BC) specimens were subjected to compression testing to evaluate the influence of bitumen emulsion on the compressive strength of cementitious cubes. A UH 500 kN testing machine was employed for the analysis. During testing, each specimen was carefully aligned with the centre of the thrust platen. The load was applied smoothly and without shock, at a constant rate corresponding to 20 ± 2 MPa of compressive stress per minute, until failure occurred. The maximum load sustained by each specimen was recorded automatically by the testing machine.

3.2.5 Microstructural analysis

Nuclear magnetic resonance (NMR) spectroscopy is a powerful analytical technique for characterising the orientation of moieties in both liquid and solid states, as well as for determining chemical structure. In this study, NMR was employed to investigate the hydration products of BC and CC specimens, as ^{29}Si NMR is particularly sensitive to the slow crystallisation of silicate phases (Rottstegge et al., 2006). Fine powders ground from the specimens were placed in 4 mm MRK rotors, and all tests were conducted at a rotational frequency of 11 kHz (Lu et al., 2022).

To further analyse the microstructural features of fractured mortar specimens obtained from the compression tests, scanning electron microscopy (SEM) was performed. Prior to imaging, specimen surfaces were coated with gold to improve electrical conductivity. The coated samples were then mounted on SEM holders using carbon tape to ensure secure attachment. Energy-dispersive X-ray spectroscopy (EDS) was subsequently carried out, including both mapping and point analyses, to determine the elemental composition of the specimens.

The TGA test was applied to analyse the hydration products mass loss in the temperature range from 25 °C to 1000 °C. The derivative curve (DTC) from the TGA mass loss curve can be applied to identify which materials' mass loss occurred in each temperature range.

The value was scaled based on the following Eq. (2) (Shi et al., 2017):

$$m_{CH} = \frac{M_{CH}}{M_H} \times m = \frac{74.09}{18.02} \times m \quad (2)$$

where m is the mass loss of water from portlandite; the M_H is the molar mass (18.02 g/mol) of H_2O ; the M_{CH} is the molar mass (74.09 g/mol); the m_{CH} is the portlandite mass fraction (wt%).

The quantification of calcium hydroxide (CH) was carried out on steam-cured cement paste in this study. CH content is commonly used as an indicator of the degree of hydration, as its formation is directly correlated with the fraction of hydrated paste (Wang et al., 2005). For this purpose, the mass of CH was recorded at a given time t and compared with the CH mass in fully hydrated reference samples. These parameters can be obtained through the following Eq. (3) (Liu et al., 2021):

$$\alpha_t = \frac{M(CH)_t}{M(CH)_\infty} \quad (3)$$

According to a previous experiment, $M(CH)_t$ is the mass of CH at time t which is determined through TGA. The $M(CH)_\infty$ is the CH mass of the totally hydrated cement

paste. The α_t is the hydration degree. If the water-cement ratio was between 0.2 to 0.5, after 90 days of curing, the pure cement paste's CH content can be regarded as the fully hydrated amount, rather than waiting for 400 days of curing (Liu et al., 2021).

Isothermal calorimetry (IC) was performed to investigate the early-age hydration heat of the pastes. Following ASTM C1702-17, the 72-hour hydration heat flow curves were measured using an eight-channel Calorimeter (CALMETRIX). To minimise external interference, the heat flow recorded during the first 30 minutes was excluded from the analysis.

Fourier transform infrared spectroscopy (FTIR) was also employed to characterise hydration products. A Jasco 4100 series spectrometer was used, with a scanning wavelength range of 400–4000 cm^{-1} .

3.2.6 Water evaporation and absorption test

Saturated specimens were placed in an oven at 105 °C, and water loss was recorded at 30-minute intervals during the first half hour and subsequently at regular intervals over the following hours. When cumulative water loss reached 1%, 5%, 10%, 15%, 20%, 30%, 40%, and 50% of the specimen mass, electrical response tests were performed at each stage until the specimen mass stabilised. Heating was terminated once the specimens reached an oven-dry condition.

To evaluate the void structure and pore ratio of bitumen–cement-based self-sensing specimens, both microscopic analysis and the water absorption method were employed. The latter followed the procedures outlined in AS 2983-18, Determination of Water Absorption. During testing, the liquid level was maintained above the specimen surface, and specimens were immersed until a constant mass was reached. They were then oven-dried at 105 °C until another constant mass was achieved, which was recorded as M_1 . The specimens were subsequently re-immersed in water for 72 hours, after which surface

moisture was removed with a cotton cloth and the saturated mass was recorded as M_2 .

The following Eq. (4) was utilised to calculate the water absorption rate, R :

$$R = \frac{M_2 - M_1}{M_1} \times 100 \quad (4)$$

3.2.7 ICP test

Inductively coupled plasma (ICP) spectroscopy was employed to measure the ion concentration of the pore solution. The solvent extraction method was adopted following the procedure outlined by Zhan et al. (2023b). Finely ground cement paste powder was mixed with deionised water at a powder-to-water ratio of 1:5 to minimise dilution effects. The mixture was then centrifuged at 5000 rpm for 10 minutes to separate the supernatant from the solid residue. Prior to ICP analysis, the supernatant was diluted with 2.0% HNO_3 to ensure stability and accuracy of the measurements. The prepared solution was subsequently analysed using the ICP facility.

3.2.8 ACIS test

Alternating current impedance spectroscopy (ACIS) was conducted to analyse the electrical properties of the specimens. A Solartron SI 1260 frequency response analyser was employed, with ZPlot and ZView software used for data acquisition and analysis. The two-probe measurement method was adopted, following the test protocols and reliability verification reported by R. Wang et al. (R. Wang et al., 2022). Cables were directly connected to the self-sensing specimens, and measurements were carried out across a frequency range of 1 Hz to 10 MHz.

3.3 Mechanical properties

3.3.1 Compressive strength

Different combinations of water–cement (W/C) and bitumen emulsion–cement (B/C) ratios were employed to investigate the compressive strength of high-performance

mortars. The comparative results are presented in Fig. 3.3. Among the tested mixtures, the highest compressive strength was achieved at a W/C ratio of 0.35 and a B/C ratio of 0.25.

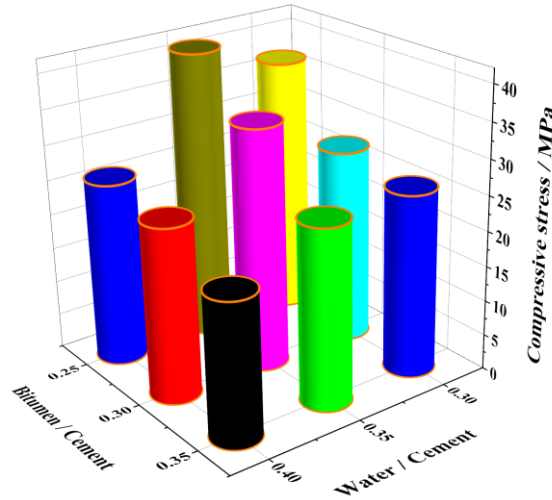


Fig. 3.3 Comparison of the compressive strength of different specimens with different W/C ratios and B/C ratios.

A W/C ratio of 0.35 and a B/C ratio of 0.25 were selected for this study to satisfy the compressive strength requirements and to control variables in subsequent experiments. Similar trends have been reported in previous research (Qiang et al., 2011).

Fig. 3.4 presents the compressive strength development of BC and CC specimens over different curing periods. For CC specimens, compressive strength reached 44.7 MPa at 28 days, after which the rate of strength gain slowed. By 60 days, the maximum compressive strength achieved was 48.76 MPa. In contrast, BC self-sensing mortars followed a different trend. At 7 days, BC specimens achieved 20.02 MPa, which increased to 30.6 MPa after 14 days. By 28 days, compressive strength plateaued, reaching 33.84 MPa with only marginal improvement thereafter.

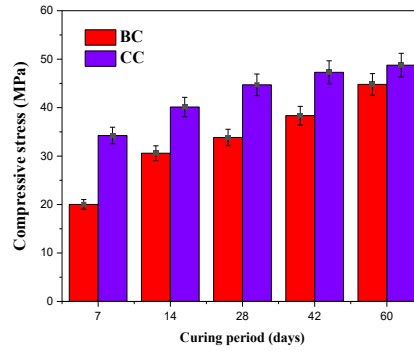


Fig. 3.4 Compressive strength development of specimens in five curing stages with W/C and B/C of 0.35 and 0.25.

Fig. 3.5 illustrates the rate of compressive strength development for BC and CC specimens. A faster early-age strength development was observed in BC specimens compared with CC specimens, which can be attributed to the influence of bitumen emulsion; however, this also resulted in a lower ultimate compressive strength. With extended curing, the compressive strength of BC specimens increased markedly, eventually reaching 44.79 MPa at 60 days, as shown in Fig. 3.4. After 60 days, the difference in compressive strength between cement-only and bitumen–cement self-sensing mortars became less pronounced. Fig. 3.5 further highlights the contrasting development rates of compressive strength in BC and CC specimens over time.

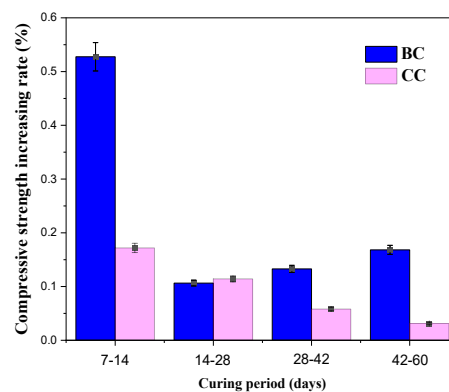


Fig. 3.5 Compressive strength increasing rate of specimens in four periods.

Analysis of the compressive strength development indicates that hydration played a critical role in strength gain during the later curing stages, particularly between 28–42 days and 42–60 days. The experimental results suggest that an internal curing mechanism

contributed to enhanced hydration in the composites. After 28 days, water released from the bitumen emulsion appeared to accelerate the hydration rate, thereby supporting further strength development. These findings are consistent with the work of (Yang et al., 2022), which confirmed that long-term internal curing can effectively promote extended hydration in cementitious composites.

Consequently, the evolution of compressive strength in BC specimens exhibited significant differences compared with CC specimens. The compressive strength of CC specimens reached 44.7 MPa at 28 days, whereas BC specimens attained only 33.84 MPa at the same age. However, the compressive strength of BC specimens continued to increase substantially with extended curing, reaching 44.79 MPa at 60 days. Specifically, BC specimens exhibited strength gains of 13.28% between 28 and 42 days and 16.82% between 42 and 60 days, representing an overall increase of approximately 30% from 28 to 60 days. This secondary hydration stage (28–60 days) enabled BC mortars to demonstrate superior long-term performance compared with their early-age behaviour (0–28 days). By contrast, CC mortars achieved only an 8.9% increase in compressive strength between 28 and 60 days. These findings confirm that the hydration process in BC specimens was enhanced by internal curing, facilitated by water released from the bitumen emulsion.

3.3.2 Hydration progress of bitumen-cementitious matrix

Hydration heat analysis revealed significant differences between BC and CC specimens. The presence of bitumen emulsion was found to limit the rate of early hydration. As shown in Fig. 3.6, CC specimens released more heat than BC specimens within the first five hours and reached their peak heat release at approximately 10 hours. In contrast, BC specimens exhibited a delayed peak at around 12 hours, indicating that bitumen emulsion impeded early hydration. Notably, BC specimens displayed a second peak at

approximately 20 hours, a phenomenon absent in CC specimens. This behaviour can be attributed to the membrane formed by the bitumen emulsion around cement particles. Initially, the membrane hindered hydration, prolonging the dormant period. As hydration products gradually disrupted this barrier, the reaction resumed without major limitations, resulting in the secondary peak. Similar findings were reported by Wang et al. (Wang et al., 2015), who observed a second peak in specimens containing bitumen emulsion after 20 hours, with a maximum heat release of 2.75 mW/g. These results support the conclusion that the emulsifiers and bitumen in the mixture prolong the dormant period, and the eventual breakdown of the emulsion film gives rise to the two distinct heat release peaks.

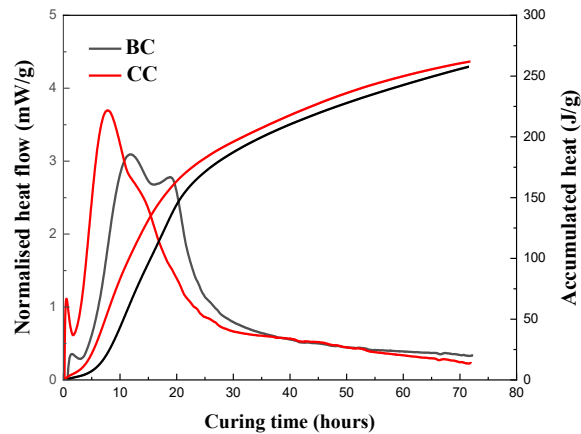


Fig. 3.6 Hydration heat of BC and CC specimens during the first 3 days.

It was also necessary to examine whether bitumen emulsion influenced the hydration process beyond the initial 72-hour period. Thermogravimetric analysis (TGA) was conducted to assess mass loss, with the corresponding endothermic peaks observed in the derivative thermogravimetric (DTG) curves. The experimental results are presented in Fig. 3.7 and Fig. 3.8. The first peak, occurring between 50 and 200 °C, corresponded to mass loss due to the evaporation of free and physically bound water. The second peak, observed between 380 and 500 °C, was attributed to the dihydroxylation of portlandite (Ca(OH)_2). The third peak, occurring between 600 and 800 °C, was associated with the

decarbonation of calcium carbonate (CaCO_3) (Hong et al., 2022; Mahmood et al., 2022; Vogler et al., 2021). Table 3.3 displays the hydration degree of CC and BC specimens.

Table 3.3 The degree of hydration of BC and CC specimens from 7 to 60 days of curing.

Curing period Samples	Degree of hydration (DoH) of samples				
	7 days	14 days	28 days	42 days	60 days
BC specimens	78.25%	85.89%	90.19%	90.64%	91.80%
CC specimens	78.94%	86.10%	91.31%	91.77%	91.89%

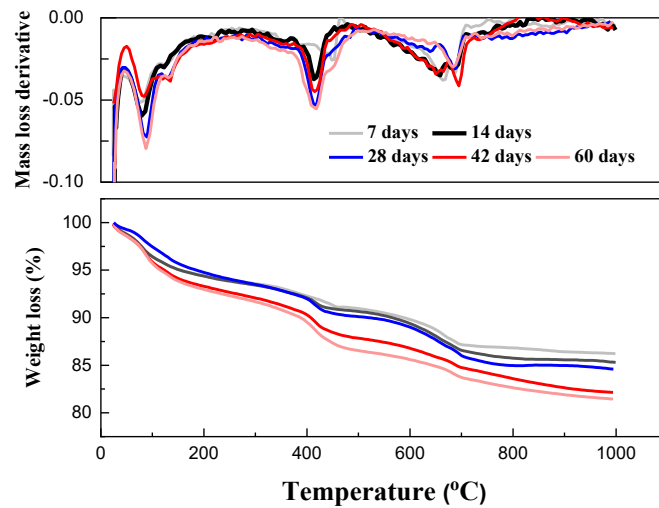


Fig. 3.7 TGA result of BC specimens.

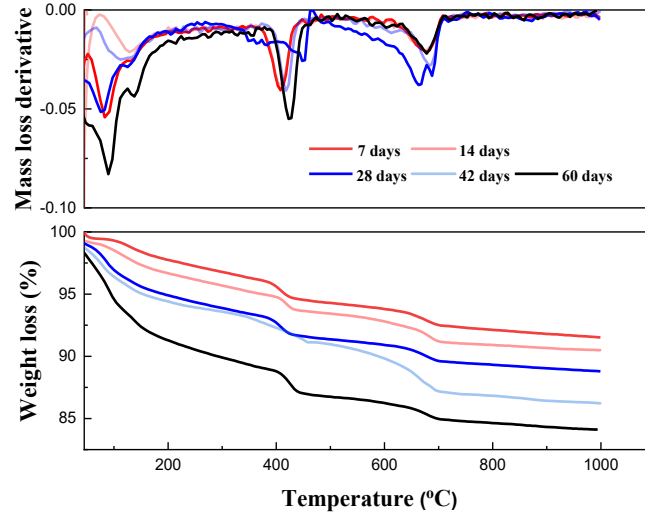


Fig. 3.8 TGA result of CC specimens.

The degree of hydration (DoH) was further evaluated using nuclear magnetic resonance (NMR) spectroscopy. Both ^1H and ^{29}Si NMR solid-state NMR analyses were performed to investigate the influence of bitumen emulsion. All measurements were conducted with 4 mm rotors at a spinning frequency of up to 11 kHz. The ^{29}Si spectra were recorded over a chemical shift range of -60 to -120 ppm. In this framework, Q_0 represents uncondensed silicate units, Q_1 corresponds to chain-end disilicate groups, Q_2 denotes short-chain disilicate middle groups, and Q_3 and Q_4 refer to silicate tetrahedra in layered structures and three-dimensional networks (Jamsheer et al., 2018). In order to analyse the hydration process and polymerisation degree of the cement, the Eqs. (5-7) were used (Chen et al., 2022):

$$\text{Degree of hydration of silicate phase: DoH} = 100\% - Q_0 \quad (5)$$

$$\text{Polymerisation degree: Pd} = Q_1 + \frac{2 \times Q_2}{Q_2 + Q_1} \quad (6)$$

$$\text{The length of the silicate chain: MCL} = 2 \times \frac{Q_1 + Q_2}{Q_1} \quad (7)$$

Q_0 , Q_1 , and Q_2 peaks were detected at 70, 80, and 85 ppm, respectively, with Q_0 associated with unreacted cement clinker (Wang et al., 2021). Table 3.4 and Table 3.5 present the hydration degrees of BC and CC specimens based on the NMR results. A

gradual decrease in the Q_0 peak was observed with extended curing, accompanied by an increase in the Q_1 and Q_2 peaks. The reduction of the Q_0 signal corresponded to the progressive consumption of unreacted cement clinker. When compared with the standard ^{29}Si MAS NMR results reported in Fig. 3.9 (Chen et al., 2022), the Q_0 peak volume continued to decline over time, while the Q_1 and Q_2 volumes consistently increased, reaching their highest levels at later curing ages. Notably, the relative changes in Q_0 , Q_1 , and Q_2 between 28 and 60 days were more substantial in the BC self-sensing specimens than in the CC specimens (Fig. 3.9 and Fig. 3.10). In comparison with the standard hydration degree development curve reported by Termkhajornkit et al. (2015) (Fig. 3.11), BC specimens demonstrated a distinct trend after 28 days of curing, ultimately achieving a higher degree of hydration than CC specimens.

Table 3.4 The BC statistical deconvolution results of ^{29}Si NMR spectra.

Curing periods	Q_0	Q_1	Q_2	Q_3	Q_4	DoH
7 days	29.50%	28.09%	19.43%	18.99%	3.98%	70.50%
14 days	17.45%	48.18%	19.42%	11.59%	3.37%	82.55%
28 days	14.86%	43.25%	16.22%	24.33%	1.35%	85.14%
42 days	14.50%	50.43%	18.91%	11.98%	4.17%	85.50%
60 days	11.73%	50.88%	14.90%	11.44%	11.03%	88.27%

Table 3.5 The CC statistical deconvolution results of ^{29}Si NMR spectra.

Curing periods	Q_0	Q_1	Q_2	Q_3	Q_4	DoH
7 days	29.05%	48.03%	9.79%	11.66%	1.47%	70.95%

14 days	17.34%	43.90%	18.21%	18.69%	1.87%	82.66%
28 days	15.71%	54.29%	17.14%	10.00%	2.86%	84.29%
42 days	13.36%	62.86%	14.14%	7.86%	1.79%	86.64%
60 days	11.59%	46.76%	30.96%	7.07%	3.62%	88.41%

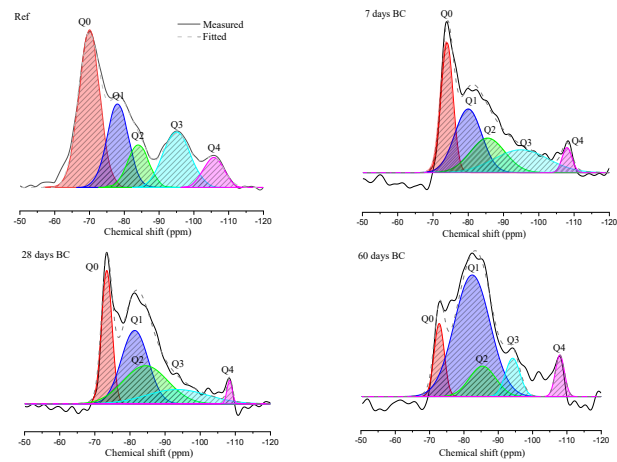


Fig. 3.9 Standard NMR results and NMR results of BC specimens under different curing periods (Chen et al., 2022)

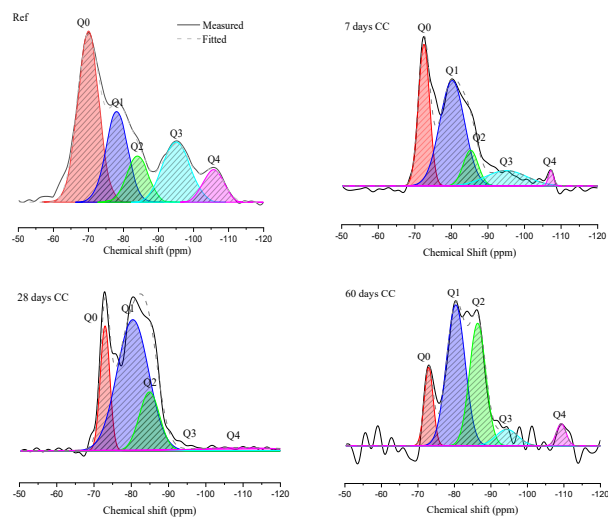


Fig. 3.10 Standard NMR results and NMR results of CC specimens under different curing periods (Chen et al., 2022)

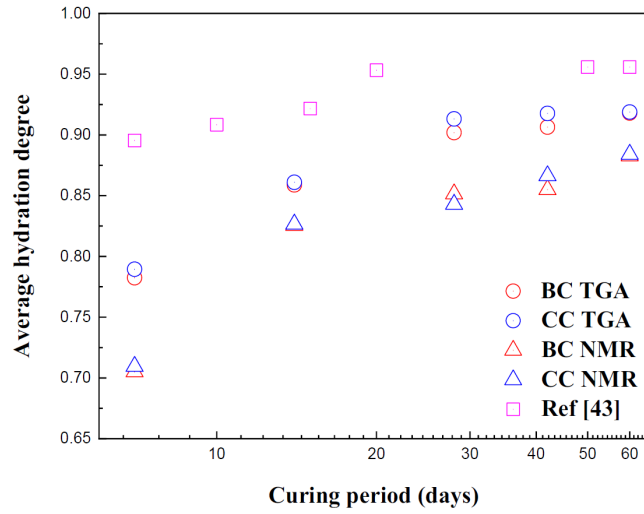


Fig. 3.11 Hydration development results of BC and CC specimens with NMR and TGA test methods.

The incorporation of bitumen emulsion reduced the early-age compressive strength of the specimens, primarily due to the formation of voids and microcracks within the mortar matrix, as well as a lower degree of hydration. However, as curing progressed, BC specimens benefited from an internal curing effect that promoted continued cement hydration, as illustrated in Fig. 3.4, Fig. 3.5, and Fig. 3.11. This internal curing mechanism sustained hydration during the later stages of curing (Yang et al., 2022), leading to a marked increase in compressive strength between 28 and 60 days. The substantial strength gain observed during this period provides clear evidence of the effectiveness of the internal curing process.

3.3.3 Effect of moisture loss on CC and BC specimens

The experimental results showed that bitumen–cement mortar exhibited slightly higher overall water loss compared with cement-based mortar, with values of 16.03% and 15.18%, respectively. Water content was calculated as the ratio of water volume to cubic specimen volume, and the rate of evaporation was monitored, as shown in Fig. 3.12. Cement-based specimens experienced rapid water loss at 105 °C, with a sharp increase

during the first two hours, reaching 27.3%. Beyond this point, the rate stabilised, culminating in a total water loss of 46.5% after 5.5 hours of heating. In contrast, bitumen–cement self-sensing specimens exhibited a slower water loss profile. During the first 1.5 hours, their water loss (18.6%) was comparable to that of the cement-based specimens. However, over the following two hours, the rate decreased significantly relative to the cement-based group. After 5.5 hours of heating, the cumulative water loss of the bitumen–cement specimens reached 35.6%. The progressive differences in water loss between BC and CC specimens are illustrated in Fig. 3.12.

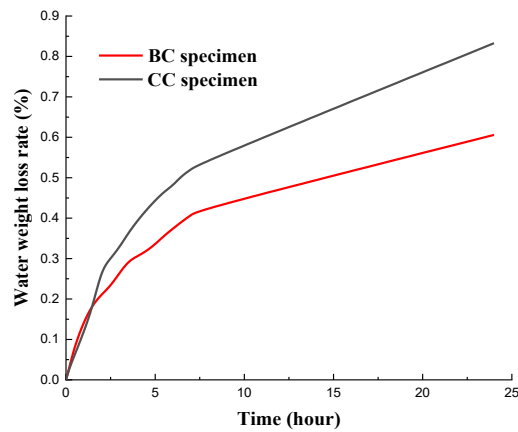


Fig. 3.12 Moisture loss speed comparison between BC and CC specimens.

During the first two hours, both BC and CC mortars exhibited comparable rates of water evaporation. Beyond the third hour, however, the BC specimens (red line) displayed a marked reduction in evaporation rate compared with the CC specimens (black line). This suggests that a higher proportion of free water was retained in the BC mortars. The incorporation of bitumen emulsion enhanced the water-retention capacity of the self-sensing mortars, allowing for greater preservation of moisture during curing. This improved water retention is expected to promote extended hydration and curing, thereby offering potential benefits for both the mechanical and electrical performance of the specimens.

3.4 Piezoresistive performance analysis

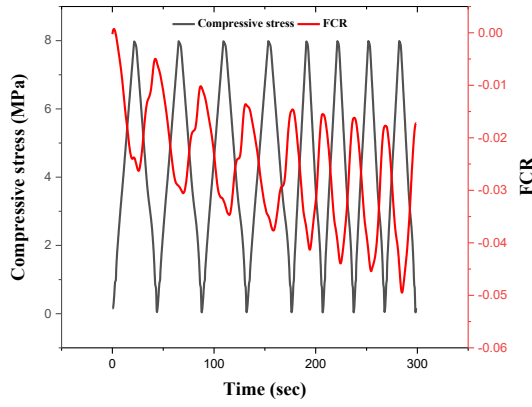
3.4.1 Piezoresistive comparison between BC and CC mortars

The water content influence on the piezoresistive performance of BC specimens was analysed in this study, due to the higher pore volume of BC samples. Based on Eq. (8):

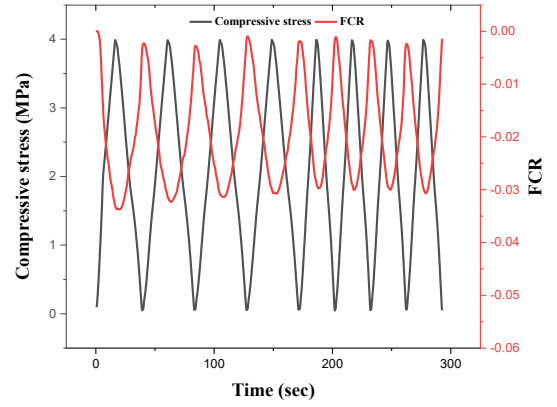
$$\frac{R-R_0}{R_0} \times 100\% = \text{FCR}\% \quad (8)$$

where the R is the resistance when the stress has been applied; R_0 is the initial resistance before loading (Rao et al., 2020). The dynamic loads were used for the self-sensing performance of specimens.

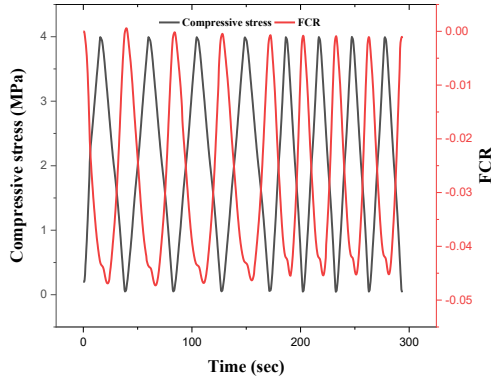
Fig. 3.14 and Fig. 3.15 present the piezoresistive performance of BC specimens. Under compression, the conductive fillers facilitated the formation of new electrical pathways. SEM analysis revealed significant accumulations of MWCNTs along both sides of microcracks, confirming their role in establishing conductive networks. As external loads varied, volumetric changes in the self-sensing specimens caused these electrical pathways to repeatedly form and break down during each loading cycle, thereby generating the observed piezoresistive response (Gupta et al., 2021; Song et al., 2023). After 60 days of curing, the piezoresistive behaviour of the specimens was evaluated. As shown in Fig. 3.13, BC specimens exhibited superior piezoresistive performance under varying loading rates compared with CC specimens. A similar investigation conducted by Dong et al. (Wong et al., 2020) examined the response of self-sensing sensors to different loading rates, reporting a FCR of less than 5% under constant compressive stress. By contrast, the BC specimens in this study demonstrated markedly enhanced performance, achieving an FCR of 14%.



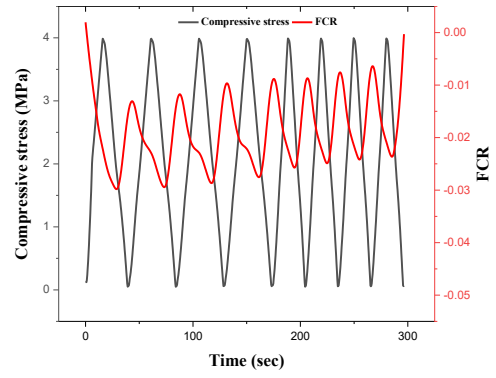
BC (0.05% MWCNTs)



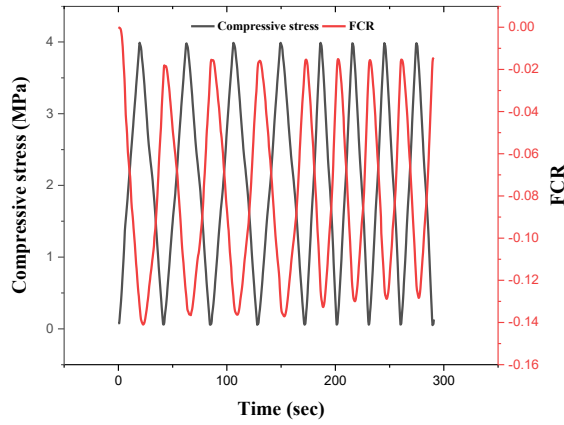
CC (0.05% MWCNTs)



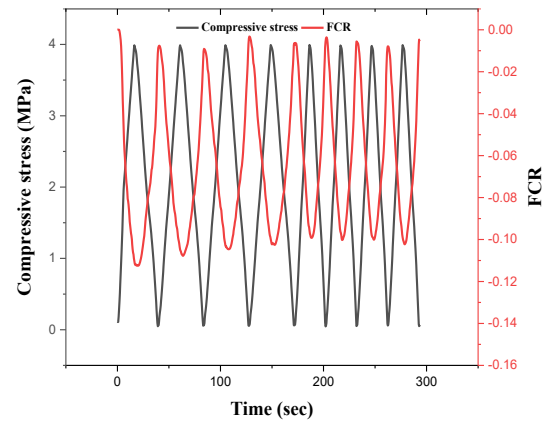
BC (0.07% MWCNTs)



CC (0.07% MWCNTs)



BC (0.5% MWCNTs)



CC (0.5% MWCNTs)

Fig. 3. 13. Electrical response performance comparison between BC mortars (left) and CC mortars (right).

Several studies have examined the piezoresistive behaviour of self-sensing cementitious sensors under varying loading rates, consistently reporting an inverse relationship

between loading rate and peak FCR values (W Dong et al., 2020; Dong, Li, Vessalas, et al., 2021). Comparable stability and sensitivity to loading changes were observed in the BC specimens of this study, aligning with these earlier findings. As illustrated in Fig. 3.16, the incorporation of bitumen emulsion increased the electrical resistance of the self-sensing composites. Furthermore, comparison of FCR values between BC and CC specimens (Fig. 3. 13) demonstrated that the piezoresistive performance of BC specimens was significantly enhanced by the addition of bitumen emulsion.

In the BC specimens, voids were observed at the interface between the bitumen and cement phases, along with additional micro-pores on the bitumen surface. As shown in Fig. 3.14, MWCNTs were frequently located along the surfaces of cracks and voids. These microstructural features facilitated the formation of new conductive pathways under applied loads. During cyclic loading, the repeated creation and disruption of these pathways produced larger variations in electrical resistance within the matrix. Consequently, BC composites demonstrated greater sensitivity to loading changes compared with CC specimens. This enhanced piezoresistive response suggests that BC sensors hold significant potential for advanced applications in TD and SHM.

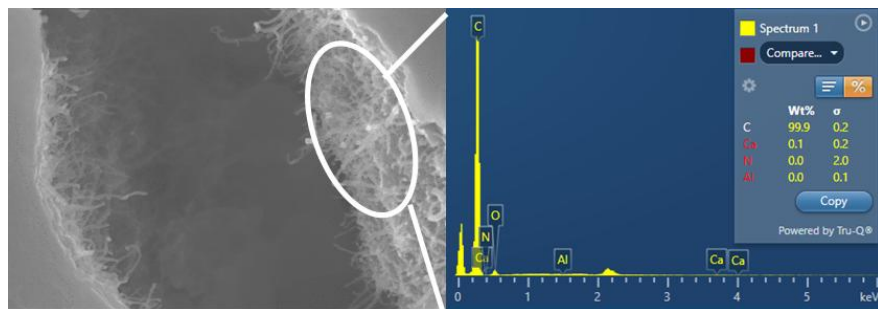


Fig. 3.14 SEM result about MWCNTs on microcracks in specimens and Dispersive Spectroscopy (EDS) results.

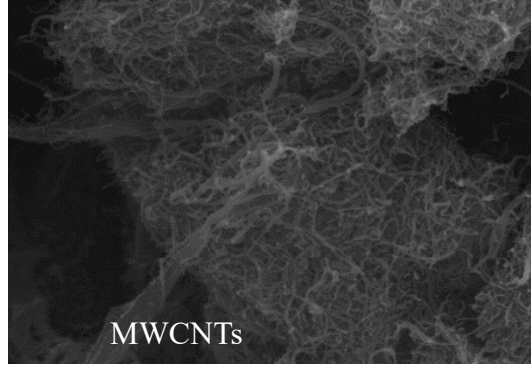


Fig. 3.15 SEM graphic of raw MWCNTs material.

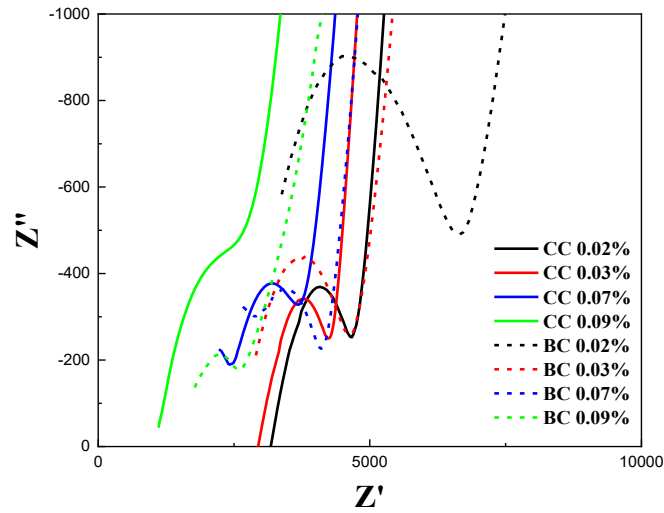


Fig. 3.16 Electrical resistance differences between BC and CC specimens.

3.4.2 Influence of water content on self-sensing performance

Conductive fillers were not the only factor influencing the piezoresistivity of the specimens. Electron conduction was also dependent on the formation of conductive pathways, which were closely related to the dispersion and interaction of fillers. As shown in Fig. 3.20, the incorporation of bitumen emulsion introduced additional micro-scale voids, a phenomenon similarly reported in previous studies (Jiang et al., 2021; X. Zhang et al., 2021). The piezoresistive performance of BC specimens under different moisture conditions is presented in Fig. 3.17, Fig. 3.18, and Fig. 3.19. Notably, specimens tested in a fully saturated state exhibited relatively low FCR values. Piezoresistivity was

subsequently measured after controlled moisture losses of 5%, 20%, and 50%. Within the 5–20% water loss range, the FCR increased and reached its maximum value. Beyond 20% water loss, however, the FCR began to decline, and by 50% water loss it stabilised, showing little further variation despite continued reductions in moisture content.

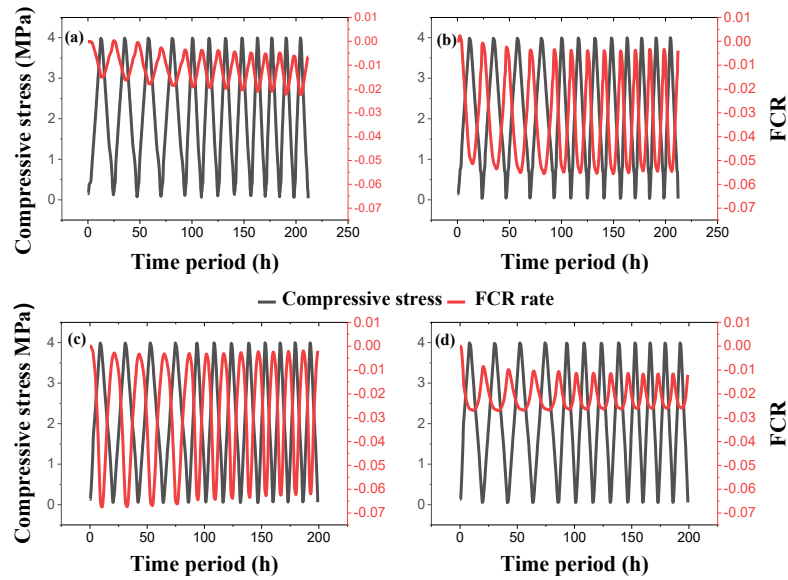


Fig. 3.17 0.05% MWCNTs BC specimens piezoresistivity test under different moisture percentages (a) saturated status; (b) 5% water weight loss; (c) 20% water weight loss; (d) 50% water weight loss.

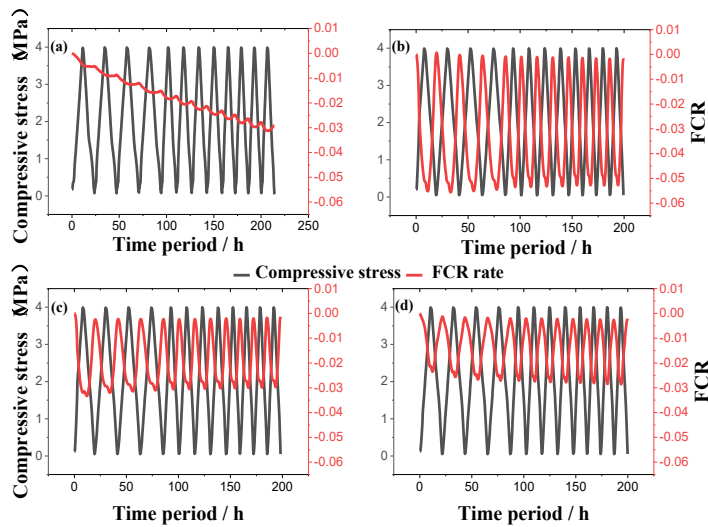


Fig. 3.18 0.07% MWCNTs BC specimens piezoresistivity test under different moisture contents: (a) saturated status; (b) 5% water weight loss; (c) 20% water weight loss; (d) 50% water weight loss.

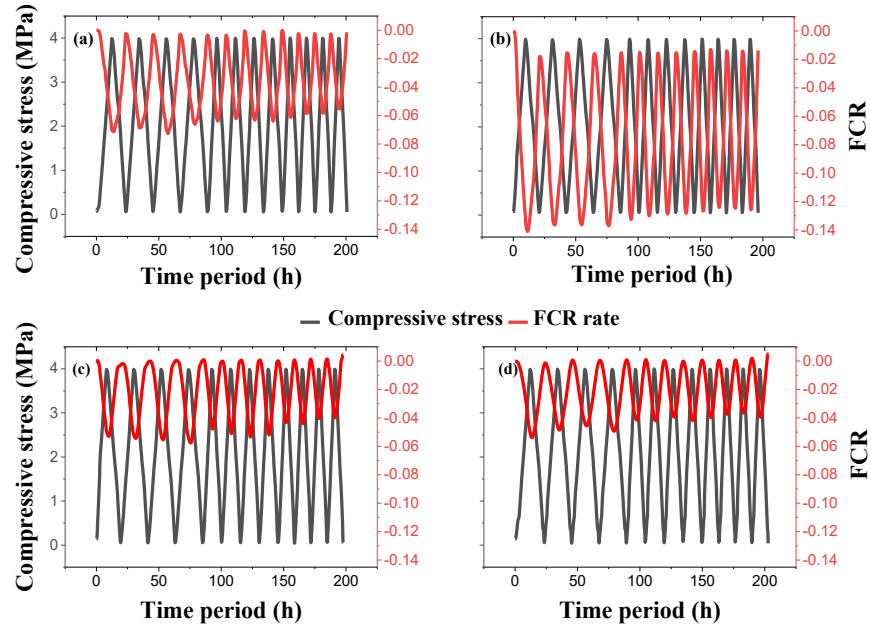


Fig. 3.19 0.5% MWCNTs BC specimens piezoresistivity test under different moisture contents: (a) saturated status; (b) 5% water weight loss; (c) 20% water weight loss; (d) 50% water weight loss.

Ion conduction was primarily governed by the transport of ions within the specimens. The ion concentrations in the pore solution are presented in Fig. 3.21. According to the ICP results, Na^+ , K^+ , and Ca^{2+} exhibited the highest concentrations. In CC specimens, however, the concentrations of these ions were relatively low compared with those reported in previous studies. Among them, Na^+ and K^+ ions played a dominant role in regulating electrical conductivity (Zhan et al., 2023b), as higher concentrations of these ions correspond to lower electrical resistance. It is important to note that elevated electrical conductivity can diminish piezoresistive sensitivity (Tian et al., 2019). Consistent with this principle, the findings demonstrated that BC composites exhibited enhanced piezoresistivity relative to CC specimens. To further assess the effect of ion conduction, additional experiments were conducted on specimens with different moisture contents, with the corresponding electrical responses shown in Fig. 3.22. When the self-sensing mortars were saturated, FCR values remained low. This behaviour can be

attributed to the ease of ion transport through micro-pores, where the combined conduction of ions and electrons reduced electrical resistance, resulting in only minor fluctuations under applied loading. Once water loss reached approximately 5%, however, a pronounced increase in FCR was observed, indicating that partial drying restricted ionic conduction and thereby enhanced piezoresistive responsiveness.

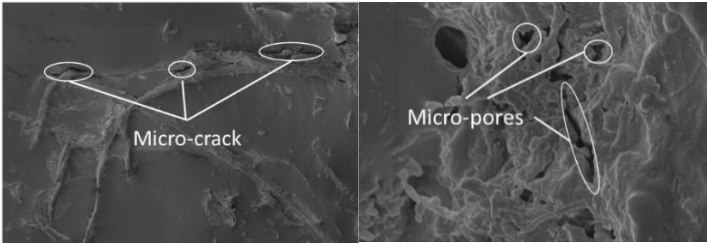


Fig. 3.20 SEM graphics of micro-surface of BC specimens.

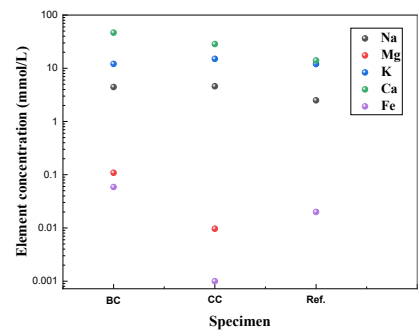


Fig. 3.21 ICP test results about the concentration of elements from BC specimens, CC specimens, and comparison with standard specimens (Zhan et al., 2023b).

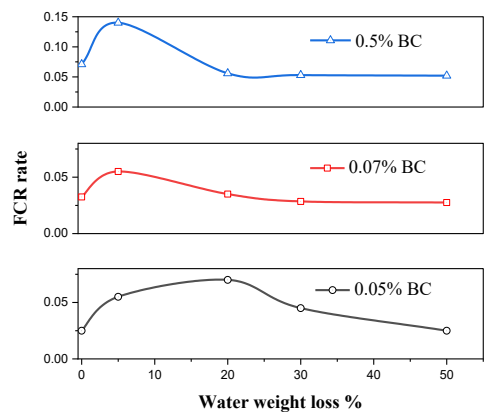


Fig. 3.22 Electrical response test results to present the moisture content influence on piezoresistivity.

With minimal water loss, the efficiency of ion conduction decreased, leading to an increase in the electrical resistance of the specimens. Under these conditions, a delicate balance was established between ionic and electronic conduction, and the slight reduction in ionic transport resulted in the FCR reaching its peak. As moisture loss progressed beyond this stage, the FCR began to decline. This reduction, however, was not indefinite, as the presence of conductive fillers provided stable electronic pathways. Once moisture loss reached approximately 30–50%, the influence of ion conduction on piezoresistivity became negligible, and the FCR stabilised at a consistent level. This conceptual relationship between water content and piezoresistivity is illustrated in Fig. 3.23.

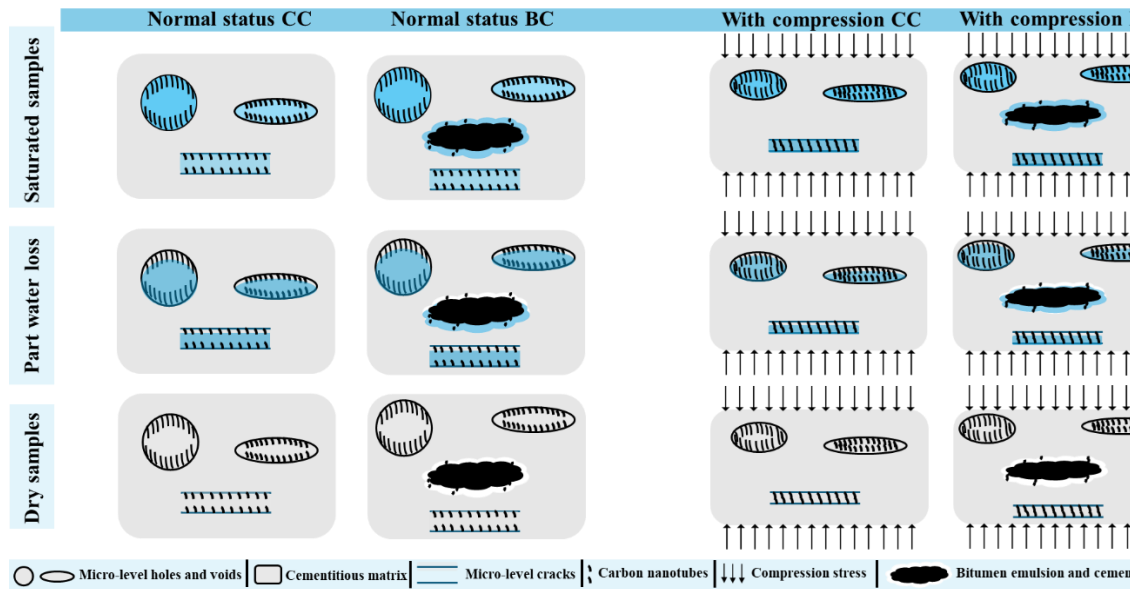


Fig. 3.23 Concept of the influence of water content on self-sensing performance.

Ion transfer within self-sensing mortars increased in proportion to the availability of free water. As shown in Fig. 3.20, microcracks at the cement–bitumen interface not only facilitated the formation of conductive pathways but also enhanced ion transport within the cementitious matrix. Consequently, the piezoresistive performance of BC mortars was strongly influenced by ionic conduction, which itself was affected by changes in moisture

content. When water loss exceeded 5%, piezoresistive performance began to decline in response to reduced ionic mobility. This trend is consistent with previous findings demonstrating that ions released during hydration reduce electrical resistance (Sun et al., 2019). The results further indicated that the impact of ionic conduction on piezoresistivity was dependent on the content of conductive fillers. For BC specimens with 0.05% filler content, the maximum FCR was observed at approximately 30% water loss, whereas specimens with 0.50% filler content achieved their peak FCR at only 5% water loss. In this study, the moisture loss level corresponding to the maximum FCR was defined as the Best Water Content for FCR. These findings highlight the critical role of moisture in governing the piezoresistive behaviour of self-sensing mortars and confirm the existence of an optimal water content ratio that maximises FCR.

3.5 Summary

The results indicate that the piezoresistive behaviour is strongly influenced by the moisture condition of the composite, with the optimum performance observed at approximately 95% saturation. This condition reflects a balance between ionic conduction and conductive network stability, which is closely related to the pore structure and water distribution within the material. This finding indicates that both water content and ion concentration within the pore solution strongly influence the self-sensing behaviour of the composites. To examine the effect of ion concentration in greater detail, Chapter 4 analyses the variations in ionic content of the pore solution and their implications for sensing performance.

In addition, changes in electrical resistance were closely linked to internal volume alterations, including pore development and crack propagation. This relationship suggests a strong correlation between strain variation, FCR, and pore structure evolution, thereby underscoring the role of microstructural changes in governing piezoresistive behaviour.

Chapter 5 therefore, addresses the interplay between strain development and piezoresistive performance, providing further insight into this mechanism.

Chapter 4: Influence of ion concentration on piezoresistivity in self-sensing sensors

This section describes the method adopted to mitigate chloride-induced corrosion resulting from the use of seawater. This consideration is critical for preserving both the mechanical integrity and electrical stability of the self-sensing cementitious composites, thereby ensuring the reliability of the experimental results.

4.1 Introduction

Compared with the limited reserves available on land, the oceans represent a far more abundant source of mineral and energy resources. In recent years, engineering advances have increasingly focused on the exploration and utilisation of these marine resources (Su et al., 2023). Seawater (SW) covers approximately 71% of the Earth's surface, with an estimated volume of 1.386 billion cubic kilometres. However, its high concentration of dissolved ions poses a significant risk of accelerating reinforcement corrosion, limiting its direct use in conventional reinforced concrete structures. The development of corrosion-resistant reinforcement materials has, nevertheless, opened new opportunities for seawater-based construction. The typical ionic composition of SW consists of approximately 55.1% chloride ions, 30.6% sodium ions, 2.3% calcium ions, and 1.1% potassium ions (Webb, 2021). Beyond the corrosive effects of chloride, the remaining three ions play a crucial role in influencing the electrical resistance of cement-based composites (Harris, 2022). Prior studies have demonstrated that alkali ions in solution, particularly sodium and potassium, reduce pore solution resistivity by enhancing ionic conductivity (Bu & Weiss, 2014; Snyder et al., 2003). Calcium ions, by contrast, affect conductivity through different mechanisms. The formation of calcium silicate hydrate (C–S–H) alters the pH of the pore solution, thereby modifying its conductive properties

(Masoumi et al., 2019) while also refining the pore network via hydration reactions, which further influences the electrical behaviour of cementitious composites (Cheng et al., 2013; Elakneswaran et al., 2009). Self-sensing cementitious sensors can be effectively integrated into concrete structures without significantly altering the ionic composition of the surrounding pore solution, even when seawater is used in the mix. Because these sensors are fabricated, cured, and matured prior to installation, they can provide reliable structural health monitoring and stress detection capabilities in marine or seawater-based environments.

4.2 Experimental program

4.2.1 Raw materials

General Purpose (GP) cement conforming to AS 3972 (Standard, 2010) was employed as the binder, while natural river sand served as the sole aggregate in mortar specimen preparation. The water–cement ratio was fixed at 0.4. Three types of mixing water were used to prepare the specimens: pure seawater (SW 100), a 1:1 mixture of seawater and tap water (SW 50), and pure tap water as the control (CO). To enhance self-sensing capability and electrical conductivity, multi-walled carbon nanotubes (MWCNTs) were incorporated as conductive fillers (Konsta-Gdoutos & Aza, 2014). In order to determine the percolation threshold of the self-sensing cementitious sensors, varying MWCNT dosages, 0.1, 0.3, 0.5, 0.7, 1.0, 1.5, and 2.0 wt% of the cement content, were introduced across all specimen groups. The detailed mixture compositions are provided in Table 4.1.

Table 4.1 The components in each specimen group

Specimen Type	CO	SW 50	SW 100
GP (g)	275	275	275
W/C	0.4	0.2	0
M/C	0.1, 0.3, 0.5, 0.7, 1.0, 1.5, 2.0	0.1, 0.3, 0.5, 0.7, 1.0, 1.5, 2.0	0.1, 0.3, 0.5, 0.7, 1.0, 1.5, 2.0
S/C	1.5	1.5	1.5
SW/C (wt)	0	0.2	0.4

Note: GP: General-Purpose cement; W/C: water/cement; M/C: MWCNTs/cement; S/C: sand/cement; SW/C: seawater/cement.

Standard seawater (SW) was prepared in accordance with ASTM D1141 (Testing & Materials, 2013), which provides a composition representative of natural ocean water (P. Li et al., 2023). The concentrations of the individual components used in the SW formulation are presented in Table 4.2. To maintain a constant pH of 8.2, an appropriate amount of NaOH was added to the solution.

Table 4.2 Details of the components for SW preparation

Component	NaCl	MgCl ₂	KCl	CaCl ₂	Na ₂ SO ₄
Concentration (g/L)	24.53	5.20	0.695	1.16	4.09

4.2.2 XRD test

After 28 days of curing, the samples were ground into powder and oven-dried at 35 °C for one week prior to XRD analysis. This relatively low drying temperature was selected to prevent decomposition of self-healing products, as confirmed by TGA results. XRD measurements were performed using an Ultima IV diffractometer equipped with a copper target, and the diffraction data were qualitatively analysed with High Score Plus software. The scanning range was set between 10° and 60° (2θ). The powdered samples were prepared by top-filling into the sample holder.

4.2.3 Other methodologies

The methodologies employed for sample preparation, compressive strength testing, TGA, SEM, EDS, early hydration heat analysis, ACIS, and ICP followed the procedures outlined in Chapter 3.

4.3 Effect of SW on hydration and strength

The heat flow and cumulative heat release results, presented in Figs. 4.1 and 4.2, indicate that seawater (SW) accelerates the hydration of cement. Specimens prepared with 50%

and 100% SW exhibited higher peak heat fluxes (0.184 W/g and 0.187 W/g, respectively) compared with the control (CO). The 100 SW specimen reached its maximum heat flow at approximately 6.6 hours, and the 50 SW specimen at around 6.7 hours, almost three hours earlier than the CO group. By contrast, the CO specimen attained a lower peak heat flow of 0.132 W/g, representing a reduction of approximately 28.84% relative to the SW mixtures.

As shown in Fig. 4.2, after 72 hours of hydration, the cumulative heat release of the 50 SW specimen was the highest at 261 J/g, followed by the 100 SW specimen at 250.48 J/g. These values were 14.24% and 9.63% greater, respectively, than those of the CO specimen. These findings demonstrate that the inclusion of SW not only accelerates early-age hydration but also increases the total heat released. Qu et al. (2021) similarly reported that chloride ions in seawater accelerate cement hydration. This effect is attributed to the elevated concentrations of Cl^- , SO_4^{2-} , and Na^+ in seawater, which act as catalysts to accelerate the hydration of alite (C_3S) (Sun et al., 2021). In addition, dissolved Ca^{2+} ions in SW contribute to the formation of hydration products, further enhancing hydration (Yaseen et al., 2020).

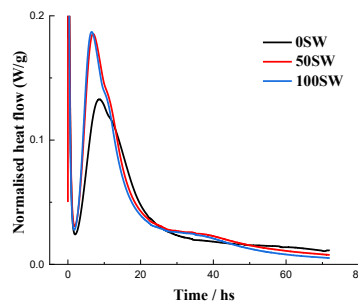


Fig. 4.1 IC test results of early hydration heat release flow curve in 72 hours

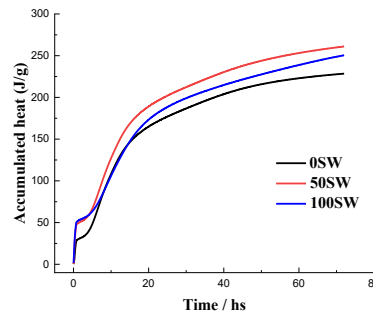


Fig. 4.2 Accumulated heat of three types of specimens in 72 hours

After 28 days of curing, XRD analysis of paste fractions from the CO, 50 SW, and 100 SW specimens is shown in Fig. 4.3. The principal phases identified included C_3S (32.1° and 41.2°), C_2S (32.5°), calcium silicate hydrate ($\approx 31^\circ$), portlandite (18°), ettringite (9.1° , 15.8° , and 23°), and calcite (23.1°). The presence of C_3S and C_2S confirmed the persistence of unhydrated clinker minerals, while the remaining phases were associated with hydration products. In addition, quartz was detected at several diffraction angles, including 20.8° , 23.9° , 26.7° , 29.4° , 36.4° , and 42.4° , indicating the presence of mineral phases beyond those typically observed in hydrated cement paste. A notable distinction was the formation of Friedel's salt, which was predominantly detected in SW-containing specimens. Its characteristic peaks at 12° , 23° , and 39.3° were more pronounced in the 50 SW and 100 SW groups than in the CO specimens, reflecting the higher chloride availability in seawater mixes. By contrast, diffraction peaks corresponding to ettringite (9.1° , 15.8° , and 23°) exhibited little variation among the mixtures, suggesting that seawater did not significantly influence its formation. Hydrotalcite, with peaks at 12° , 23° , and 35.8° , was found in considerable quantities in the SW specimens, likely due to the abundance of magnesium and sulphate ions. Seawater also promoted the formation of monosulphate phases, evidenced by peaks at 11.3° , 30.3° , and 35.1° , consistent with the elevated sulphate concentrations in the SW mixtures (Ragab et al., 2016). Brucite was detected at 18.6° , 38° , and 50.9° , though only as weak peaks, likely reflecting the

relatively low magnesium content of seawater, much of which had already been consumed in forming hydrotalcite and monosulphate. Overall, the use of seawater altered the phase composition of the cementitious composites by generating additional reaction products absent in the control specimens. The formation of these phases not only reduced the availability of free water but was also associated with higher hydration heat release, which is expected to contribute to the improved compressive strength of SW-based samples.

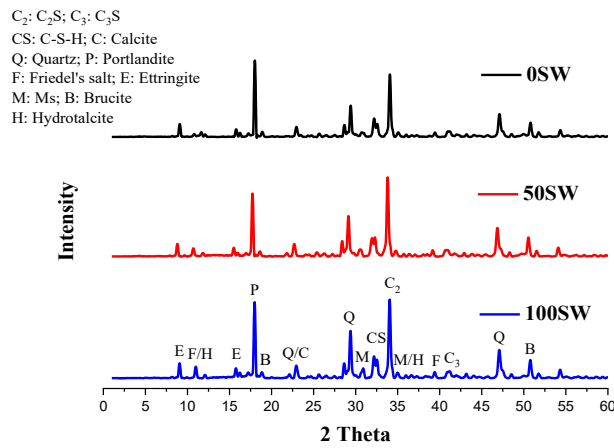


Fig. 4.3 XRD results of three types of specimens after 28 days curing

Fig. 4.4 presents the TGA results, which reflect the typical products of cement hydration. The corresponding DTG curves display three distinct peaks. The first major mass loss, observed between 50 and 200 °C, is attributed to the evaporation of free water, the release of bound water from Friedel's salt, and the partial decomposition of calcium silicate hydrate. The second peak, occurring between 400 and 500 °C, corresponds to the thermal decomposition of portlandite. The third peak, detected between 600 and 750 °C, is associated with the decarbonation of calcite (Lu et al., 2018). Notably, the 50 SW and 100 SW specimens exhibited greater mass losses in the 450–500 °C range compared with the CO group. Specifically, the 50 SW specimens lost 1.38% of their total mass, whereas the CO group lost 1.19%. This additional loss is likely due to the release of coordinated water and the thermal decomposition of M–S–H gel (Tartaglione et al., 2008). The formation of

M–S–H gel is attributed to the presence of free Mg^{2+} ions in seawater, although its poorly crystalline or amorphous structure renders it undetectable by XRD. Owing to the relatively low magnesium content of seawater, however, the overall difference in mass loss between SW and CO specimens remained modest. In the 600–750 °C range, the 50 SW and 100 SW specimens exhibited mass losses of 2.5% and 2.7%, respectively, compared with 2.3% for the CO specimens. Although this variation is minor, the additional mass loss may be associated with the thermal decomposition of MgCO_3 (Jin & Al-Tabbaa, 2013; P. Li et al., 2020).

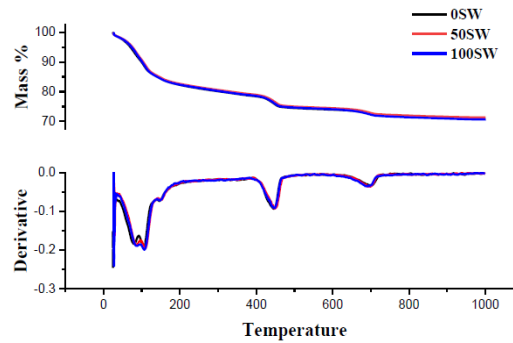


Fig. 4.4 TGA test results of 0SW, 50SW, and 100 SW specimens after 28 days curing Fig. 4.5 presents the compressive strength results of specimens prepared with and without SW. The 100 SW group achieved the highest strength at 75.09 MPa, followed by the 50 SW group at 62.16 MPa. In contrast, the CO specimens reached 57.18 MPa, representing reductions of 8.71% and 31.32% relative to the 50 SW and 100 SW groups, respectively. These strength developments are consistent with the enhanced hydration identified through the XRD and TGA analyses. The improvement can be attributed to the presence of chloride and sulphate ions in seawater, which promote hydration reactions, thereby contributing to the increased compressive strength, as confirmed by both mineralogical and thermal characterisations (Qu et al., 2021).

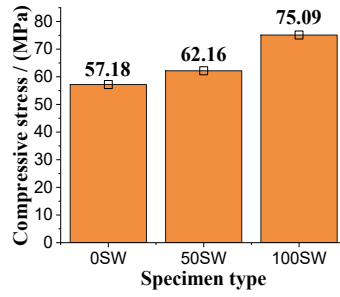


Fig. 4.5 Compressive strength test results of 0SW, 50SW, and 100SW specimens.

4.4 Ion concentration analysis

The ionic concentration of the pore solution plays a critical role in determining the electrical resistance of cementitious composites. In this study, the concentrations of Na^+ , K^+ , Ca^{2+} , Fe^{2+} , and Mg^{2+} were examined, with the ICP results shown in Fig. 4.6. To replicate the ionic composition of seawater, salts such as NaCl , MgCl_2 , KCl , and CaCl_2 were incorporated into the SW mixes (Table 4.2). In addition, the GP cement used as the binder contained minor amounts of K_2O , Fe_2O_3 , and Na_2O , which also contributed to the overall ionic content (Zhan et al., 2023b). Thus, this analysis focused on five key metal ions, accounting for contributions from both seawater and the cement matrix. Marked differences were observed in sodium ion concentrations between the SW specimens and the CO group. This can be attributed to the inherently high Na^+ content of seawater, which introduced significantly more free sodium ions into the pore solution. In contrast, Ca^{2+} concentrations remained relatively consistent across all specimen types. This stability is likely due to the incorporation of free calcium ions into hydration products, a process also supported by the TGA results. Specifically, the greater mass loss observed in the 600–800 °C range suggests a higher quantity of Ca^{2+} -bearing hydration products in the SW specimens compared with the control group. The enhanced compressive strength of the SW specimens further corroborates the increased formation of calcium-based hydration compounds.

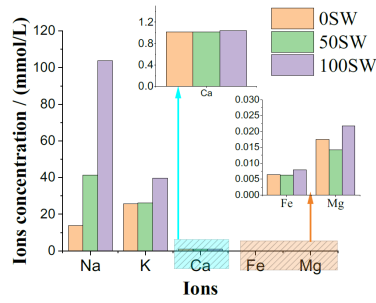


Fig. 4.6 The ion concentration of five metal ions in the pore solution

Because Na^+ , K^+ , and Ca^{2+} ions exert a pronounced influence on piezoresistive behaviour, their concentrations were measured separately. A clear correlation was observed between electrical resistance and the ionic concentration of the pore solution (Deng et al., 2024). As illustrated in Fig. 4.6, elevated concentrations of Na^+ , K^+ , and Ca^{2+} reduced the electrical resistance of the specimens, thereby diminishing piezoresistive sensitivity. This trend is consistent with both the present findings and previous reports (Dong et al., 2019). Conversely, the high concentration of chloride ions in seawater promoted cement hydration, producing a denser microstructure with finer pores and consequently increasing the electrical resistance of the specimens. Thus, while free alkali ions such as Na^+ and K^+ enhance conductivity, chloride ions contribute to microstructural densification, which raises resistivity. This dual mechanism may explain why the 50 SW specimens exhibited superior piezoresistive performance compared with both the CO and 100 SW groups.

4.5 Percolation thresholds of self-sensing cementitious sensors

Electrical impedance spectroscopy was employed to determine the percolation thresholds of the self-sensing cementitious specimens. The results for the 50 SW group are presented in Fig. 4.7. In the impedance spectra, the horizontal axis represents the real component, corresponding to the electrical resistance of the cementitious matrix, while the vertical axis represents the imaginary component, corresponding to the electrical reactance. The intercept on the real axis indicates the intrinsic resistance of the specimen. This method

enables the quantification of the contribution of each phase within the composite matrix to the overall resistance (Gallardo & Provis, 2021). Unlike conventional DC resistance measurements obtained with multimeters, EIS measurements across a wide frequency range provide deeper insight into the internal structure of the mixture and minimise polarisation effects, which are common in semi-conductive materials (Wen, 2020). Based on the impedance test results, the percolation thresholds of the self-sensing specimens were identified, as shown in Fig. 4.8. These thresholds define the optimal operating range in which the composites exhibit effective piezoresistive behaviour. Within this range, the matrix demonstrates the maximum FCR, reflecting the highest sensitivity to externally applied mechanical loading (Deng et al., 2025).

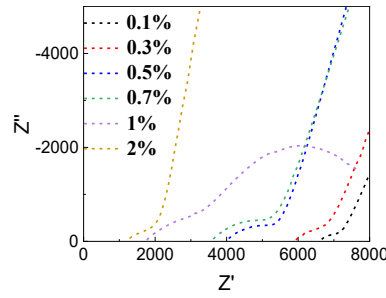


Fig. 4.7 Electrical impedance test results (50 SW, representative of the rest)

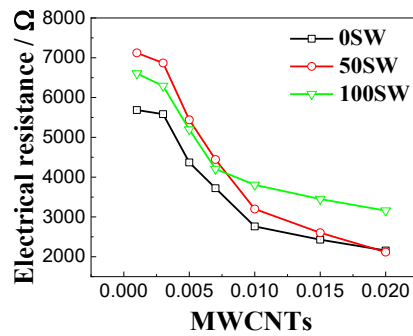


Fig. 4.8 Percolation thresholds of three types of self-sensing cementitious sensors

Fig. 4.8 presents the percolation thresholds of the three types of self-sensing cementitious sensors. Specimens prepared with 100% SW exhibited higher electrical resistance, which can be attributed to the absorption of chloride ions by C–S–H gel, resulting in a reduction

of macropore volume within the matrix (Li et al., 2021). The percolation threshold curve for the 100 SW specimens was located above that of the CO group, reflecting the higher resistance. Furthermore, the percolation threshold range narrowed in the 100 SW specimens: while the upper bound remained at 0.3 wt% MWCNTs, the lower bound shifted upward to 0.7 wt%, as indicated by the inflection in the impedance curve beyond this concentration.

By contrast, the percolation threshold range of the 50 SW specimens, spanning 0.3–1.0 wt% MWCNTs, was comparable to that of the CO specimens. However, the 50 SW group exhibited a wider distribution of electrical resistance within this range, accompanied by a steeper slope of the impedance curve. This behaviour is primarily attributed to the presence of Na^+ , K^+ , and Ca^{2+} ions, which strongly influence electrical resistance (Zhan et al., 2023b) and, by extension, affect the piezoresistive performance of the specimens.

4.6 Piezoresistivity

The piezoresistive behaviour of self-sensing sensors containing 0.5% and 0.7% MWCNTs was evaluated through their electrical response, expressed as the FCR according to Eq. (1), with the results presented in Fig. 4.9. Under cyclic loading and unloading, the FCR exhibited a response that closely mirrored the applied mechanical stress. This behaviour is attributed to changes in pore volume during compression, which facilitate the formation of new conductive pathways within the cementitious matrix (Wang et al., 2025).

		Stress-FCR
MWCNTs wt%	0.5	0.7

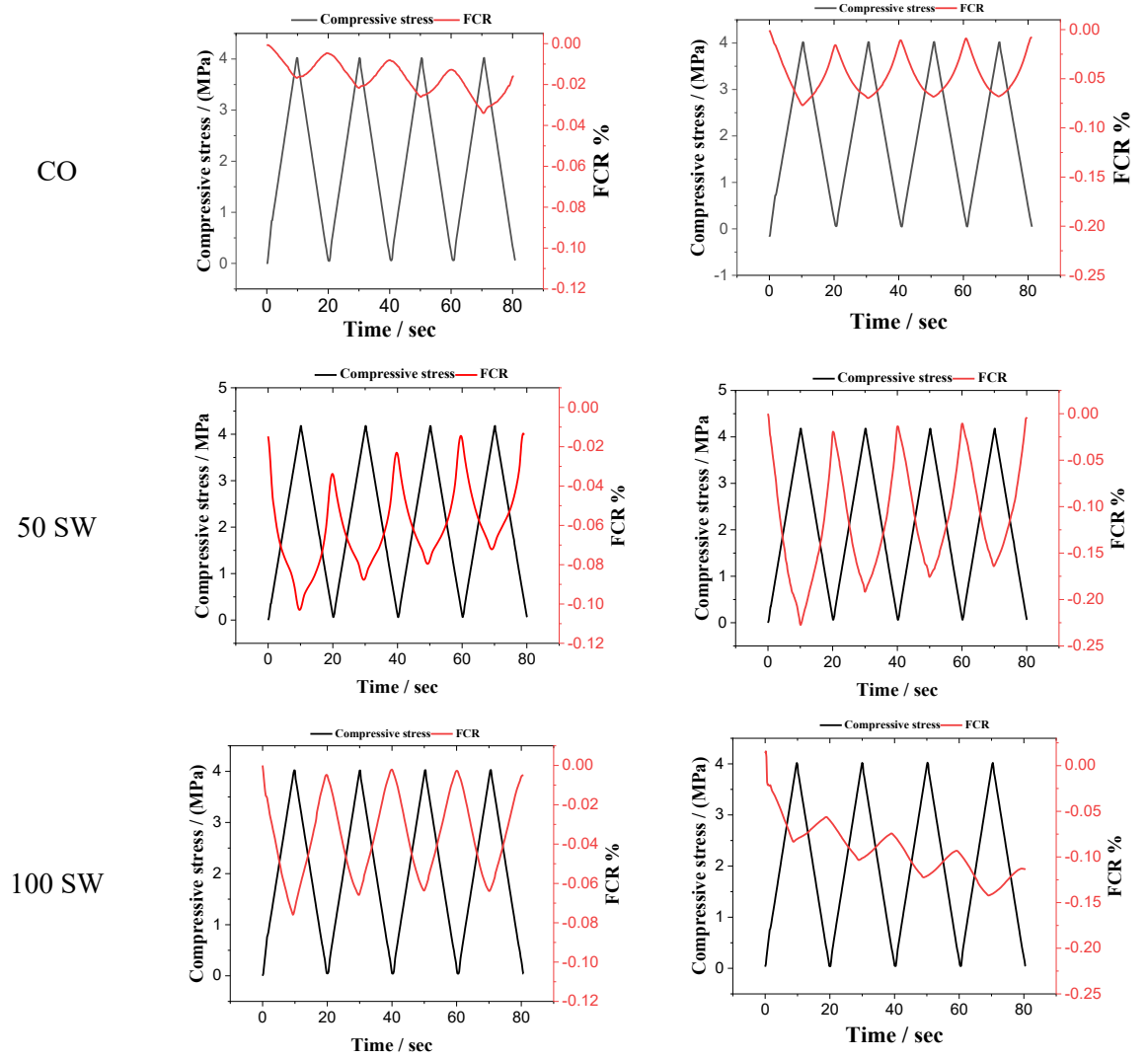


Fig. 4.9 Compressive stress and FCR rate relationship of three types of self-sensing cementitious sensors

Fig. 4.9 presents the results of the self-sensing tests. The SW specimens exhibited greater sensitivity to mechanical loading than the CO group, as reflected by the larger changes in fractional change in resistance (FCR). This enhancement was more pronounced in specimens containing 0.7 wt% MWCNTs compared with those containing 0.5 wt%. For example, at 0.5 wt%, the CO specimens achieved a maximum FCR of approximately 0.035%, whereas the 100 SW specimens reached 0.065%. The 50 SW group demonstrated the highest performance, achieving an FCR of about 0.100%. A similar trend was observed at 0.7 wt% MWCNTs: the CO specimens attained an FCR of around 0.070%,

which increased to 0.140% in the 100 SW group. Notably, the 50 SW specimens again outperformed the others, reaching a maximum FCR of 0.370%.

These findings demonstrate that conductive fillers contribute to the formation of conductive networks, while the presence of free ions in the pore solution of SW specimens increases the number of conductive pathways generated under mechanical loading. However, direct comparison between the 50 SW and 100 SW groups highlights a diminishing effect at high ion concentrations. Specifically, the 100 SW specimens exhibited lower FCR values than the 50 SW group. This behaviour can be explained by the inherently lower electrical resistance of the 100 SW specimens: the elevated ion concentration creates abundant conductive channels even in the unloaded state, thereby limiting the capacity to form additional pathways under applied stress. Consequently, further increases in ion concentration beyond an optimal threshold do not enhance, and may even impair, piezoresistive performance.

Overall, the results suggest that there exists an optimal combination of MWCNT dosage and ionic concentration that maximises piezoresistivity. Both 50 SW and 100 SW specimens possessed more extensive conductive networks prior to loading compared with the CO group. However, under cyclic compression and release, the CO specimens generated fewer new conductive pathways despite containing the same amount of MWCNTs. This distinction underscores the role of free ions in the pore solution of SW-based specimens, which enhances conductive network formation and leads to superior FCR responses relative to specimens prepared with tap water.

4.7 Chloride diffusion influence and resistance method

4.7.1 Introduction

Given that seawater was incorporated into self-sensing specimens to alter ion concentrations, this study was extended to investigate an effective approach for improving chloride diffusion resistance. Previous research has demonstrated that bitumen emulsion-modified concrete exhibits excellent durability, with enhanced diffusion resistance identified as a key contributing factor (Liu et al., 2025; Rajhans et al., 2019; Shi et al., 2024; H Zhang et al., 2021; Zhou & Shi, 2025). Zhang et al. (H Zhang et al., 2021) reported that the incorporation of an optimal dosage of bitumen emulsion significantly improves the long-term performance of cementitious mixtures. Similarly, other studies have shown that the inclusion of bitumen enhances the resistance of cement-bitumen composites to sulphate ion ingress (Li et al., 2022). Despite these advances, limited research has specifically examined the chloride diffusion resistance of composites incorporating either cationic or anionic bitumen emulsions. To address this gap, the present study systematically evaluates the chloride ion diffusion performance of such mixtures. A comprehensive suite of characterisation techniques, including SEM, EDS, XRD, and TGA, was employed to validate and substantiate the experimental findings, thereby providing new insights into the application potential of bitumen emulsions as organic additives for enhancing the durability of cementitious materials.

4.7.2 Raw materials

GP cement conforming to AS 3972 specifications was used as the binder in this study. Two types of bitumen emulsions—anionic and cationic—were incorporated into the cement-bitumen mixtures. The mixing procedure involved the dry blending of cement and river sand in a Hobart mixer, followed by the gradual addition of bitumen emulsion and water. The bitumen emulsions, supplied by Boral Construction Company, had their

properties summarised in Table 4.3. Specimens were cast in cylindrical moulds measuring 100 mm in diameter and 200 mm in height. Three groups were prepared: cementitious composites incorporating cationic bitumen emulsion (CSS), those with anionic bitumen emulsion (ASS), and a control group without bitumen emulsion (CO). The detailed mix proportions for each group are listed in Table 4.4. All specimens were cured for 28 days before testing.

Table 4.3 The properties of bitumen emulsion

	Bitumen	Water	Emulsifier	Boiling point	pH	Expected use temp	Max use temp
Anionic/Cationic bitumen emulsion	65%	35%	<2%	100 °C	>10	20 °C	70 °C

Table 4.4 Mixture design of the cementitious cylinder samples

Elements	Cement	W/C ratio	CB/C ratio	AB/C ratio	S/C ratio
CSS	1 kg	0.35	0.25	--	2.0
ASS	1 kg	0.35	--	0.25	2.0
CO	1 kg	0.35	--	--	2.0

Note: W/C: water/cement ratio; CB/C: cationic bitumen/cement ratio; AB/C: anionic bitumen/cement ratio; S/C: sand/cement ratio. The water content in the bitumen emulsion was considered.

4.7.3 Rapid migration, rapid chloride penetration, and volume of permeable void test methodologies

The non-steady-state chloride migration coefficient was determined in accordance with the NT Build 492 standard testing protocol (492, 1999). Cylindrical specimens (100 × 200 mm in size) were first cast, and disk samples (100 × 50 mm) were subsequently

extracted from the midsection. For the test, a 10% NaCl solution served as the chloride source, while a 0.3 N NaOH solution was used as the catholyte. A voltage in the range of 60–300 V was applied to drive chloride ions through the specimens. The duration of testing varied between 6 and 24 hours, depending on the applied voltage and specimen response. Following the test, each specimen was split, and a 0.1 mol/L AgNO₃ solution was sprayed onto the freshly fractured surface. The resulting colour changes clearly delineated the chloride penetration front, confirming ion transport under non-steady-state conditions. A representative specimen cross-section, with AgNO₃ marking the chloride diffusion boundary, is shown in Fig. 4.10.

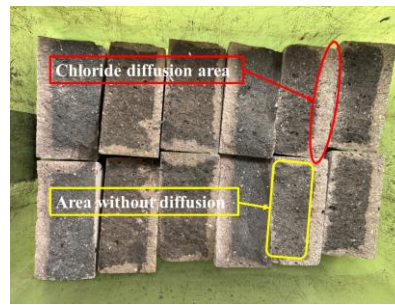


Fig. 4.10 Illustrating the specimens after the NT-Build 492 test

The Rapid Chloride Permeability Test (RCPT) was employed to evaluate chloride resistance performance, with a reduced applied voltage of 10 V (Noushini & Castel, 2018). Although the standard RCPT protocol prescribes a voltage of 60 V, this was avoided in accordance with ASTM C1202 due to the risk of specimen overheating (Gomes et al., 2025). Three specimens from each group, CSS, ASS, and CO, were tested. Disk samples measuring 100 mm in diameter and 50 mm in thickness were prepared by saw-cutting the central portion of the originally cast 100 × 200 mm cylindrical specimens. The data acquisition system used for testing is illustrated in Fig. 4.11.



Fig. 4.11 The data acquisition system of RCPT

The volume of permeable voids (VPV) was determined in accordance with ASTM C642 to assess the interconnected pore structure of the cementitious mixtures. Specimens were first oven-dried at 105 °C until a constant mass was achieved, recorded as M_{dry} . They were then immersed in water until full saturation was reached, and the saturated mass was recorded as M_{sat} . Finally, each specimen was weighed while submerged in water to obtain the submerged mass, denoted as M_{sub} . Using these three mass values, Eq (1) was used to figure out the VPV.

$$\text{VPV}(\%) = \frac{M_{\text{sat}} - M_{\text{dry}}}{M_{\text{sat}} - M_{\text{sub}}} \times 100\% \quad (1)$$

4.7.4 Methodologies

The methodologies employed for sample preparation, compressive strength testing, TGA, SEM, EDS, and XRD followed the procedures outlined in Chapters 3 and 4.

4.7.5 Chloride diffusion performance

The NT Build 492 test method was employed to evaluate the chloride migration resistance of the CSS, ASS, and control specimens. During the test, key parameters, including the initial and final temperatures of the NaOH solution, specimen thickness, chloride penetration depth, and applied voltage, were recorded. Using Eq. (4), the non-steady-state migration coefficient, $D_{\text{nssm}} (\times 10^{-12} \text{m}^2/\text{s})$ was calculated, indicative of chloride ion movement through the cementitious matrix.

$$D_{nssm} = \frac{0.0239(273+T)L}{(U-2)t} (x_d - 0.0238 \sqrt{\frac{(273+T)Lx_d}{U-2}}) \quad (2)$$

Where U: absolute value of the applied voltage; T: average value of the final and initial temperature of the solution; L: thickness of samples; t: test duration; x_d : penetration depths. The control group's chloride migration coefficient was $15.10 \times 10^{-12} \text{ m}^2/\text{s}$, while the ASS specimens had a lower value of $11.42 \times 10^{-12} \text{ m}^2/\text{s}$. The CSS samples resulted in the lowest migration coefficient at $8.36 \times 10^{-12} \text{ m}^2/\text{s}$, which means they were the most resistant to chloride penetration amongst the three groups investigated. Fig. 4.12(a) presents the chloride penetration profiles. The results clearly demonstrate that the incorporation of bitumen emulsion effectively reduced chloride migration within the specimens. Among the two emulsion types evaluated, the cationic bitumen emulsion exhibited superior performance, providing the greatest resistance to chloride diffusion.

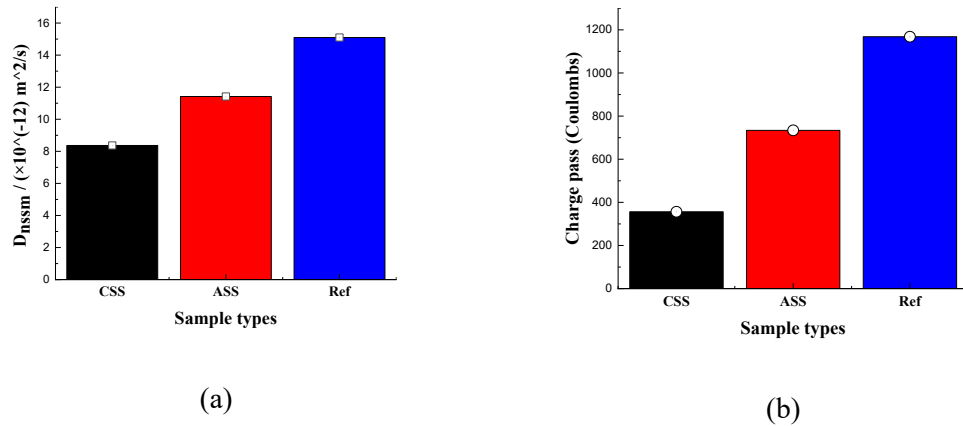


Fig. 4.12 The chloride diffusion test results (a) NT-build 492 result; (b) RCPT result. The RCPT was conducted to validate the findings of the NT Build 492 test, with the results shown in Fig. 4.12(b). Three specimens from each group were tested, and the reported values represent the average charge passed under a constant voltage of 10 V over 6 hours. The CSS specimens exhibited the lowest charge passed (356.29 Coulombs), indicating superior resistance to chloride permeability. The ASS group showed a

moderate value of 733.74 Coulombs, while the control group recorded the highest charge at 1168.14 Coulombs. These RCPT results corroborate the NT Build 492 findings, confirming that the incorporation of bitumen emulsion reduces chloride ion penetration, with CSS specimens demonstrating the greatest resistance among the modified mixes.

4.7.6 Analysis of chloride diffusion influence on specimens

Following the NT Build 492 test, XRD analysis was performed on the cementitious specimens, with the results presented in Fig. 4.13. As river sand was employed as the fine aggregate, distinct diffraction peaks corresponding to minerals such as kaolinite, quartz, and zeolites were observed. In addition, broad peaks at approximately 25.5° and 36° were detected, indicative of the presence of an amorphous phase, most likely associated with the formation of C–A–S–H gel (Tahri et al., 2021).

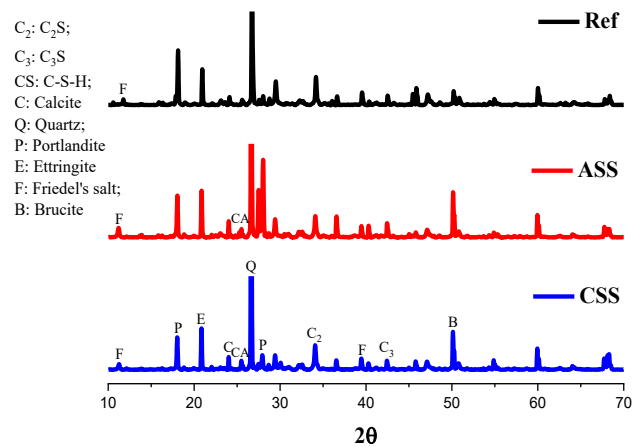


Fig. 4.13 The XRD results of three types of specimens after the NT-build 492 diffusion test

Distinct diffraction peaks at approximately 11.3° and 21° confirmed the presence of Friedel's salt, while a weaker peak near 40° may be attributed to the incorporation of chloride ions. In specimens containing anionic bitumen emulsion (ASS), a greater amount of chloride ions likely interacted with C_3A , leading to increased formation of Friedel's salt. Owing to variations in crystal structure, the diffraction peak of Friedel's salt

produced through C₃A–chloride reactions can shift toward 27.5°. Consistent with this expectation, the XRD data for the ASS specimens displayed a pronounced peak near 27.5°, corroborating this interpretation.

TGA was employed to identify phase transformations and decomposition products within the temperature range of 25–1000 °C. As illustrated in Fig. 4.14, distinct mass loss regions were associated with specific decomposition processes. Water evaporation occurred between 50 °C and 150 °C, while C–S–H predominantly decomposed below 200 °C. Ca(OH)₂ decomposition was observed between 400 °C and 600 °C, and CaCO₃ decomposition occurred between 600 °C and 800 °C. In addition, mass loss between 230 °C and 410 °C indicated the thermal decomposition of Friedel’s salt, consistent with the findings of (Shi et al., 2017). Friedel’s salt also undergoes dehydration between 30 °C and 150 °C, losing up to six water molecules, which accounts for the pronounced mass loss in this temperature range. Table 4.5 summarises the mass loss percentages observed across the different thermal regions. The DTG results showed a distinct mass-loss peak between 100 °C and 150 °C, with the ASS and CO specimens exhibiting greater losses compared with the CSS specimens (1.48%). This finding suggests a lower quantity of free or loosely bound water in CSS specimens. Furthermore, decomposition of portlandite between 400 °C and 600 °C provided additional confirmation of its presence in the specimens (Yin et al., 2013). Within this range, CSS specimens exhibited a mass loss of approximately 2.03%, lower than that of the ASS group. Given that the flash point of bitumen exceeds 250 °C, as specified in AS 2008, the CO specimens were excluded from this comparison to avoid potential interference from bitumen-related thermal degradation.

Table 4.5 Mass loss percentage in each temperature range based on TGA

Temperature range	100-150 °C	400-600 °C	600-800 °C
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ASS	2.01 wt%	3.12 wt%	5.67 wt%
CSS	1.48 wt%	2.03 wt%	4.23 wt%
CO	1.69 wt%	2.37 wt%	2.68 wt%

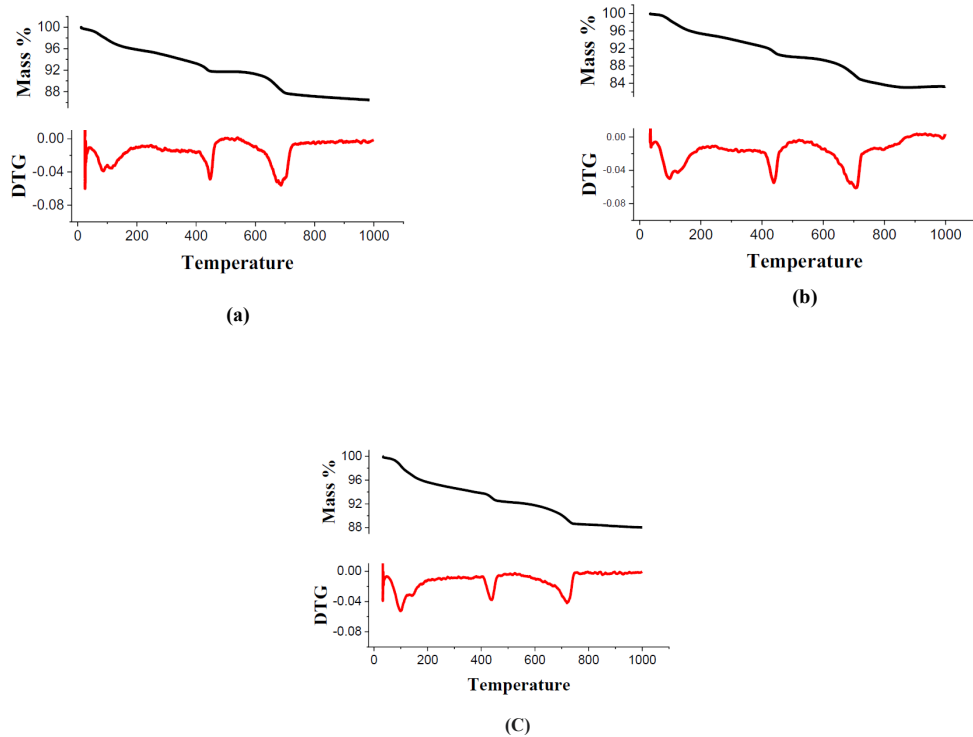


Fig. 4.14 TGA test results after chloride diffusion test (a) CSS; (b) ASS; (c) controlled group

The microstructure of the bitumen–cement composites was examined using SEM and EDS. Fig. 4.15(a) illustrates the surface morphology of bitumen in the CSS specimens, while Fig. 4.15(b) presents the corresponding EDS elemental mapping. As an organic material, bitumen is characterised by high carbon and oxygen contents, confirming that the observed surface region is predominantly composed of bitumen. By contrast, only trace amounts of chlorine, sulphur, and other salts were detected. The relatively low calcite content further indicates that bitumen forms a continuous coating around the cement particles, thereby protecting the hydration products from direct environmental

exposure (Jansen et al., 2021). For comparison, Fig. 4.17(a) presents the surface morphology of the unmodified cementitious mixture.

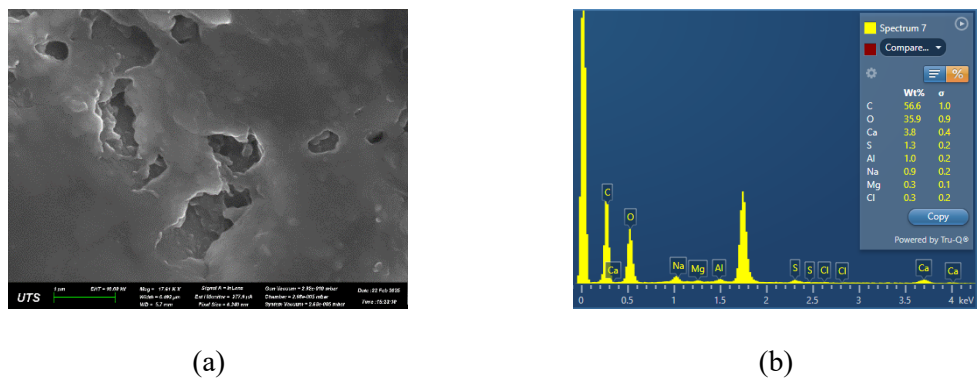


Fig. 4.15 (a) SEM graphic of cationic bitumen in CSS specimen, (b) EDS map scan result

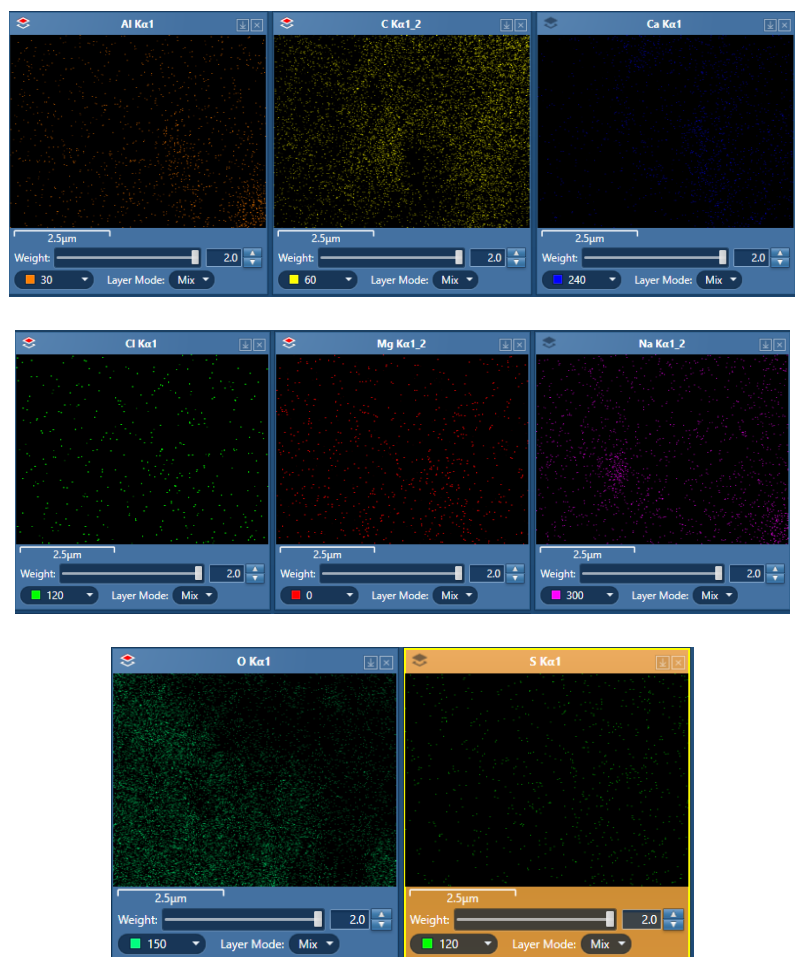
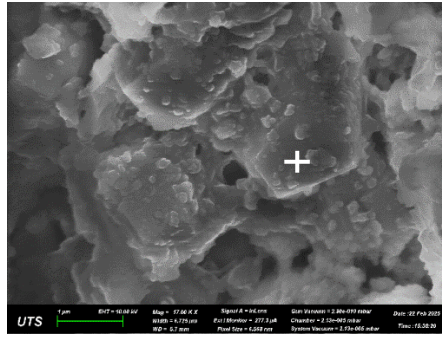
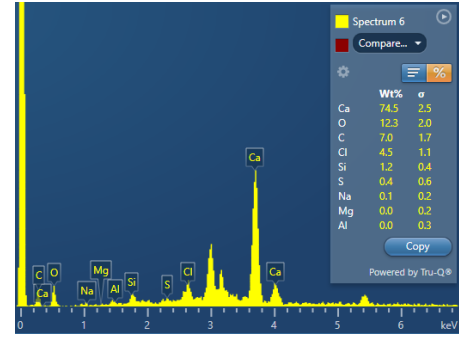


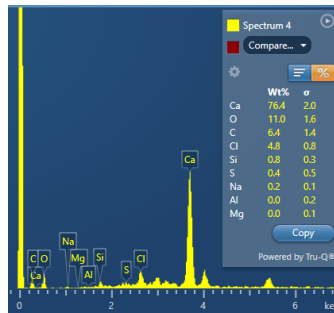
Fig. 4.16 EDS map scan results of bitumen surface for each element



(a)



(b)



(c)

Fig. 4.17 (a) SEM scan results of cementitious hydration products, (b)EDS map scan of (a), (c) EDS point scan result in (a)



Fig. 4.18 EDS map scan of cementitious hydration products for each element

For the CO specimens, EDS mapping was performed to assess the effects of chloride diffusion. As shown in Fig. 4.17(b), the calcium content in the analysed region reached 74.5%, attributable to the accumulation of hydration products in that area (Sargam & Wang, 2021). In contrast, EDS analysis in Fig. 4.18 revealed a chloride concentration of 4.5 wt%, confirming that chloride ions were able to migrate through the cement matrix. By comparison, the EDS data from the bitumen surface (Fig. 4.15) showed only trace chloride levels (approximately 0.3 wt%), indicating that the bitumen layer served as an effective diffusion barrier. Point analyses conducted on the surface of hydration products further validated the elemental mapping results, demonstrating that chloride is a major constituent within the subsurface of cementitious matrices, thus confirming its infiltration into the cement phase. Together, Fig. 4.15(b) and Fig. 4.17(b) illustrate that bitumen forms a continuous coating on the cement surface, impeding chloride transport (Brand & Roesler, 2017). Moreover, the absence of measurable chloride on the bitumen surface corroborates that chloride ions were unable to penetrate the bitumen layer, thereby emphasising the barrier function of the bitumen membrane in restricting chloride ingress.

4.8 Summary

The incorporation of seawater (SW) was found to accelerate the hydration process in self-sensing specimens, thereby enhancing compressive strength after 28 days of curing. SW-based mixtures also generated additional hydration products, such as Friedel's salt, which are indicative of more extensive hydration reactions. Ion conduction played a pivotal role in influencing the percolation thresholds of the self-sensing cementitious sensors. With increasing conductive filler content, a greater number of conductive pathways were established in SW specimens compared with CO specimens, owing to ionic contributions. Consequently, the electrical resistance gap between 0.1 wt% and 1.0 wt% MWCNTs in the 50 SW group was wider than that observed in the CO group. This ionic effect also

steepened the slope of the percolation threshold curves. In specimens with high ion concentrations and reduced pore volumes, such as the 100 SW group, the initial electrical resistance was lower than in the 50 SW group. At the same time, the higher ionic concentration facilitated the early formation of conductive networks, although a balanced ionic concentration, exemplified by the 50 SW specimens, proved more effective in producing composites with greater sensitivity to mechanical stress. The SW influence was shown in Fig. 4. 19.

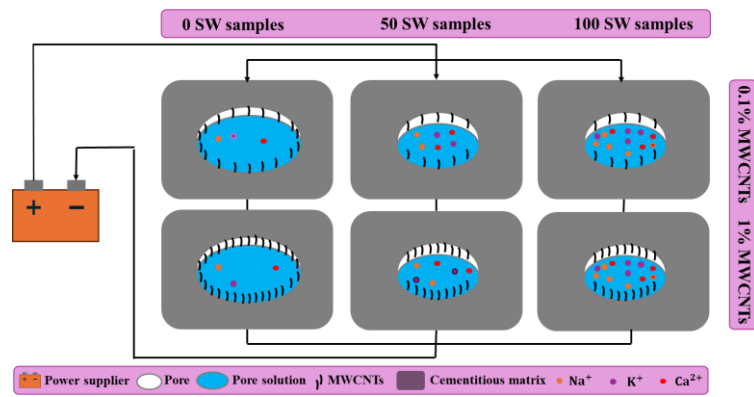


Fig. 4. 19. Explanation of ion conduction influence on percolation thresholds of self-sensing specimens

Results from both the NT Build 492 and RCPT tests demonstrated that the incorporation of bitumen emulsion into cement mixtures substantially enhanced resistance to chloride diffusion. The bitumen emulsion formed a protective barrier against ion ingress without significantly impeding hydration in the early stages, and its influence diminished further after adequate curing. Among the modified specimens, those containing cationic bitumen emulsion (CSS) exhibited superior performance relative to their anionic counterparts (ASS), attributable to stronger electrostatic interactions with negatively charged hydration products such as C–S–H, which resulted in a denser microstructure. Overall, this study establishes that the use of seawater and bitumen emulsion not only improves the self-sensing capability of cementitious composites but also enhances their resistance to chloride penetration. These findings provide a strong foundation for the development

and practical application of cement-bitumen composites as multifunctional pavement materials with improved durability and performance.

Chapter 5: Relationship between strain change and FCR using machine learning algorithm

5.1 Introduction

Asphalt, inherently an insulating material, can have its electrical properties modified through the incorporation of conductive fillers, thereby improving the overall conductivity of asphalt mixtures (Ahmedzade & Sengoz, 2009; Wu et al., 2005). However, material deterioration over time has been shown to weaken the piezoresistive performance of asphalt composites (Liu & Wu, 2009; Liu et al., 2008; Rizvi et al., 2016). The foundation of self-sensing asphalt composites lies in the addition of conductive fillers (Apostolidis et al., 2016; Liu et al., 2014; A. O. Monteiro et al., 2017). Commonly employed fillers include CB, CNTs, and CFs, each of which affects both the mechanical performance and electrical conductivity of asphalt-based composites (Schumacher & Thostenson, 2013; Wu et al., 2005; S. Wu et al., 2013). Incorporation of fibrous fillers can transform asphalt from an insulating to a semi-conductive composite, enabling it to exhibit measurable electrical responses under applied loads (Azhari & Banthia, 2012). Nevertheless, the piezoresistive performance of CF-reinforced asphalt mixtures can be affected by factors such as data noise and sand volume (Allam et al., 2022; Belli et al., 2018). In contrast, CB enhances the electrical conductivity of self-sensing mixtures, and its piezoresistive response under cyclic loading can be further improved when combined with silica fume (W Dong et al., 2020). CNTs have been shown to enhance both piezoresistivity and mechanical properties (Dong, Guo, et al., 2021), with the “sandwich model” of CNTs providing significant improvements in conductivity while exerting only limited effects on mechanical strength (W. Dong et al., 2020). Among the three fillers, CF

emerges as the most effective for asphalt-based self-sensing composites, as it simultaneously improves both mechanical strength and piezoresistive behaviour.

Vehicle speed and weight can be evaluated using this novel approach (Han et al., 2009; Han et al., 2011; Shi & Chung, 1999), enabling optimisation of traffic management through real-time monitoring of traffic conditions. Moreover, the development of weigh-in-motion (WIM) systems can be further advanced using traffic data collected by self-sensing systems (Birgin et al., 2020). When cracking occurs, the electrical resistance of the structure increases, thereby influencing piezoresistivity. The self-sensing response to loading variations can therefore be used to assess pavement damage levels. This capability underpins the application of self-sensing sensors for SHM.

Experimental data from self-sensing sensors exhibit repetitive patterns, making them well-suited for predictive modelling through machine learning (ML), a branch of artificial intelligence. ML models can be developed directly from empirical data without reliance on programming or rule-based algorithms (Mitchell, 1997). Previous studies have demonstrated the potential of ML for infrastructure-related applications, including pipeline and atmospheric corrosion prediction (Ji & Ye, 2023; Zhi et al., 2020), micro-level damage prediction in concrete beams (Zhou et al., 2021), and non-destructive evaluation of fibre-reinforced plastics (Roh et al., 2021).

Machine learning (ML), when trained on empirical input data, reduces dependence on theoretical models and enhances decision-making efficiency. Artificial neural networks (ANNs), for instance, can be trained with multiple datasets comprising inputs and corresponding targets to optimise model outputs and improve predictive accuracy. As an example, the shear contribution of fibre-reinforced plastics in reinforced concrete structures has been successfully analysed using an adaptive neuro-fuzzy inference system (ANFIS) (Naderpour & Alavi, 2017). Moreover, integrating ML can significantly

strengthen the capabilities of SHM systems. However, the combined application of ML models and self-sensing asphalt sensors remains underexplored. The relationship between strain and FCR provides a basis for evaluating the material's sensing performance, as mechanical deformation alters the conductive network within the composite. In this study, machine learning algorithms are employed to model the relationship between strain and FCR, enabling improved prediction of sensor behaviour. Accordingly, the objective of this chapter is to examine the application potential of self-sensing asphalt concrete and to develop an ML-based predictive model for analysing self-sensing data.

5.2 Experimental program

5.2.1 Raw materials and mixture preparation

This study examines the relationship between piezoresistive performance and strain variation in asphalt-based self-sensing sensors. The specimens were prepared at approximately 175 °C, a temperature that minimises the influence of humidity and ion concentration on self-sensing behaviour. Under these conditions, the piezoresistive response is governed primarily by the conductive fillers and internal volume changes within the specimens. Accordingly, asphalt concrete was selected as the material system to investigate the correlation between self-sensing performance and strain behaviour.



(a)

(b)

Fig. 5.1 (a) bitumen binder under normal status and (b) Furnace used to melt the asphalt binder.

In accordance with AS 2008-2013, Class C450 asphalt was employed as the binder in this chapter, with its specifications provided in Table 3.1. Fig. 5.1(a) illustrates the typical appearance of C450 asphalt. River sand was used as the fine aggregate, while 10 mm coarse aggregate was incorporated into the asphalt concrete. Carbon fibres (CFs), with a length of 1 mm and a diameter of 7 μm , were utilised as conductive fillers to establish conductive networks. To evaluate the percolation thresholds of asphalt-based self-sensing sensors, eight CF dosages were prepared: 0.5%, 1.0%, 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, and 4.0%. Fig. 5.1(b) shows the LABEC furnace used to melt the asphalt. To reduce viscosity, the asphalt binder was heated to 175 °C, at which point its viscosity was below 3 Pa·s, consistent with the findings of Wang et al. (Wang et al., 2012). Uniform dispersion of CF within the asphalt mixture is essential for improving the electrical performance of asphalt-based self-sensing sensors (Zhang et al., 2016; Zhang et al., 2017). According to AS 2891, the melting point of asphalt is 105 ± 5 °C. However, to ensure proper mixing, the asphalt binder was heated to 175 °C for two hours. Simultaneously, both the aggregates and CF were preheated at the same temperature. Following this stage, the preheated CF was incorporated into the asphalt binder and mixed for ten minutes to achieve homogeneous dispersion. The CF–asphalt mixture was then reheated at 175 °C for an additional two hours. Subsequently, aggregates were introduced into the mixture, which was further heated in the furnace at 175 °C for another two hours to complete the final heating stage of specimen preparation. The resulting mixture was poured into cylindrical moulds and allowed to cure at room temperature before demoulding after 24 hours. Throughout both mixing and moulding, heating plates were employed to maintain

the required mixture temperature. Table 5.1 presents the eight CF dosages applied in this study, ranging from 0.5% to 4.0% at 0.5% intervals.

Table 5.1 Components of asphalt-based self-sensing specimens.

Materials	Material content							
CF wt%	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
Asphalt binder / g	71.82	71.82	71.82	71.82	71.82	71.82	71.82	71.82
Coarse aggregates / g	594	594	594	594	594	594	594	594
Fine aggregates / g	546	546	546	546	546	546	546	546

CFs exhibit exceptional thermal stability, withstanding temperatures up to 3000 °C in inert environments (Sanoj & Kandasubramanian, 2014). However, graphitisation of CFs occurs when the temperature approaches 2000 °C, while oxidation and degradation can take place within the range of 400–600 °C (Lebedev et al., 2020; Sauder et al., 2004). In this study, the maximum processing temperature was limited to 175 °C. Therefore, the potential influence of elevated temperature on CF integrity can be considered negligible.

5.2.2 Strain change evaluation

The digital image correlation (DIC) method was employed to measure the strain response of the asphalt-based self-sensing (ASS) specimens. To ensure surface uniformity, the asphalt cylinders were ground prior to testing. The prepared surfaces were then spray-painted to create a random speckle pattern of white spots, facilitating accurate strain tracking during the DIC test, as illustrated in Fig. 5.2(a, b), together with the corresponding results. Both the specimens and the camera were positioned at the same horizontal level to minimise shadow interference and ensure full surface coverage of the speckle pattern (Kinda et al., 2023).

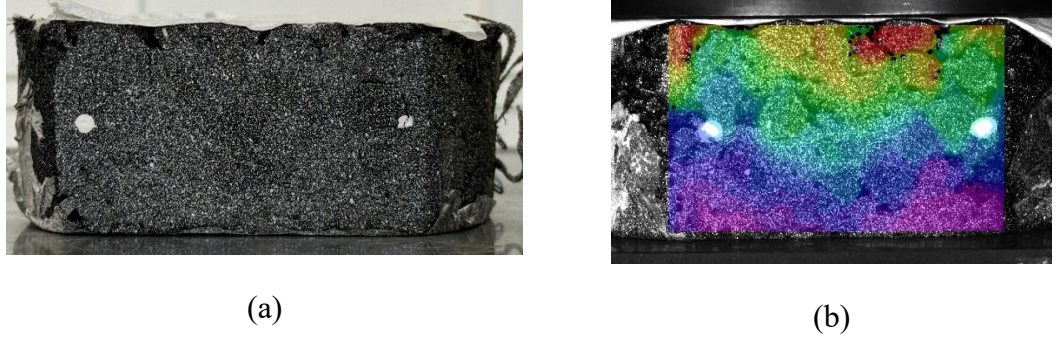


Fig. 5.2 (a)The asphalt-based self-sensing specimen with scatter painting and (b) the DIC test result.

5.2.3 Machine learning modelling and validation

The piezoresistive performance tests provided a sufficiently large dataset to support the development of a machine learning (ML) model for predictive analysis. High-performance computational models not only enhance the efficiency of self-sensing systems but also reduce dependence on expert interpretation (H. Li et al., 2023). Importantly, artificial neural networks (ANNs) can generate reliable outputs even when confronted with previously unseen input data (Roh et al., 2021), making them particularly advantageous for analysing the experimental dataset obtained in this study. In this work, an ANN model was trained using the self-sensing performance results. The selected inputs were the electrical response, expressed as the fractional change in resistance (FCR), and the percentage of conductive filler content, while the output parameter was the strain change of the specimens. The ANN algorithm incorporated a cost function to quantify the difference between the predicted and experimental outputs, with the cost value representing the total error across all training samples. Iterative optimisation was performed to minimise this cost, thereby improving predictive accuracy. The overarching objective was to ensure that the ANN-predicted strain values closely approximated the experimental measurements. Accordingly, the piezoresistivity data and conductive filler content were adopted as the primary input parameters for the ANN algorithm.

The Levenberg-Marquardt algorithm was utilised for ANN training and listed in the following Eqs. (3) and (4) (Roh et al., 2021):

$$H = J^T J \quad (3)$$

$$g = J^T e \quad (4)$$

Eq. (3) is the Levenberg-Marquardt algorithm's second-order training speed without calculating the Hessian matrix. Eq. (4) is the calculation method of the gradient, where J is the Jacobian matrix, and e is the vector of network errors. In the Levenberg-Marquardt algorithm, Eqs. (3) and (4) are used as Newton-like updates.

$$x_{k+1} = x_k - [J^T J + \mu I]^{-1} J^T e \quad (5)$$

Where μ is a scalar. The success training numbers can be increased when the scalar is decreased. Eq. (5) reduces to Newton's method when μ equals zero. Eqs. (3) to (5) were implemented in MATLAB for model development. In this framework, the conductive filler content and fractional change in resistance (FCR) were used as input parameters, while the strain of the asphalt-based self-sensing (ASS) specimens served as the target output. Additional parameters, such as compressive strength, may also be incorporated as input variables in cases where FCR and filler content alone are insufficient for effective algorithm training. The machine learning framework was implemented in MATLAB using a single-layer model architecture, selected to ensure methodological transparency and reduce computational complexity. The dataset used for machine learning analysis was obtained from experimental measurements conducted under cyclic loading conditions. A total of over 10,000 data points were collected, consisting of paired strain and fractional change in resistance (FCR) values. This large dataset provides sufficient coverage of the strain–piezoresistive relationship and enables reliable model training and validation.

5.2.4 Optical microscope

This chapter investigated the micro-holes within asphalt composites. To monitor changes in internal voids, specimens were subjected to compression using an AGX 50 kN testing machine, with the applied compressive stress maintained for ten minutes. Immediately after load release, the specimens were examined using an optical microscope. The optical microscopy setup is shown in Fig. 5.3. A Carl Zeiss Axiotech Vario 100 HD microscope was employed, equipped with an Eakins 48 MP FHD camera for high-resolution imaging.

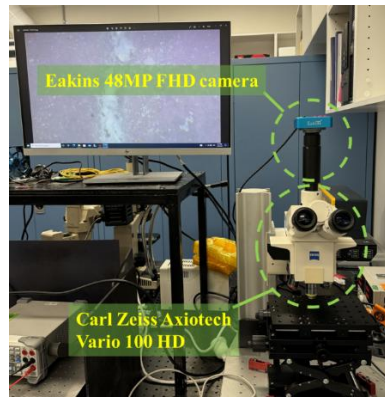


Fig. 5.3 The optical microscopic system

5.3 Percolation thresholds

Previous studies have employed analytical models to investigate the percolation thresholds of asphalt-based self-sensing (ASS) composites (García-Macías et al., 2017). Birgin et al. (Birgin et al., 2022) utilised EVIzero, a sustainable asphalt substitute, combined with carbon fibres (CFs) to fabricate self-sensing sensors, with their percolation thresholds illustrated in Fig. 5.4. In this study, a similar analytical approach was adopted to evaluate the percolation thresholds of ASS specimens, as shown in Fig. 5.5. In both cases, the CF content ratio is represented on the horizontal axis, while the electrical resistance of the specimens is plotted on the vertical axis.

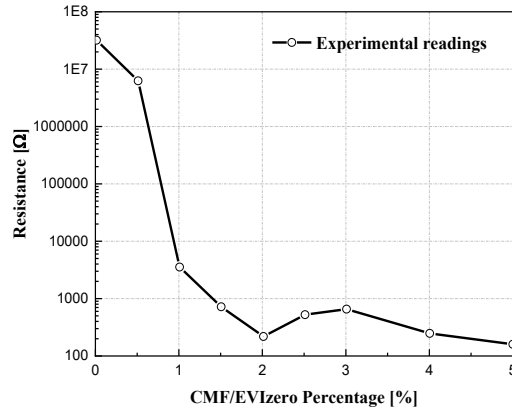


Fig. 5.4 Percolation thresholds of EVIzero-based self-sensing sensors (Birgin et al., 2022).

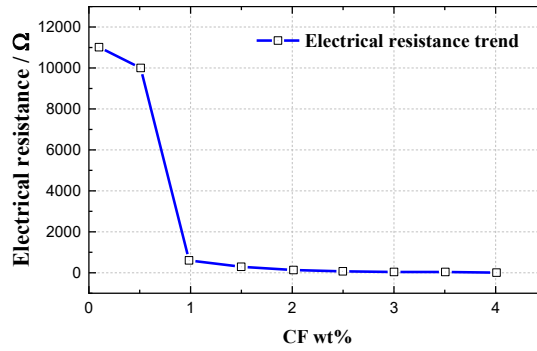


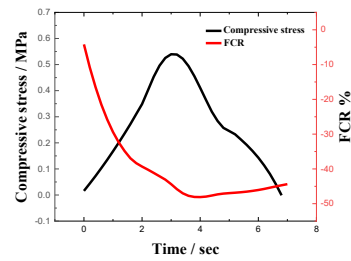
Fig. 5.5 Percolation thresholds of self-sensing asphalt-based sensors.

According to the analytical model proposed by (Kumar & Rawal, 2016). The critical volumetric fraction is 1.0%. Experimental findings further validated the percolation thresholds of EVIzero-based self-sensing sensors, with 1.0% CF content identified as the most effective doping level (Birgin et al., 2022). In the present study, the experimental results indicated an upper percolation threshold of 0.5% and a lower threshold of 1.0%. These findings demonstrate consistency with those of Birgin et al (Birgin et al., 2022), suggesting comparable percolation behaviour. Accordingly, a CF content of 0.5–1.0 wt% can be regarded as the effective operational range for asphalt-based self-sensing (ASS) specimens.

5.4 Piezoresistivity

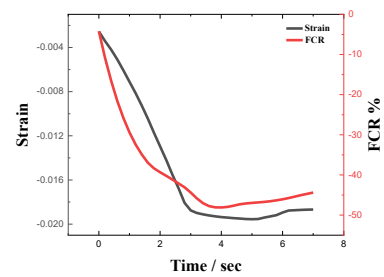
Fig. 5.6 illustrates the relationship between fractional change in resistance (FCR) and the compressive stress–strain response of the asphalt-based self-sensing (ASS) specimens. During the single loading process, the FCR initially decreased continuously. When the dynamic load reached its peak value, however, the FCR of the ASS specimens attained its maximum response.

Compressive stress-FCR

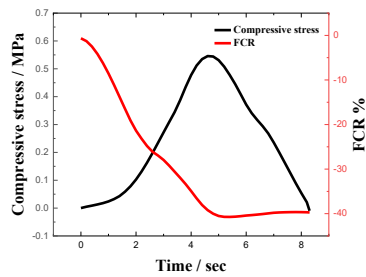


0.5% CFs

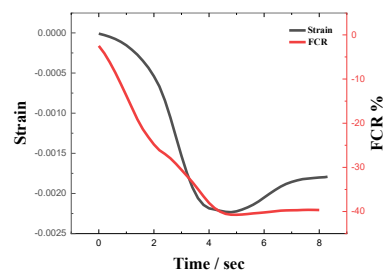
Strain-FCR



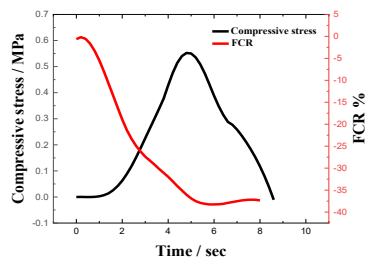
0.5% CFs



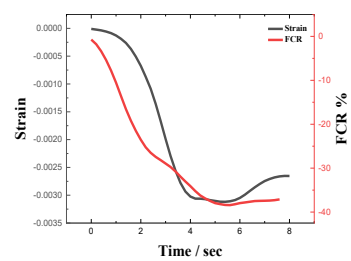
0.7% CFs



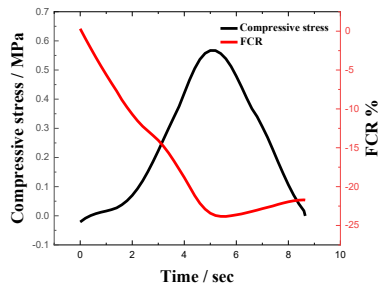
0.7% CFs



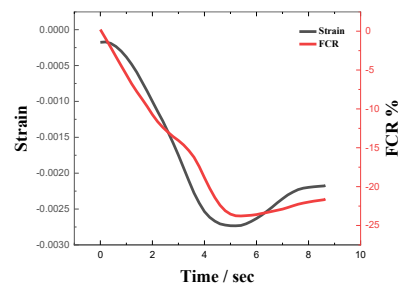
0.8% CFs



0.8% CFs



1.0% CFs

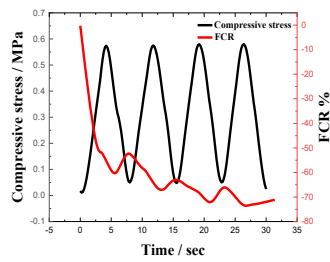


1.0% CFs

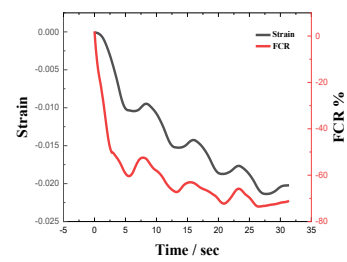
Fig. 5.6 Relationship of Fractional change in resistance (FCR) with compressive stress and strain in single load test.

Compressive stress-FCR

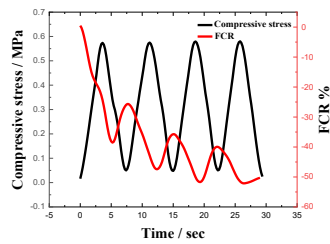
Strain-FCR



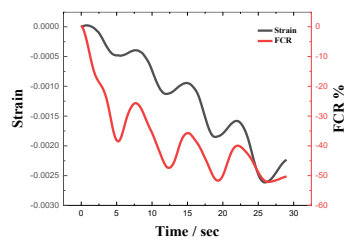
0.5% CFs



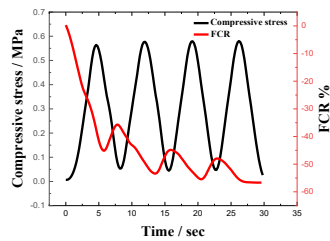
0.5% CFs



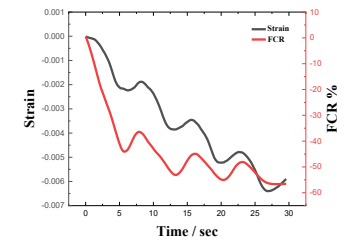
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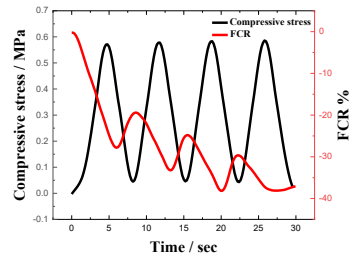
0.7% CFs



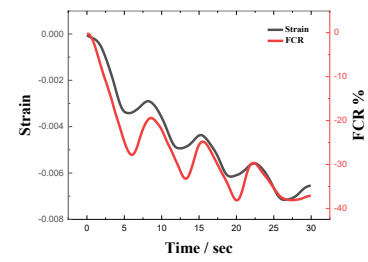
0.8% CFs



0.8% CFs



1.0% CFs



1.0% CFs

Fig. 5.7 Relationship of Fractional change in resistance (FCR) with compressive stress and strain in cyclic load test.

The strain–electrical results revealed a stronger correlation between self-sensing performance and strain variations in the ASS specimens than between stress and electrical response. During cyclic loading, strain fluctuations directly influenced the electrical signal. The initial FCR decreased progressively with each loading cycle, and the overall FCR trend continued to decline throughout the entire process. This behaviour differed from that observed in cement-based self-sensing specimens (Lee et al., 2022). As shown in Fig. 5.6 and Fig. 5.7, the correlation between strain change and electrical response strengthened as the applied stress increased. Within a single loading cycle, the FCR rose with increasing strain; however, upon unloading, the strain did not return to its initial state, indicating a temporary deformation of the material. In this process, the FCR initially increased and then stabilised. Comparable observations were reported in previous research on asphalt mixtures incorporating electric arc furnace slag and graphene nanoplatelets as conductive fillers, where a similar methodology was employed to assess piezoresistive behaviour (Gulisano et al., 2022b). A related phenomenon was also described by (Li et al., 2024). Nevertheless, unlike these studies, the present results emphasise the necessity of accounting for strain variations when interpreting the electrical response of asphalt-based self-sensing composites. Without this consideration, the

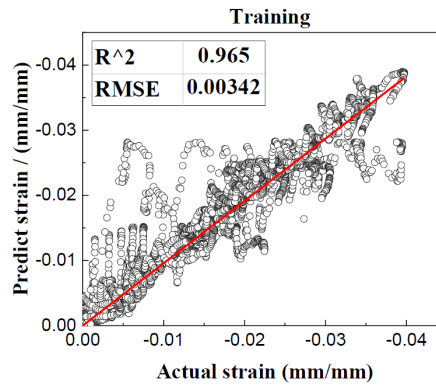
analysis remains incomplete. Due to the delayed strain recovery, the peak FCR lagged behind the peak compressive stress.

Future studies should therefore prioritise detailed examination of strain–electrical coupling in asphalt-based self-sensing specimens. Overall, the ASS samples exhibited high sensitivity to loading changes, underscoring their strong potential for applications in TD and SHM.

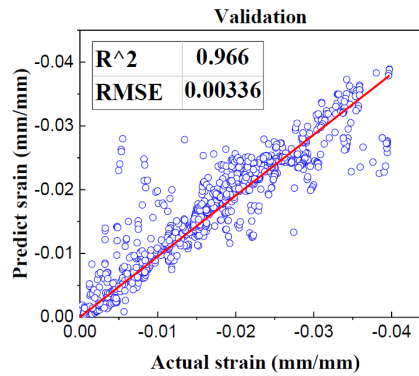
5.5 Predicted response from trained ML model

Fig. 5.8 presents scatter plots illustrating the relationship between the actual and predicted results. The root mean squared errors (RMSEs) for the training and validation datasets were 0.00342 and 0.00336, respectively. The corresponding coefficients of determination (R^2) were 0.965 and 0.966, indicating an excellent fit between the measured and predicted strain values. These results confirm that the machine learning (ML) model trained with the experimental dataset demonstrates high predictive reliability. Compared with previous work, in which the applied algorithm achieved a lower R^2 value of approximately 0.93, the present model exhibits superior performance. To further evaluate the reliability of the MATLAB-based approach, an independent dataset was tested using a separate set of ASS specimens with controlled CF contents of 0.5%, 0.7%, 0.8%, and 1.0%, as illustrated in Fig. 5.5. The corresponding test results are presented in Fig. 5.9. A strong agreement was observed between the measured and predicted strain values, with a coefficient of determination (R^2) of 0.963. These findings demonstrate that the predictive accuracy of the algorithm is closely linked to the CF content, even though the primary focus of this study was on the optimal range of ASS specimens. Overall, the evaluation of this supplementary dataset validates that the ML approach provides a robust and reliable method for detecting strain variations in asphalt composites by monitoring changes in electrical resistance.

The ML algorithm demonstrated substantial potential for enhancing TD efficiency, as evidenced by the experimental results. A previous study also developed a learning algorithm for TD applications (García-Macías et al., 2017). Compared with traditional approaches, the ML model simplifies the otherwise complex data analysis process while simultaneously improving result reliability. Once trained with practical datasets, the artificial neural network (ANN) can directly generate predictive algorithms. Consequently, when new input data are introduced, the algorithm adapts automatically, thereby increasing the operational efficiency of the self-sensing system. In contrast to earlier algorithms, the ML approach employed in this study offers a more streamlined and effective framework for improving TD performance (García-Macías et al., 2017).



(a)



(b)

Fig. 5.8 Plot graphic of (a) training and (b) validation results of strain for the whole dataset.

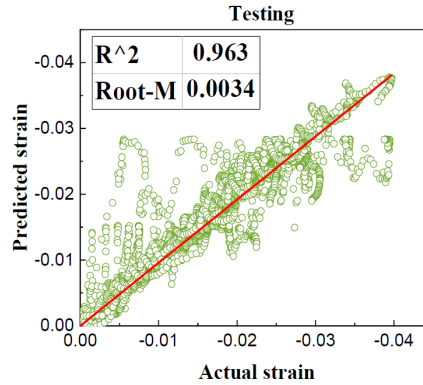


Fig. 5.9 The reliability testing of trained algorithm test results by extra datasets.

5.6 Summary

This study investigated the performance of self-sensing asphalt sensors and analysed the percolation thresholds for CF concentration using the four-electrode method, digital image correlation (DIC), and artificial neural network (ANN) modelling. The optimal CF content range for effective sensing was identified as 0.5–1.0 wt%. A machine learning model trained on the experimental dataset achieved an R^2 value exceeding 0.96, highlighting the strong potential of ML for accurately predicting strain based on self-sensing data. Furthermore, the findings demonstrated a direct correlation between strain variation and piezoresistive behaviour. Fig. 5.10 is summarised to illustrate this relationship between FCR and strain change. During the load-releasing process, the electrical resistance of the self-sensing cementitious matrix can recover to its original value following the loading and unloading process. However, the ASS samples cannot revert to their original form as efficiently as cement-based sensors. Because of that, the conductive path in ASS that formed during the loading process will be partly kept in the micro-holes until the next loading. That's the reason why ASS specimens were chosen in this study.

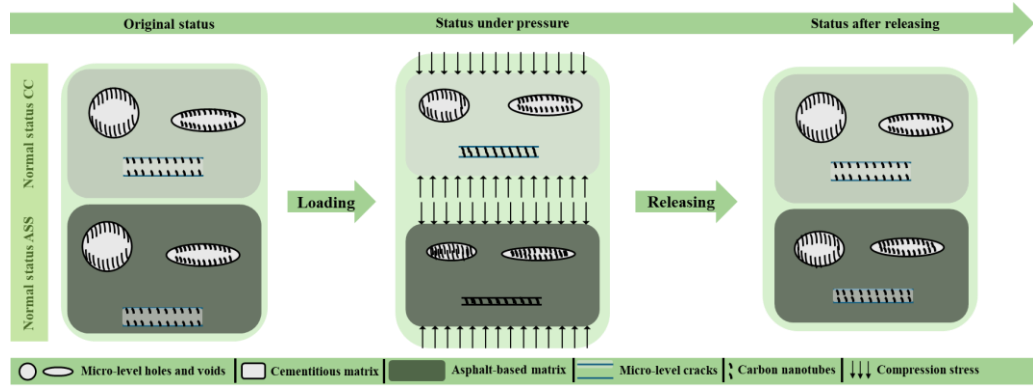


Fig. 5.10. Comparison of micro holes and micro-cracks change under compressive stress between cement-based samples and ASS.

This relationship can be attributed to the influence of strain on internal volume changes, which facilitates the formation of new conductive pathways. Consequently, the shape and volume of pores and cracks in the ASS specimens are altered during loading. To provide deeper insight into this mechanism, Chapter 6 presents a detailed analysis of the influence of internal structural characteristics on the piezoresistivity of ASS specimens.

Chapter 6: Influence of pore structure on self-sensing sensors

6.1 Introduction

Section 5.1 provided a detailed introduction to asphalt-based self-sensing sensors. The rationale for selecting asphalt-based rather than cement-based sensors in this study lies in their high melting temperature, which minimises the influence of humidity and ion concentration (as discussed in earlier sections) on self-sensing performance. Consequently, the primary factors affecting piezoresistive behaviour in asphalt-based systems are the content of conductive fillers and internal volume changes associated with strain. In this study, the piezoresistive properties of self-sensing asphalt sensors were systematically examined. Cyclic compressive stress was applied to evaluate the electrical response of the specimens, thereby enabling an assessment of their potential application in traffic detection operations. In addition, micro-computed tomography (Micro-CT) scanning was employed to analyse the impact of pore structure on piezoresistivity. As CF were selected as the conductive filler in this section, the analysis focused specifically on the role of micro-scale pores.

6.2 Experimental program

6.2.1 Raw materials

C450-grade bitumen was selected for this study as it is widely used in pavement applications and satisfies the performance requirements specified in AS 2008. The investigation focuses on the influence of pore structure on the piezoresistive behaviour of asphalt-based self-sensing (ASS) sensors by incorporating aggregates of three nominal sizes, 4.75 mm, 9.5 mm, and 13.2 mm, in accordance with AS 2150. CF were employed as the conductive filler, with a content of 1.0% by weight chosen based on prior research (Deng et al., 2025), which demonstrated that this dosage optimises electrical conductivity

and provides stable, precise piezoresistive performance (see Fig. 5.7). The experimental specimens were categorised into three groups, with their mix compositions detailed in Table 6.1. Although CF is inherently conductive, the bitumen binder functions as an electrical insulator and hinders the development of continuous conductive pathways. Similarly, the aggregates act as barriers that further restrict the connectivity of the conductive network. As a result, the electrical response of the self-sensing asphalt specimens must be evaluated under mechanical loading, where changes in the internal pore structure govern the continuity and effectiveness of the conductive network.

Table 6.1 Components of asphalt-based self-sensing sensors

Coarse aggregate sizes (mm)	Asphalt Binder (g)	Coarse aggregates (g)	Fine aggregates (g)	Carbon fibre (g)
4.75	75	625	575	0.75
9.5	75	625	575	0.75
13.2	75	625	575	0.75

6.2.2 CT scan and data analysis

HeliScan X-ray computed tomography (CT) was employed to investigate the internal matrix of the ASS specimens. The system was equipped with a GE Phoenix NanoFocus X-ray tube (180 kV/15 W) and a flatbed detector offering a pixel resolution of 3072×3072 . Two specimen geometries were examined: cylindrical samples (100 mm in diameter and 50 mm in height) and cubic samples ($50 \times 50 \times 50 \text{ mm}^3$). Because materials of differing densities absorb X-rays at different rates, CT imaging enabled the visualisation of the internal structure of the self-sensing asphalt composites. The Avizo software package, capable of both three-dimensional reconstruction and quantitative

analysis, was used to model and characterise the pore structure and spatial distribution within the ASS specimens, thereby providing detailed insight into their internal geometry. To characterise pore features, X-ray CT scanning was performed by capturing a sequence of two-dimensional slice images throughout the specimen volume. As illustrated in Fig. 6.1(a), the lower density of pores relative to aggregates and asphalt binder resulted in markedly reduced grey values, which facilitated their identification. An interactive thresholding technique was applied to distinguish pore regions from the solid matrix, with pore locations highlighted in blue in each slice, as shown in Fig. 6.1(b). This approach effectively captured both internal and surface-connected pores, allowing for accurate segmentation of void space. Subsequently, Avizo was used to reconstruct a three-dimensional pore network model, enabling precise visualisation and quantitative assessment of the internal pore structure in the ASS specimens.

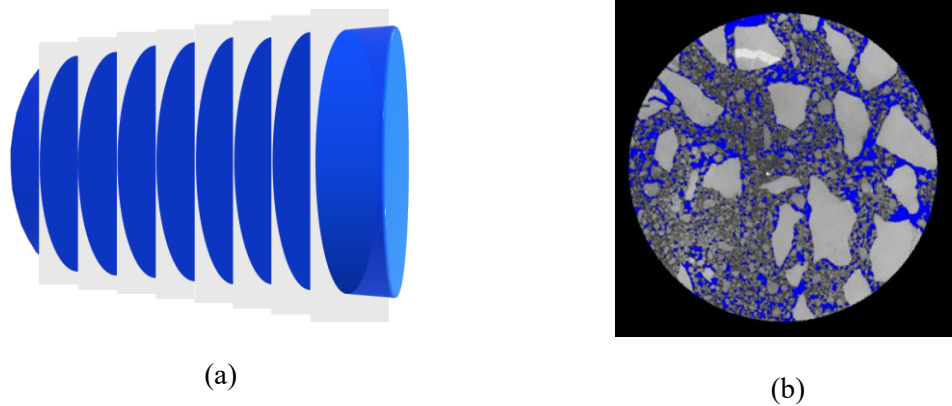


Fig. 6.1 (a) shows the CT-scan method, (b) shows the slice graphic after CT-scan

6.2.3 3D pore structure model

Prior to modelling the pore structure, the full CT-scanned volume of the ASS specimens was cropped to a standard cylindrical region. This preprocessing step eliminated the influence of edge voids and surface irregularities typically caused by coarse aggregates, thereby ensuring that the analysis reflected the intrinsic pore structure. The cylindrical model is illustrated in Fig. 6.2. Quantitative analysis of the pore network was then

undertaken to characterise its connectivity. For three-dimensional pore reconstruction, a sphere-filling algorithm was employed, which enabled precise representation of pore size, spatial distribution, and interconnectivity within the cropped volume. This methodology provided critical insight into the internal microstructure of the ASS composites, offering a robust basis for subsequent correlation with piezoresistive performance (Dong & Blunt, 2009).

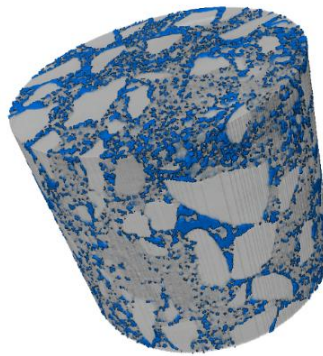


Fig. 6.2 The cylinder model of the asphalt specimen after CT-scan

6.3 Piezoresistivity

The three specimen groups, prepared with aggregate sizes of 4.75 mm, 9.5 mm, and 13.2 mm, exhibited distinct self-sensing behaviours under cyclic compressive loading. As demonstrated in this study, all specimens generated measurable electrical responses to applied cyclic loads, thereby confirming the effectiveness of CF in enhancing piezoresistivity. Aggregate size was found to exert a pronounced influence on the performance of the composites as piezoresistive materials. Variations in aggregate dimensions altered the internal conductive network and the distribution of stress within the matrix, which in turn affected the electrical response under loading. A comparative analysis of the three groups' piezoresistive performance is presented in Fig. 6.3.

Stress - FCR

Strain - FCR

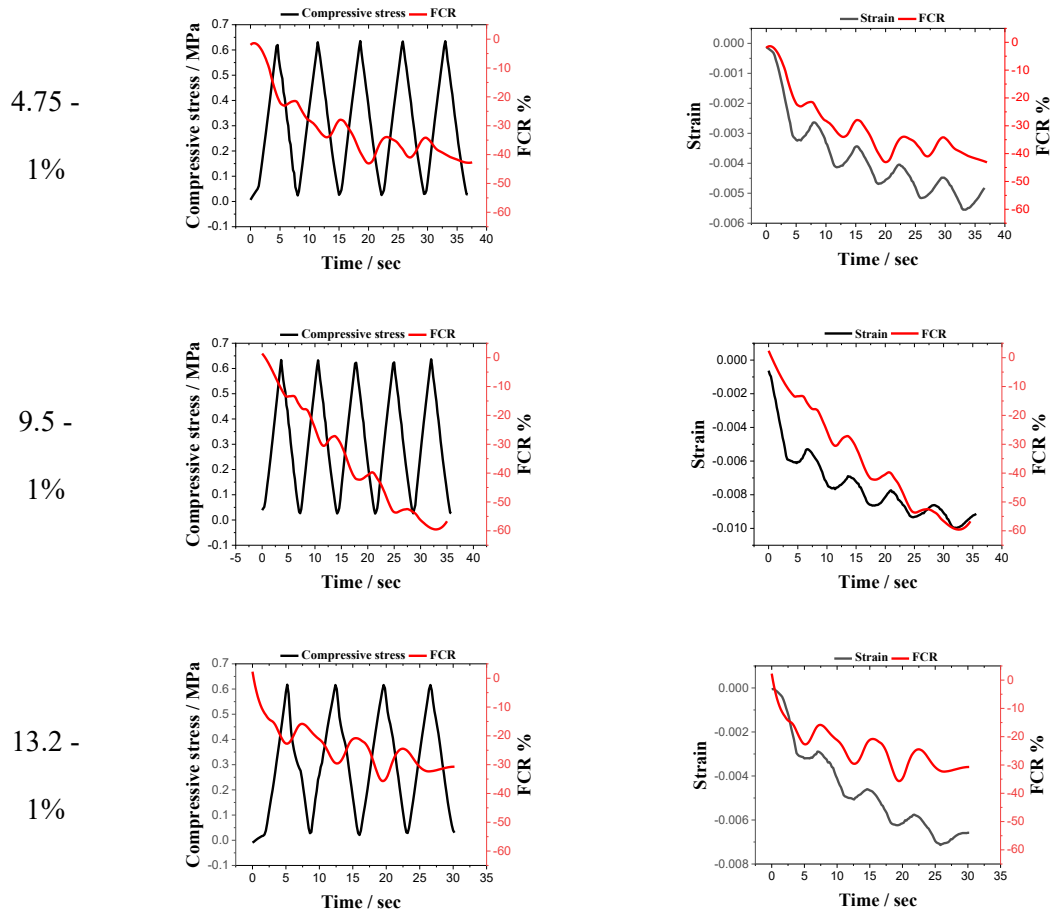


Fig. 6.3 Piezoresistivity test of ASS with different-sized coarse aggregates

Among the three specimen groups, the 9.5 mm ASS samples exhibited the highest FCR values, reaching a maximum of 60%. By contrast, the 4.75 mm composites achieved an FCR of 43.2%, while the 13.2 mm group demonstrated the lowest response at 28.2%. Under identical cyclic compressive loading conditions, the 9.5 mm specimens proved to be the most sensitive to stress variations, highlighting the strong influence of aggregate size on sensing efficiency. Furthermore, the FCR trends observed across all samples closely paralleled the simulated strain profiles, confirming a robust correlation between mechanical deformation and electrical response in asphalt-based self-sensing composites. These findings are consistent with earlier work (P. Li et al., 2018), which emphasised viscoelasticity as a defining nonlinear property of asphalt composites. When subjected to cyclic compressive loads, the experimentally measured strain behaviour mirrored the

simulated strain response. Due to their viscoelastic nature, the ASS specimens did not fully recover their original shape after unloading, and the internal pore structure failed to return entirely to its initial configuration. This delayed recovery directly affected electrical conductivity. During loading, the embedded carbon fibres (CFs) were displaced within the matrix, creating new conductive pathways through inter-fibre contact. Owing to viscoelastic deformation and residual strain, some of these conductive channels persisted after unloading, leaving the specimens' electrical resistance temporarily lower than its original value (Deng et al., 2023). This phenomenon effectively simulated the FCR response, reflecting the dynamic interplay between microstructural rearrangement and changes in electrical resistance. Because the ASS specimens were fabricated at elevated temperatures, ion conduction contributed minimally to their sensing behaviour. Instead, electron conduction played the dominant role in governing piezoresistivity. Following deformation, changes in pore volume facilitated the reorganisation of conductive fillers, thereby promoting the formation of new conductive paths and enhancing electron transport through the composite. Taken together, these results demonstrate that the piezoresistive behaviour of ASS specimens is primarily dictated by pore size, pore connectivity, and microstructural evolution under stress.

6.4 Pores characteristics

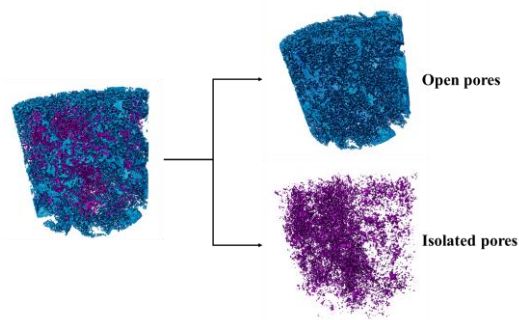
6.4.1 Porosity

The number and volume of pores are widely recognised as key indicators of the porosity of asphalt-based self-sensing (ASS) specimens. In this study, X-ray CT scanning was employed to quantify both the total porosity and the average pore volume across the different aggregate gradations. The results revealed that specimens prepared with 13.2 mm aggregates exhibited the highest porosity, reaching 6.4%. This value is consistent with the findings of (Ren et al., 2023), who reported porosities ranging from 5.07% to

7.44% in asphalt samples incorporating aggregates of similar size. In contrast, porosity decreased to 3.9% in specimens containing 9.5 mm aggregates and further declined to 3.5% in those with 4.75 mm aggregates. Analysis of pore size distribution showed that pores smaller than 1 mm³ accounted for approximately 99% of the total pore count in all three specimen groups. Notably, the 9.5 mm ASS specimens contained the highest proportion of ultra-fine pores, with 97% of the pores measuring less than 0.1 mm³. This high percentage is attributed to the optimal aggregate gradation, which provides a balanced combination of fine and coarse particles, thereby fostering the formation of smaller and more uniformly distributed pores within the asphalt matrix.

6.4.2 Pore size distribution

The pore structures of the three groups of ASS specimens with different aggregate sizes (4.75 mm, 9.5 mm, and 13.2 mm) were examined through quantitative analysis and reconstructed three-dimensional models, as illustrated in Fig. 6.4. In these visualisations, open pores are highlighted in blue, while closed (isolated) pores are shown in purple. The models reveal that the majority of open pores were concentrated along the specimen surfaces, whereas most isolated pores were distributed deeper within the specimen core. Among the three groups, the 9.5 mm ASS specimens exhibited the lowest total pore count, confirming that their internal structure was denser and more compact compared with the 4.75 mm and 13.2 mm counterparts.



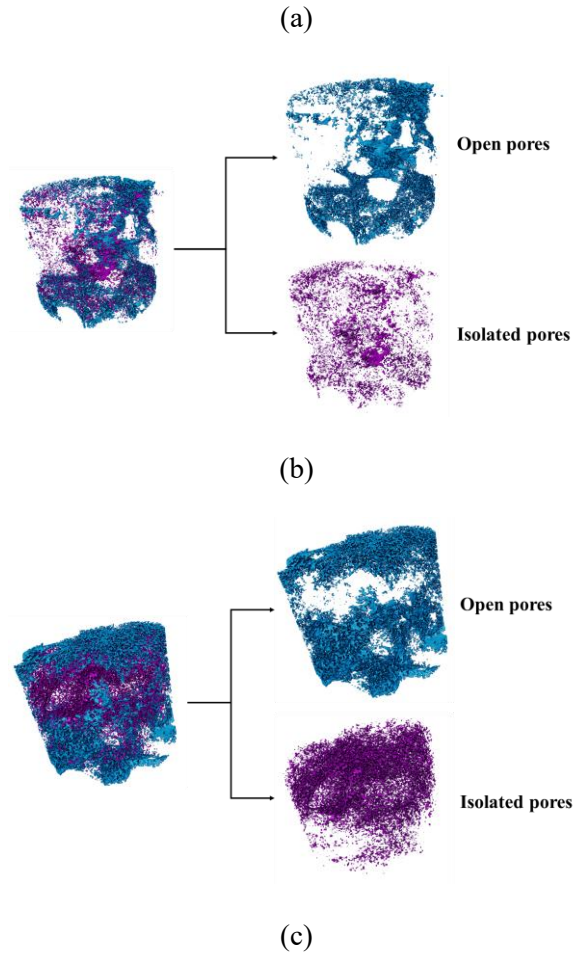


Fig. 6.4 Open pore and Isolated pores in different ASS specimens: (a) 4.75 ASS; (b) 9.5 ASS; (c) 13.2 ASS.

To clearly differentiate the pore structures among the three types of specimens, the quantities of open and isolated pores are summarised in Table 6.2. Across all groups, the number of isolated pores was substantially greater than that of open pores, a trend consistent with previous studies and thereby reinforcing the reliability of CT-based pore classification in asphalt composites (Ren et al., 2023). Analysis of isolated pores with volumes between 0.001 and 0.5 mm³ revealed that these voids did not merge or coalesce into larger cavities. Instead, as corroborated by earlier research, isolated pores predominantly underwent volumetric reduction rather than structural integration with adjacent pores (Ren et al., 2023). Among the three groups, the 9.5 mm ASS specimens exhibited the highest concentration of pores within the smallest volume category (0.001–

0.005 mm³), with 748 open pores and 16,692 isolated pores recorded. By contrast, the 13.2 mm specimens contained the greatest proportion of pores in the larger volume range of 0.005–50 mm³. The distributions of open and isolated pores across different volume ranges are depicted in Fig. 6.5. Apart from the smallest pore category, the overall proportion of pores in the 9.5 mm specimens was consistently lower than that of the other two groups. Notably, the 4.75 mm and 13.2 mm specimens exhibited broadly comparable pore distributions across most ranges. These findings confirm that the 9.5 mm ASS specimens concentrated the majority of their pores in the 0.001–0.005 mm³ range, thereby demonstrating a finer and denser pore structure relative to the 4.75 mm and 13.2 mm counterparts.

Table 6.2 The number of different-sized pores in three types of specimens								
Total amount of pores in different volume ranges		0.001-0.005	0.005-0.01	0.01-0.05	0.05-0.1	0.1-0.5	0.5-1	1-50
4.75 ASS	Open pores	699	331	531	134	166	27	33
	Close pores	9653	4095	6239	1252	1139	115	39
9.5 ASS	Open pores	748	218	411	111	140	28	16
	Close pores	16692	2953	4106	613	433	29	12
13.2 ASS	Open pores	710	356	627	172	217	39	42
	Close pores	10619	4992	8055	1862	1803	174	100

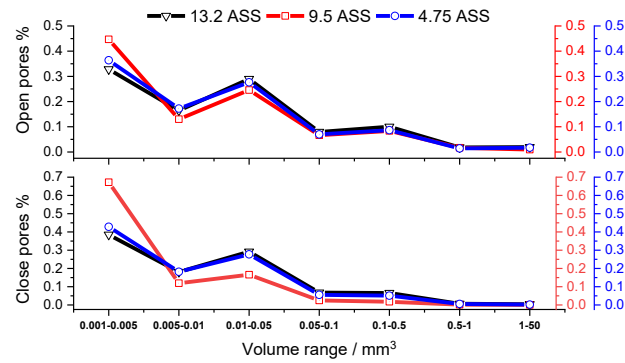


Fig. 6.5 Volume distribution of open and isolated pores

6.4.3 Pore volume

Fig. 6.6 illustrates the distribution of isolated pore volumes across seven predefined ranges for the three ASS specimen groups. The 9.5 mm specimens exhibited the highest cumulative isolated pore volume within the smallest category (0.001–0.005 mm³), reaching 39.56 mm³. However, as the pore size increased, the 13.2 mm specimens showed a more rapid accumulation of pore volume. In the 0.1–0.5 mm³ range, they recorded the highest value at 362.05 mm³. This was followed by a reduction to 120.39 mm³ in the 0.5–1.0 mm³ range, before rising again to 250.43 mm³ in the largest category. By comparison, although the 4.75 mm specimens contained smaller aggregates than the 9.5 mm group, their total isolated pore volume was consistently higher, reaching 331.44 mm³ overall. Notably, the 4.75 mm specimens exceeded the 9.5 mm group in every volume range except the smallest (0.001–0.005 mm³), where the 9.5 mm specimens remained dominant.

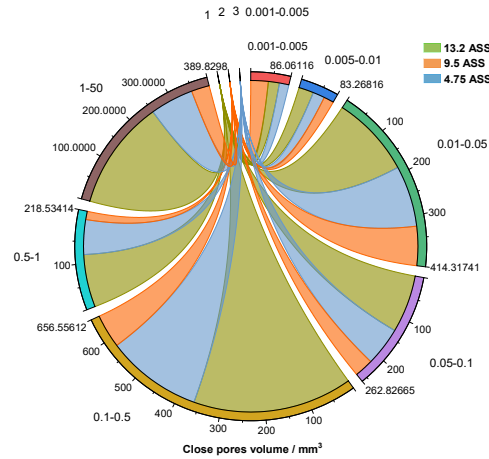


Fig. 6.6 The isolated pore volume distribution in three types of specimens

Fig. 6.7 presents the distribution of open pore volumes across different size ranges. In this study, open pores are defined as voids connected to the external surface of the specimens. When evaluating the piezoresistive behaviour of ASS composites, both isolated and open pores must be considered, as each contributes to the disruption and reconfiguration of the conductive network under applied stress. As clarified in Fig. 6.4, open pores differ from isolated pores by their connectivity to the specimen boundary. The results show that pores within the 1–50 mm³ range accounted for approximately 58% of the total open pore volume, with the 9.5 mm ASS specimens exhibiting the lowest capacity in this category. Conversely, similar to the trends observed for isolated pores, the smallest volume range (0.001–0.005 mm³) dominated the distribution of open pores across all specimen types. Among the three groups, the 13.2 mm ASS specimens consistently displayed the largest total open pore volume, confirming their relatively more porous internal structure.

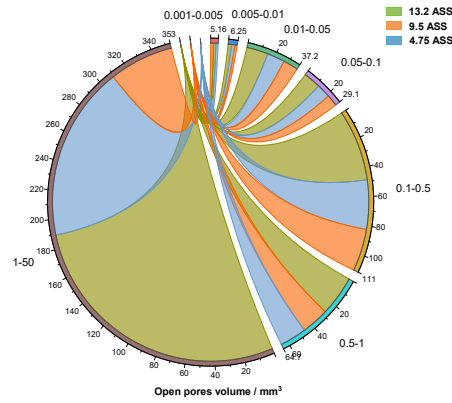


Fig. 6.7 The open pore volume distribution in three types of specimens

6.4.4 Pore equivalent diameter

This study investigates the equivalent diameter (EqDiameter) of both open and isolated pores in the ASS specimens, as it represents a key descriptor of pore geometry and provides a robust metric for analysing pore size distribution. The following equation shows the calculation method of Eqdiameter:

$$d_{3D} = \sqrt[3]{\frac{6V_{3D}}{\pi}} \quad (1)$$

Where V_{3D} : the volume of pores. The experimental results are shown in Fig. 6.8 and Fig. 6.9. For isolated pores, the experimental results demonstrated that the majority fell within an equivalent diameter (EqDiameter) range of 0.1–0.2 mm across all specimen groups. The 9.5 mm ASS group exhibited the highest concentration within this interval, with 14,594 pores identified. This distribution is consistent with the pore volume data, confirming that a substantial proportion of pores in ASS specimens are microscale. A secondary peak in pore frequency was observed in the 0.2–0.3 mm range, after which the number of pores declined steadily with increasing EqDiameter. The maximum recorded EqDiameter was between 3 and 4 mm; however, pores of this magnitude were infrequent. As previously noted, the total pore volume decreased progressively as pore size increased,

with pores exceeding 0.5 mm in EqDiameter contributing less than 10% of the overall pore population in all groups. In this study, pores with EqDiameters greater than 5 mm were classified as large-diameter pores. Open pores were more likely to fall within this category, comprising 10–20% of the pore population, depending on aggregate size. Notably, the 9.5 mm ASS group contained the lowest proportion of large EqDiameter pores, irrespective of whether they were open or isolated.

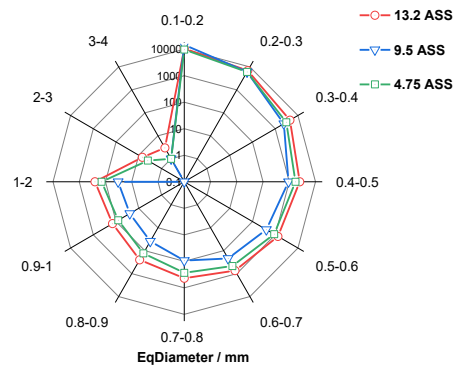


Fig. 6.8 The Isolated pores' Eqdiameter distribution in three different groups of specimens

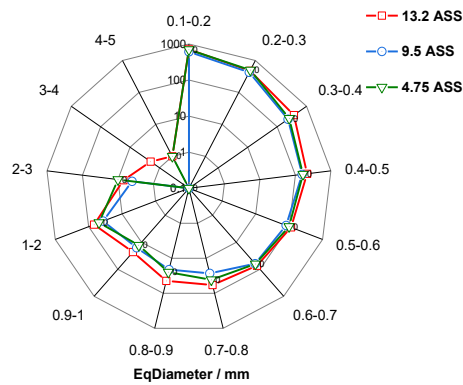


Fig. 6.9 The open pores' Eqdiameter distribution in three different groups of specimens

6.5 Summary

This research integrated macro-scale self-sensing assessments with micro-CT pore structure analysis to examine the correlation between piezoresistivity and internal pore

characteristics of asphalt-based self-sensing (ASS) composites. The experimental findings revealed that specimens with 9.5 mm coarse aggregates exhibited the highest fractional change in resistance (FCR), while those prepared with 13.2 mm aggregates demonstrated more stable piezoresistive responses than the other two groups. CT analysis further confirmed that pore structures varied significantly with aggregate size. Specifically, reducing the prevalence of large pores and increasing the proportion of finely distributed small pores substantially enhanced the FCR performance of ASS composites. This chapter highlights the critical role of internal pore structure in governing piezoresistive behaviour and recommends that future studies explicitly account for pore morphology when evaluating the performance of asphalt-based self-sensing sensors.

Chapter 7: Multifunctional cementitious composites:

Integration of self-healing and self-sensing

7.1 Introduction

To achieve multifunctional composites, this section summarises the key findings from the analysis of systems that integrate both self-sensing and self-healing capabilities, with particular consideration of the influence of ion concentration on piezoresistive performance. In this context, SW-mixed cementitious composites were examined. Owing to the growing demand for reticulated drinking water in concrete production, SW has attracted increasing attention as a potential alternative. Prior research has demonstrated that SW accelerates cement hydration and enhances the early-age mechanical properties of cementitious materials (Younis et al., 2018). Furthermore, SW promotes autogenous healing, as the dissolved CO_2 and ionic species facilitate the precipitation of calcium carbonate, a key compound contributing to crack closure and strength recovery in cementitious composites (Jacobsen & Sellevold, 1996). This self-healing process may thus be regarded as an extension of cement hydration. Elevated concentrations of Mg^{2+} , Ca^{2+} , and OH^- , ions in SW further accelerate hydration reactions, facilitating the formation of secondary hydration products. According to Wang et al. (2023), SW enhances the generation of healing compounds with improved structural integrity and hardness, particularly when calcium aluminate (CA) is employed as a self-healing agent. (Liu et al., 2019) observed that the crack-healing rate in marine environments is significantly higher than that in tap water. In addition, SW contributes to the densification of the pore structure, thereby increasing the electrical resistance of cementitious mortars (Buenfeld et al., 1986). (Deng et al., 2024) highlighted the critical role of ion conduction

in governing piezoresistive behaviour, underscoring the application potential of SW for the design of multifunctional cementitious composites.

This study seeks to advance the development of multifunctional cementitious composites by integrating self-sensing and self-healing capabilities within SW systems, enabling real-time monitoring of structural health alongside autonomous crack repair. To the best of the authors' knowledge, this represents the first systematic investigation into the use of SW as mixing water in a multifunctional framework specifically tailored for pavement applications. The research design incorporates comprehensive self-sensing evaluations and wet–dry cycle assessments, supported by a suite of advanced characterisation techniques, including X-ray diffraction (XRD), thermogravimetric analysis (TGA), AC impedance spectroscopy (ACIS), inductively coupled plasma (ICP) analysis, scanning electron microscopy (SEM), and energy-dispersive X-ray spectrometry (EDS).

7.2 Experimental program

7.2.1 Raw materials

General Purpose (GP) cement conforming to AS 3972 was employed as the binder, with a fixed water-to-cement ratio of 0.40. Multi-walled carbon nanotubes (MWCNTs) were incorporated as conductive fillers at dosages of 0.1 wt% and 0.3 wt% of cement mass. Crystalline admixture (CA) was introduced at a constant dosage of 1.0 wt%, as identified by Lin et al. (2023) as the optimum level for achieving significant self-healing performance. To evaluate the influence of seawater (SW) on the self-sensing and self-healing functionalities, three types of mixing water were adopted: 100% SW, 50% SW, and 0% SW (control). Deionised water was used to minimise the influence of background ionic content in the pore solution. The mixed proportions of all cementitious composites are detailed in Table 7.1. Mortar specimens were prepared for self-sensing tests, whereas

cement paste samples were used for self-healing evaluation. In certain cases, samples were designed to serve dual testing purposes to maximise experimental efficiency.

Table 7.1 Details of mix proportions (mass ratio compared to cement)

Test	Specimens	Cement ratio	Sand (S/C)	Deionised Water (W/C)	SW (SW/C)
Self-sensing test	0% SW Multifunctional mortars	1	1.5	0.4	-
Strain Test	50% SW Multifunctional mortars	1	1.5	0.2	0.2
TGA, ICP, ACIS test	100% SW Multifunctional mortars	1	1.5	-	0.4
Self-healing test;	0% SW Multifunctional pastes	1	-	0.4	-
TGA, XRD, SEM,	50% SW Multifunctional pastes	1	-	0.2	0.2
EDS test;	100% SW Multifunctional pastes	1	-	-	0.4

Note: Sand/Cement (S/C); Deionised Water/Cement (W/C); Seawater/Cement (SW/C); Multiwalled Carbon Nanotubes/Cement (M/C).

The synthetic SW was prepared in the laboratory in accordance with ASTM D1141, ensuring consistency in ion composition with that of natural seawater (Bogue, 1955; P. Li et al., 2023). The concentration of each ionic constituent in the ASTM D1141-prepared SW is summarised in Table 7.3. To maintain a stable pH of 8.2, sodium hydroxide (NaOH) solution was added, following the specifications of the ASTM D1141 protocol. The key properties of crystalline admixture used in this study are provided in Table 7.2.

Table 7.3 Ion concentrations of SW (mmol/L)

Ions	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	Cl ⁻	SO ₄ ²⁻
Value	484.3	10.5	10.6	54	565.5	29.2

Table 7.4 Chemical compositions of raw materials

Chemical compositions (%)	CA
CaO	49.24
Al ₂ O ₃	3.73
SiO ₂	14.15
P ₂ O ₅	0.17
SO ₃	1.83
K ₂ O	0.74
TiO ₂	0.12
V ₂ O ₅	< 0.01
Cr ₂ O ₃	0.01
Mn ₃ O ₄	0.08
Fe ₂ O ₃	1.76
Na ₂ O	9.15
MgO	3.27
LOI	15.78

7.2.2 Sample preparation

Previous studies have emphasised that achieving full dispersion of conductive fillers in the mixing water is critical to ensuring the uniform preparation of cementitious composites (Hassan et al., 2017). In this study, an ultrasonicator was employed to facilitate the homogeneous dispersion of MWCNTs in the mixing water. Two mould geometries were adopted: prisms (40 × 40 × 160 mm³) and cubes (50 × 50 × 50 mm³). The dosage of CA was fixed at 1 wt% of cement, following the recommendation of (Lin et al., 2024) to achieve optimal self-healing performance.

To prepare the cementitious samples, all dry constituents were first blended in a 5 L laboratory Hobart mixer for 3 minutes. Subsequently, half of the mixing water containing the dispersed MWCNTs was added and mixed for 1 minute, followed by the gradual incorporation of the remaining water. Mixing was then continued at medium speed for a

further 3 minutes. The inner surfaces of the mixing bowl were scraped with a trowel to ensure complete homogenisation, after which mixing proceeded for an additional 2 minutes. The fresh pastes and mortars were then cast into designated moulds for self-healing and self-sensing tests and compacted on a vibrating table for 2 minutes to eliminate entrapped air. After 24 hours of curing, the specimens were demoulded and transferred to a curing tank for continued conditioning. The specimens were cured and tested under controlled environmental conditions, with relative humidity maintained at approximately 95% and temperature at 23 °C. These conditions were selected to minimise environmental fluctuations and to ensure consistent evaluation of piezoresistive and self-healing behaviour.

7.2.3 Dry and wet self-healing test after 28 and 56 days

Controlled cracks were introduced into the self-healing prisms using a universal testing machine (UTM) under displacement-controlled loading at a constant rate of 0.05 mm/min. Loading was applied until surface cracks became visible but prior to complete structural failure, as schematically illustrated in Fig. 7.1. Following crack induction, specimens were immersed in different curing tanks containing tap water. For each group, six prisms were subjected to wet–dry (WD) curing cycles, while an additional three were maintained under continuous water saturation (Roig-Flores et al., 2016). Each WD cycle consisted of five days of wet curing immediately after demoulding, followed by five days of air-dry curing at ambient laboratory conditions, with a total of four cycles applied. To prevent further propagation of cracks during curing, the specimens were secured with rubber bands. Healing progress was monitored using an optical microscope (Carl Zeiss Axiotech Vario 100HD) equipped with an Eakins 48 MP FHD camera. Cracks were induced by applying a controlled compressive load to the specimens, as illustrated in Figure 7.1. The same loading conditions were applied to all samples to ensure consistent crack formation.

This approach enabled comparable crack locations and geometries across specimens, thereby minimising variability in the initial crack condition prior to the self-healing process. Baseline images were captured immediately after crack formation, and subsequent images were recorded after each curing cycle to assess healing development. A representative specimen with a visible surface crack is shown in Fig. 7.2. For quantitative analysis, crack areas were evaluated using the greyscale method in ImageJ®. The self-healing performance was evaluated using image-based analysis. As shown in Figure X, the crack region is represented by the white area in the processed images. As the healing process progresses, the white area decreases due to crack closure and the formation of healing products. The healing ratio was quantified as the ratio of the crack (white) area to the total analysed area. A reduction in this ratio indicates the extent of crack closure during the healing process. All images were standardised to a resolution of 235 pixels in width and 1301 pixels in length to ensure consistency and comparability across specimens.

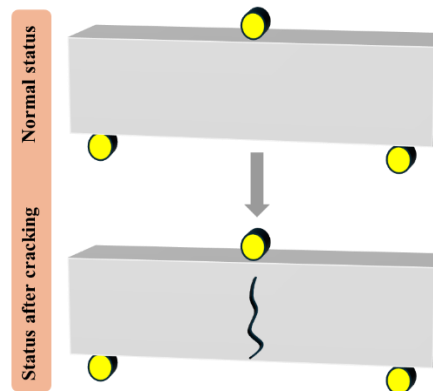


Fig. 7.1 The crack initiation process of self-healing paste.

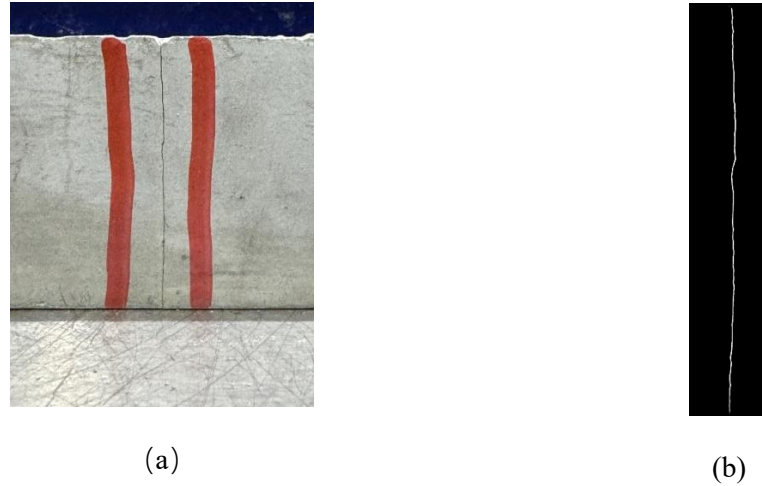


Fig. 7.2 (a) Multifunctional samples cracks; and (b) cracks after grey scale measurement (scaled by ImageJ)

7.2.4 Other methodologies

Other methodologies were same as Chapters 3 and 4.

7.3 Piezoresistivity

Fig. 7.3 illustrates the piezoresistive behaviour of self-sensing specimens prepared with and without seawater (SW). Prior studies have established that the electrical response of self-sensing cementitious composites is predominantly governed by electron conduction mechanisms (Wang & Aslani, 2022). In this investigation, the incorporation of SW induced a pronounced influence on the fractional change in resistance (FCR). For the control (CO) specimens, the FCR measured 0.9% at 0.1 wt% MWCNTs but decreased to 0.2% when the filler dosage increased to 0.3 wt%, under a compressive stress of 3.4 MPa. By contrast, specimens prepared with 50% SW exhibited a marked enhancement in piezoresistive performance, achieving FCR values of 2.0% and 2.75% for 0.1 wt% and 0.3 wt% MWCNTs, respectively. However, this trend was not sustained in the 100% SW specimens, where the FCR values declined to 1.3% and 1.4% at the corresponding filler dosages. These findings highlight that the self-sensing behaviour of cementitious composites is governed not only by the concentration of conductive fillers but also by the ionic composition of the pore solution, as influenced by the proportion of SW used as

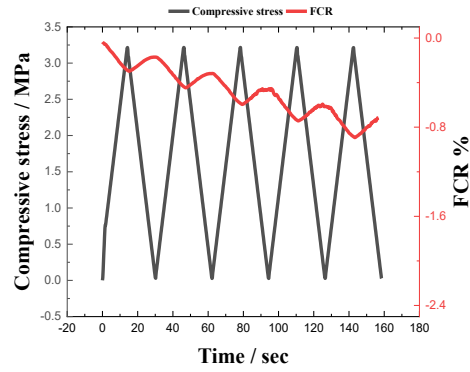
mixing water. This observation aligns with the conclusions of (Deng et al., 2024), who demonstrated the critical role of pore solution chemistry in modulating piezoresistive performance.

The operating mechanism of self-sensing sensors relies on the generation of supplementary conductive pathways within the composite matrix under compressive loading (Deng et al., 2024; Dong, Li, Vessalas, et al., 2021). Upon unloading, these pathways are disrupted, resulting in reversible changes in the material's electrical resistance. During each loading-unloading cycle, the electrical resistance of the self-sensing cementitious composites fluctuated consistently with the applied mechanical stress. When compared with earlier findings (Dong, Li, Wang, et al., 2022), the FCR values obtained in this study were lower than the 0.9% previously reported under equivalent compressive stress. Importantly, the observed increase in FCR can be attributed to the dual effect of seawater incorporation: the presence of additional ions in the pore solution and the facilitation of new conductive pathways, both of which enhance the piezoresistive response.

The ion concentrations in the pore solutions of the specimens were quantified using ICP, and the results are presented in Fig. 7.4. Consistent with previous research (Zhan et al., 2023b), the presence of Na^+ , K^+ , and Ca^{2+} ions was found to enhance the electrical conductivity of the composites. The concentrations of Ca^{2+} ions in the 0 SW and 50 SW specimens were comparable, whereas the 100 SW specimens exhibited a substantially higher concentration, reaching 1.04 mmol/L. A similar trend was observed for K^+ ions across the three groups. In contrast, the concentration of Na^+ ions in the 50 SW specimens were approximately three times greater than in the 0 SW group, reaching 41.25 mmol/L, while the 100 SW specimens exhibited a marked increase to 103.72 mmol/L,

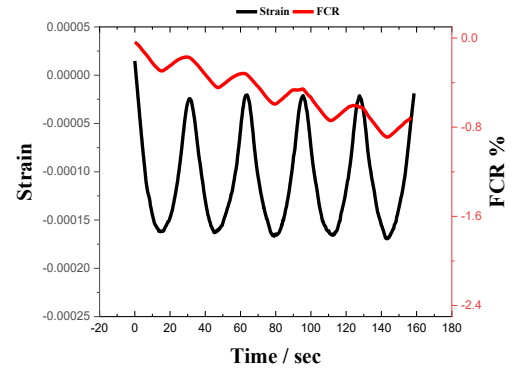
approximately 7.5 times higher than the control. These findings highlight that the elevated Na^+ concentration plays a critical role in modulating the FCR, thereby exerting a dominant influence on the piezoresistive behaviour of the specimens.

Compressive stress - FCR



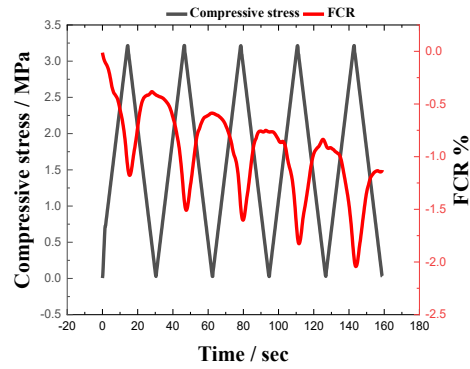
0 SW

Strain - FCR

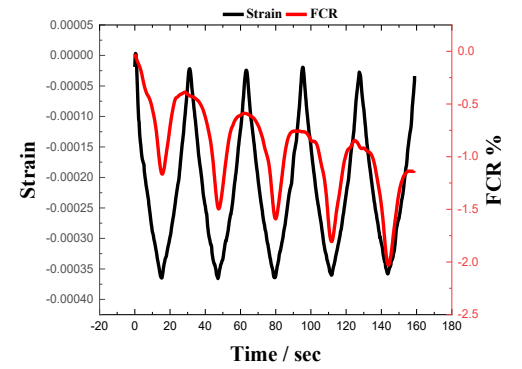


0 SW

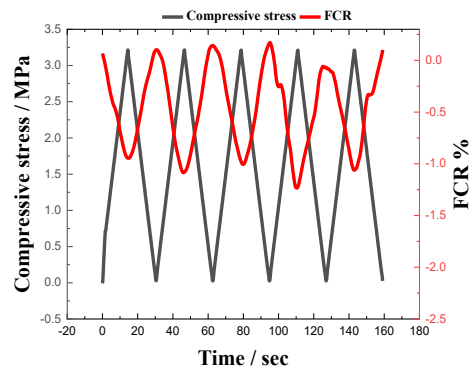
0.1 %



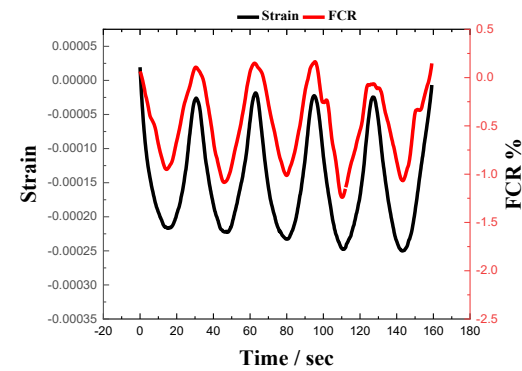
50% SW



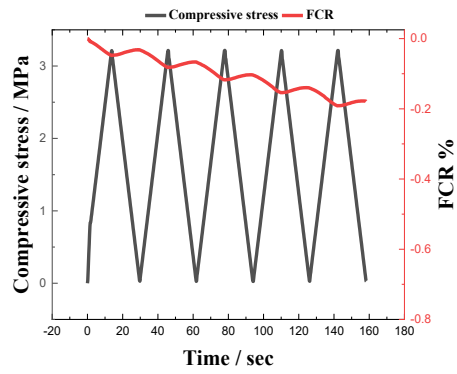
50% SW



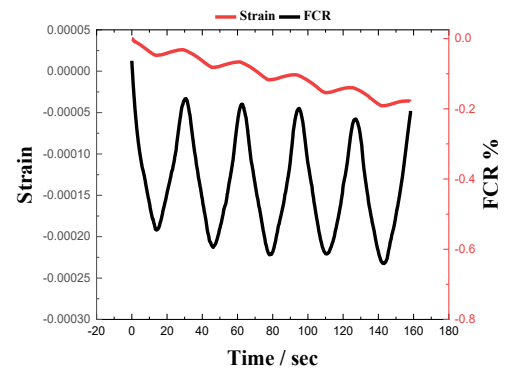
100% SW



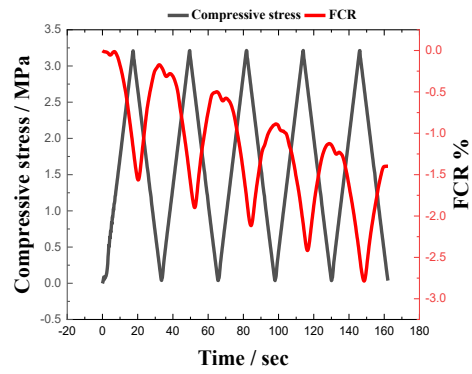
100%SW



0 SW

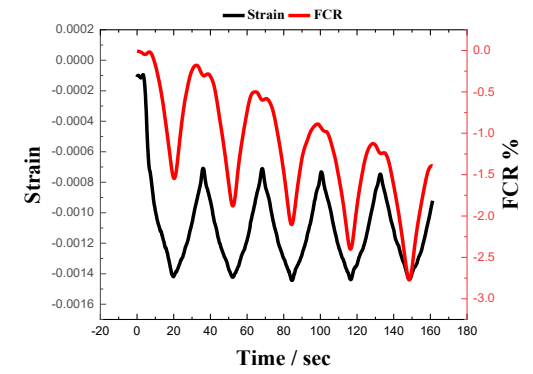


0 SW

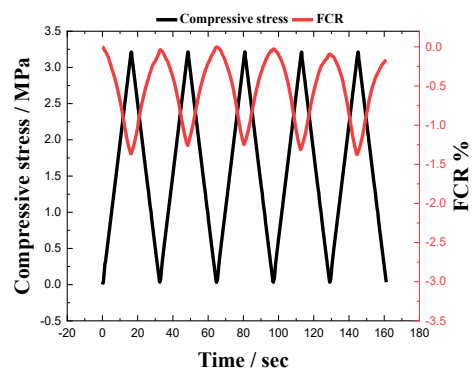


0.3 %

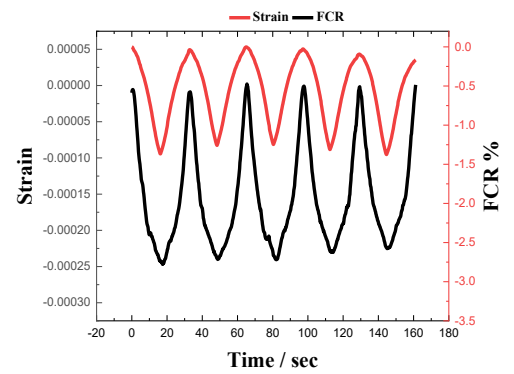
50% SW



50% SW



100% SW



100% SW

Fig. 7.3 Piezoresistivity test of multifunctional specimens with 0.1% and 0.3% content of MWCNTs

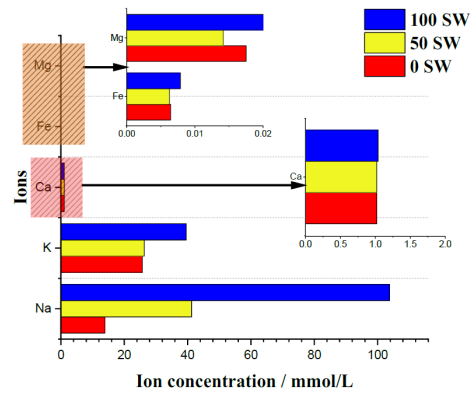


Fig. 7.4 Ions concentration (mmol/L) of multifunctional specimens with different SW content

7.4 Mechanical and electrical properties

Fig. 7.5 presents the electrical impedance data of the self-sensing specimens. A rightward shift of the impedance curve indicates an increase in electrical resistance. Among all groups, the 50 SW specimens exhibited the highest resistance, suggesting the presence of an optimal ion concentration threshold. Furthermore, specimens mixed with SW displayed higher resistance than the CO specimens. This finding implies that chloride ions from SW were physically absorbed by C–S–H gels (P. Li et al., 2021). The use of SW in cementitious mixtures is also known to reduce the volume of macropores; together with the formation of additional hydration products, this contributes to the increased resistivity observed in SW-based specimens (Buenfeld et al., 1986). To validate these observations, ACIS testing was conducted, and the results are shown in Fig. 7.5. After 28 days of curing, the compressive strength of the specimens consistently increased with higher SW content (Fig. 7.6). This trend indicates that SW promotes hydration, likely through the formation of chloride-containing reaction products, a finding further corroborated by the TGA results presented in Section 3.3. Fig. 7.4 reveals that the Na^+ concentration in the pore solution of the 100 SW specimens was 2.5 times greater than that in the 50 SW group. This suggests that Na^+ plays a limited role in the generation of

hydration products. Instead, chloride ions appear to enhance electrical resistance by promoting the formation of additional reaction products, whereas excessive Na^+ increases ionic mobility, thereby reducing the overall resistivity of the cementitious matrix (Zhan et al., 2023b).

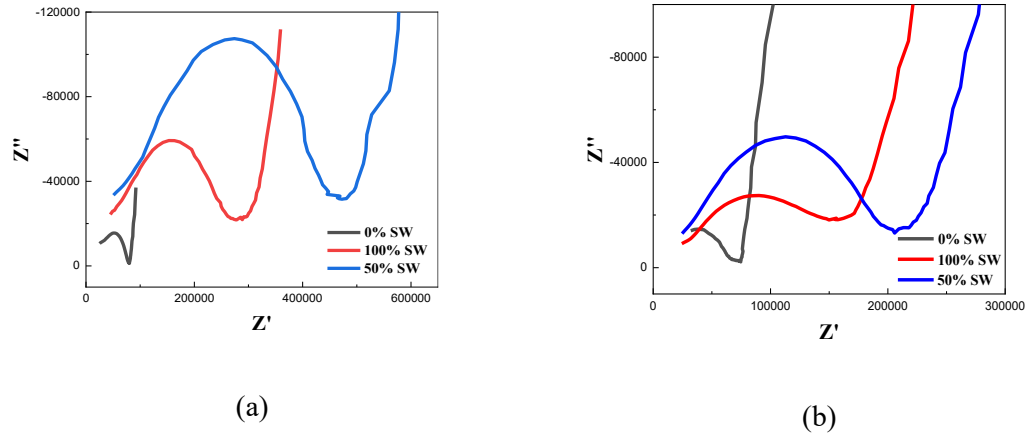


Fig. 7.5 Electrical impedance test results of (a) 0.1% MWCNTs and (b) 0.3% MWCNT self-sensing specimens.

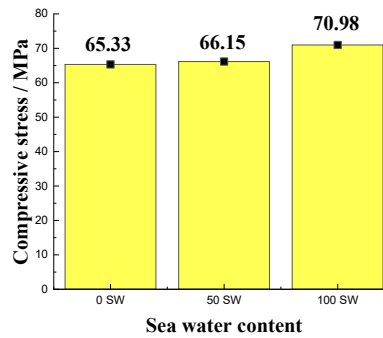


Fig. 7.6 Compressive strength of specimens with/without SW.

Fig. 7.7 presents the thermogravimetric analysis (TGA) results of all self-sensing specimens after 56 days of curing. In the control group, the first peak in the derivative thermogravimetric (DTG) curve occurred between 50–200 °C, corresponding to the evaporation of free water and dehydration of hydration products. The second peak, observed between 350–450 °C, was associated with the decomposition of $\text{Ca}(\text{OH})_2$, a

primary hydration product of cement. The third peak, located between 600–800 °C, confirmed the decarbonation of CaCO_3 , reflecting carbonation processes within the cementitious matrix (Liu et al., 2019). In contrast, the specimens prepared with 100% seawater (SW) exhibited distinct thermal behaviour. Notably, an additional peak appeared between 450–500 °C in the DTG curve. This mass loss is attributed to the decomposition of magnesium silicate hydrate (MSH) gel, specifically due to the release of coordinated water from the MSH structure. The appearance of this peak evidences the formation of MSH as a hydration product, arising from the elevated magnesium ion concentration in SW, thereby differentiating the hydration mechanism from that of conventional cementitious systems (Tartaglione et al., 2008). Another distinct peak, absent in the control group, was detected between 550–600 °C, corresponding to the decomposition of MgCO_3 (Jin & Al-Tabbaa, 2013). The final DTG peak, recorded between 600–800 °C, corresponded to calcium carbonate decarbonation. In the control group, this peak was associated with a 1.82% mass loss, whereas in the 100 SW specimens, the mass loss increased to 2.39%. The additional mass loss in the SW-based specimens can be ascribed to the concurrent decomposition of both CaCO_3 and MgCO_3 within this temperature range. The production of magnesite demonstrates that magnesium ions from SW interact with carbonate species during hydration (P. Li et al., 2020), leading to the formation of additional hydration products, as further evidenced in previous findings (Zhan et al., 2023b). Consequently, the compressive strength of the multifunctional specimens was enhanced, as illustrated in Fig. 7.6.

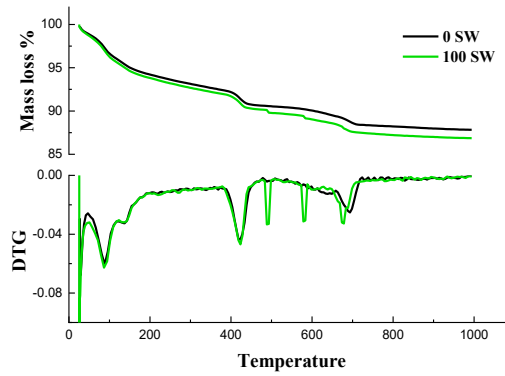


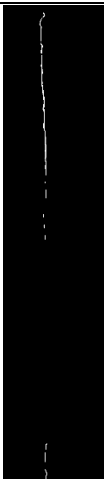
Fig. 7.7 TGA test results of self-sensing samples

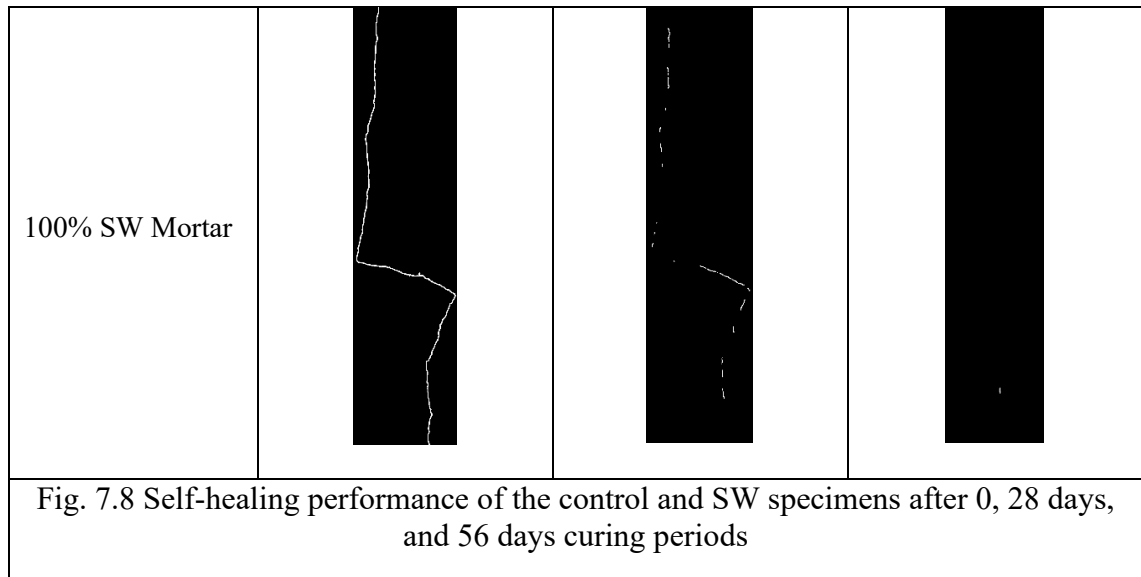
7.5 Self-healing performance

The self-healing performance of the cementitious specimens, recorded using an FHD camera and analysed with ImageJ®, is presented in Fig. 7.8. After 28 days of curing, visible crack closure, either complete or partial, was observed, accompanied by white precipitates identified as healing products within the cracks. The extent of healing varied according to mix composition, with the most pronounced healing occurring in specimens prepared with 100% seawater (SW), as shown in Fig. 7.8. Quantitative image analysis results, summarised in Table 7.5, reveal that the healing rate after 28 days was 58.24% for the 0% SW group, 65.04% for the 50% SW group, and 84.57% for the 100% SW specimens. After 56 days, the self-healing rate of the CO specimens increased to 88.98%, while both the 50% and 100% SW specimens achieved healing rates exceeding 95%. The dominant self-healing products were identified as calcium carbonates, as confirmed by XRD and TGA analyses. Notably, under wet–dry (WD) curing cycles, the 100% SW specimens demonstrated a faster initial healing response compared to the other groups. By contrast, specimens subjected to saturated curing exhibited negligible healing; hence, their performance is not discussed further.

Table 7.6 Self-healing rate of specimens

Type of specimen	0 days/area	28 days/area	56 days/area	Healing rate %	
				0-28	0-56
0 SW specimens	6223	2599	686	58.24%	88.98%
50% SW specimens	5724	2001	128	65.04%	97.76%
100% SW specimens	7143	1102	76	84.57%	98.94%

	0 days	28 days	56 days
0 SW Mortar			
50% SW Mortar			



7.6 Self-healing products analysis

TGA and XRD analyses were employed to identify the self-healing products. The TGA results (Fig. 7.9) reveal that the mass loss observed between 105 °C and 200 °C was primarily associated with the evaporation of bound water from C–S–H in the self-sensing specimens (Lothenbach et al., 2012). Mass losses between 400–450 °C and 600–700 °C corresponded to the decomposition of Ca(OH)_2 and the decarbonation of CaCO_3 , respectively. After the incorporation of seawater (SW), a substantially larger mass loss was detected within the 105–200 °C range. This increase is attributable to the release of bound water from ettringite, Friedel’s salt, C–S–H, and hydrotalcite phases (Álvarez-Ayuso & Nugteren, 2005; Yue et al., 2018). Across all specimens, the greatest mass loss occurred between 600–800 °C, corresponding to the decarbonation of CaCO_3 .

Quantitative analysis of the percentage mass losses associated with each component enabled the estimation of self-healing product composition. In the control group, mass losses were 3.68% for bound water, 1.08% for Ca(OH)_2 , and 19.33% for CaCO_3 . In the 50 SW group, the respective values were 3.26%, 1.16%, and 19.98%. In the 100 SW group, mass losses increased to 3.78% for bound water, 1.22% for Ca(OH)_2 , and 20.49% for CaCO_3 .

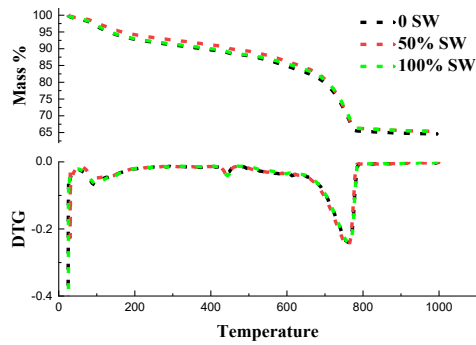


Fig. 7.9 TGA test results of self-healing products from three types of specimens

Fig. 7.10 presents the XRD results of the self-healing products. Consistent with earlier investigations (J. Li et al., 2024), the main crystalline phases identified were portlandite, with characteristic peaks at 18° , 34.1° , and 47.5° , and calcite, with peaks at 23° , 29.4° , 39.3° , and 47.5° . Tobermorite, the crystalline analogue of C–S–H, was detected at 28.7° and 32.2° . In addition to these major phases, several secondary hydration products were identified. Friedel’s salt was detected at 12° , 23° , 31.7° , and 39.3° , while ettringite appeared at 15.8° , 19° , and 23° . Hydrotalcite was observed at 12° , 23° , and 35.8° , and brucite at 18° , 39.3° , and 62.3° . Notably, both Friedel’s salt and hydrotalcite were detected at 12° in the 50% and 100% SW groups. Ettringite was evident at 15.8° but absent in the CO specimens. The presence of ettringite and Friedel’s salt indicates the incorporation of Cl^- and SO_4^{2-} ions from seawater into the self-healing products during crack-sealing. Furthermore, the dissolution of Ca^{2+} and OH^- ions into the curing water in cracked regions elevated the local ion concentration, thereby facilitating the formation of ettringite and Friedel’s salt (J. Li et al., 2024). Enhanced crystallinity of these products is associated with an improved self-healing process.

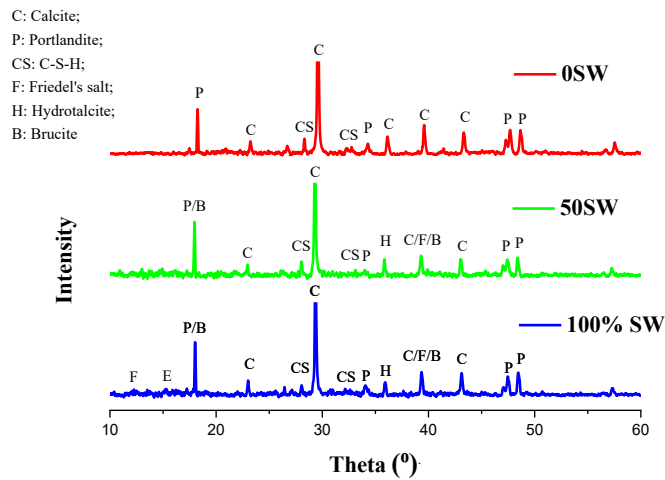


Fig. 7.10 XRD consequence of self-healing products of three types of specimens

The combined results of TGA and XRD demonstrate that the use of seawater (SW) promotes the formation of additional hydration products, which enhance the self-healing capacity of cementitious materials. In particular, CaCO_3 readily precipitates in the SW environment owing to the facile dissolution of Ca^{2+} ions, representing a key mechanism of the healing process. Moreover, the presence of phases such as ettringite and Friedel's salt, generated through the interaction of SW-derived ions with the cement matrix, further strengthens the self-healing mechanisms by contributing to crack filling and microstructural densification.

7.7 Summary

The study employed CA as a self-healing agent and MWCNTs as conductive fillers to develop multifunctional cementitious composites. Various techniques, including XRD, TGA, FTIR, ICP, and cyclic loading tests, were used to analyse hydration and self-healing products, ion conduction, and piezoresistive behaviour.

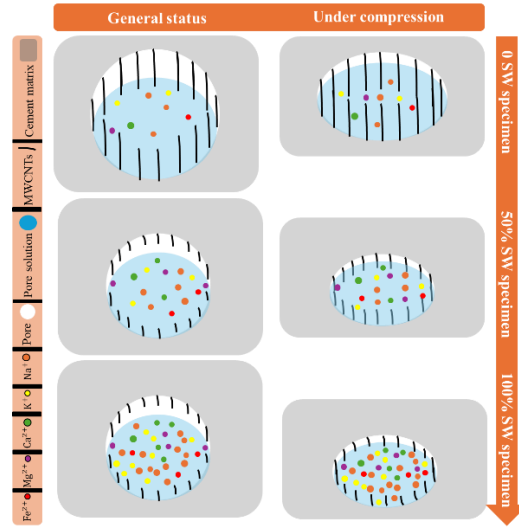


Fig. 7.11. The condition in pores under general and loading status of three types of samples

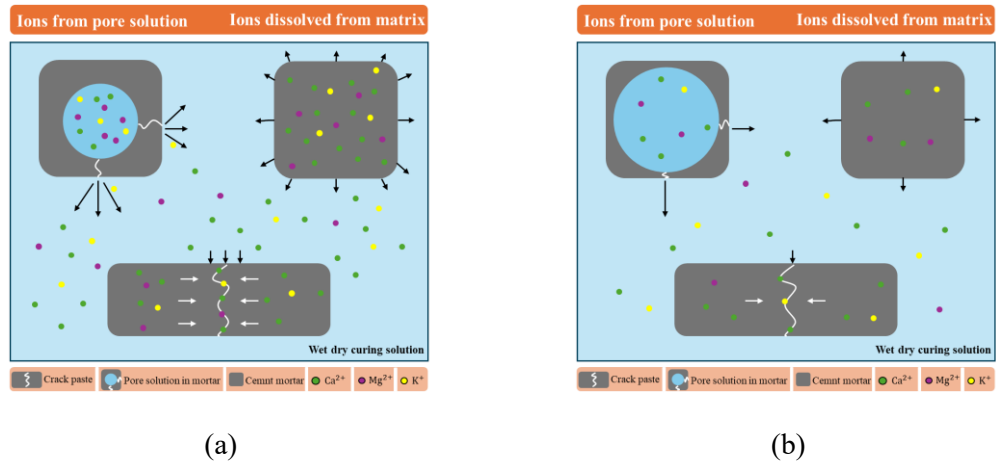


Fig. 7.12. Ions released from pore solution and dissolved from cement mortar to wet curing solution in wet dry cycle to enhance the self-healing performance of multifunctional cementitious matrix with (a) SW content; (b) and without SW

The electrical resistance gap between loading and unloading was increased. This phenomenon was presented in Fig. 7.11. As mentioned above, it can be concluded that piezoresistivity was not only influenced by the number of conductive fillers but also affected by ion concentration in the pore solution. In that case, the percolation thresholds of self-sensing cement-based sensors may be influenced by the ion concentration of the cementitious matrix.

The ions from seawater can enhance the performance of CA to accelerate the self-healing process. This phenomenon was illustrated in Fig. 7.12. As Ca^{2+} is one of the major ions that influence the self-healing process, and it has been applied to form the healing products during the self-healing process. Because of that, a low concentration of Ca^{2+} was found in ICP results from uncracked specimens. At the same time, the efficiency of the self-healing process has been improved. The high concentration of ions that has been dissolved into the solution, especially Ca^{2+} , K^+ , and Mg^{2+} , can accelerate the precipitation on the exposure surface of cracks. The SW accelerated the self-healing process due to the presence of Ca^{2+} and Mg^{2+} , 50 SW specimens presented the highest healing rate in the early stage, and the increased Na^+ concentration in SW mixes (e.g., 41 mmol/L) significantly improved the piezoresistivity of the multifunctional specimens. In summary, under the influence of SW, both self-healing and self-sensing technologies have been correspondingly enhanced.

Chapter 8: Multifunctional cementitious composites:

Integration of self-sensing and thermal responsiveness

8.1 Introduction

This chapter summarises the experimental findings on integrating self-sensing functionality with thermal responsiveness. The application potential of the multifunctional mixture was evaluated, and the synergistic mechanisms between the two functions were investigated. Structural Health Monitoring (SHM) has been widely recognised as an effective strategy for assessing infrastructure performance (Ba et al., 2024; Fawad et al., 2023; Jia et al., 2025). While thermochromic (TC) materials can mitigate heat accumulation and reduce cracking caused by temperature fluctuations, they are unable to provide information on the internal condition of cementitious systems. To address this limitation, this study combines TC materials with self-sensing cementitious sensors, which have already demonstrated reliable performance in monitoring structural health (H. Wang et al., 2021). Two key conduction mechanisms—electron conduction and ion conduction—affect the piezoresistive performance of cementitious composites. Electron conduction is governed by functional fillers, and the incorporation of conductive materials significantly enhances matrix piezoresistivity (Lee et al., 2022). Among various conductive fillers, carbon nanotubes (CNTs) have been most extensively studied. Owing to their large specific surface area, multi-walled carbon nanotubes (MWCNTs) provide superior electrical conductivity and enhance the self-sensing capacity of cementitious composites (Hong et al., 2024). The ion conduction, caused by the ions in the pore solution of cementitious composites, has also been analysed (Deng et al., 2024). Ion conduction, in contrast, is governed by the concentration of ions present in the pore solution, with Na^+ , K^+ , and Ca^{2+} being the primary contributors to electrical

conductivity (Zhan et al., 2023b). To advance infrastructure functionality, this study explores the combined application of self-sensing sensors and thermochromic coatings, aiming to develop multifunctional cementitious systems with both monitoring and thermal-control capabilities.

This study investigated the thermochromic (TC) properties and piezoresistive behaviour of seawater (SW)–mixed cementitious composites. Due to the elevated concentrations of Na^+ , K^+ , and Ca^{2+} , SW is expected to substantially increase the ionic content of the pore solution (T. Liu et al., 2024). The temperature responsiveness of TC coatings was evaluated using ultraviolet–visible (UV–Vis) spectroscopy, Fourier transform infrared spectroscopy (FTIR), and Raman spectroscopy (RS) to characterise light absorbance, transmittance, and spectral intensity. In addition, X-ray diffraction (XRD), thermogravimetric analysis (TGA), inductively coupled plasma (ICP) analysis, energy-dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM) were employed to assess the influence of SW on cement hydration and microstructural development.

8.2 Experimental program

8.2.1 Raw materials and sample preparation

Multi-walled carbon nanotubes (MWCNTs) were employed as conductive fillers in the self-sensing cementitious specimens (SSCS). The hydration process can be enhanced by seawater (SW) and free ions such as Na^+ , K^+ , and Ca^{2+} released into the pore solution (Qu et al., 2021; Zhan et al., 2023a). To examine the influence of ion conduction on piezoresistive behaviour, 50% of the mixing water was replaced with SW. A series of MWCNT dosages, 0.1%, 0.3%, 0.5%, 0.7%, 1.0%, and 2.0% by weight of cement, was tested to determine the percolation threshold. Electrical impedance measurements were

conducted to evaluate the electrical resistance of each sample, and the percolation thresholds are presented in Fig. 8.1. Based on these results, a dosage of 0.5% MWCNTs (denoted as 0.005 in Fig. 8.1) was selected for subsequent experiments. The mix proportions and experimental parameters are summarised in Table 8.1. The selected proportions of MWCNTs and seawater are discussed in detail in Section 4, where the percolation behaviour and the influence of ionic concentration on material performance are systematically analysed.

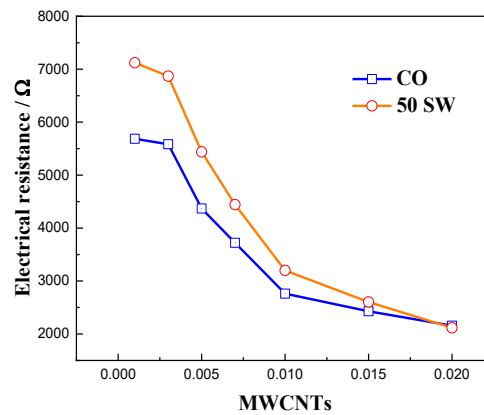


Fig. 8.1 Percolation thresholds of CO and 50 SW self-sensing specimens; CO indicates no seawater, and 50 SW indicates 50% of the mix water is replaced with seawater

Table 8.1 Components of cementitious composites

Sample type	Cement	S/C	W/C	SW/C		TC coating
Sample without coating	100%	1.5	0.4	CO	—	—
			0.2	50 SW	0.2	
Sample with red coating	100%	1.5	0.4	CO	—	Coated with Red
			0.2	50 SW	0.2	
	100%	1.5	0.4	CO	—	

Sample with black coating			0.2	50 SW	0.2	Coated with Black
Sample with blue coating	100%	1.5	0.4	CO	–	
			0.2	50 SW	0.2	Coated with Blue

Note: S/C: sand/cement ratio; W/C: water/cement ratio; SW/C: seawater/cement ratio; TC coating: thermochromic coating; CO: control group specimens without SW; CC: uncoated control specimens; 50 SW: specimen with 50% SW.

A General Purpose (GP) cement conforming to AS 3972 (Standard, 2010) was used as the binder. A solution consisting of 50% deionised water and 50% seawater (SW) was prepared in accordance with ASTM D1141 (Testing & Materials, 2013). The pH of the SW was adjusted to 8.2 by adding NaOH pellets (P. Li et al., 2023), and the ion concentrations are summarised in Table 8.2. MWCNTs were ultrasonicated in the mixing water for 40 min at a constant frequency of 40 kHz to ensure uniform dispersion. The MWCNT suspension was then combined with SW and added to the dry cement–sand mixture in a Hobart mixer. Mixing was continued for at least 5 min to achieve homogeneity. The fresh mixture was subsequently cast into $50 \times 50 \times 50 \text{ mm}^3$ moulds embedded with copper mesh electrodes, which remained in place during setting and hardening. After 24 h of initial curing, the specimens were demoulded and transferred to a curing chamber maintained at $23 \pm 2 \text{ }^\circ\text{C}$ and 98% relative humidity for 28 days.

Table 8.2 Ion concentration of SW (mmol/L)

Ions	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	Cl [−]	SO ₄ ^{2−}
Concentration	484.3	10.5	10.6	54	565.5	29.2

After 28 days of curing in the chamber, the specimens were conditioned in a controlled environment maintained at $23 \pm 2 \text{ }^\circ\text{C}$ and $50 \pm 3\%$ relative humidity for a minimum of

three days. This step ensured that the specimen surfaces reached an undersaturated state suitable for the application of TC coatings. One surface of each self-sensing specimen was then coated with a designated TC coating, as described in Table 8.1. Three types of colour coatings, black, blue, and red, were investigated, with further details provided in Table 8.3. The coatings were applied manually using a paintbrush, after which the specimens were placed in a climate chamber at 25 °C until the coatings dried completely. To evaluate the thermochromic response, the coated samples were subsequently transferred to an oven set at 31 °C. A visible change to a lighter shade within 2 min confirmed the activation and responsiveness of the TC coatings.

Table 8.3 Components of three different colours of TC coating

Components	TC Black	TC Blue	TC Red
Polyoxymethylenemelamine	20.1 wt%	20 wt%	19-27.2 wt%
Styrene maleic anhydride monomethyl-maleate polymer	6.2 wt%	6.0 wt%	5.8-7.2 wt%
Methyl stearate	50 wt%	50 wt%	45.6-47.5 wt%
Ethyl stearate	20.3 wt%	20 wt%	15.2-15.8 wt%
ODB-II	3.0 wt%	—	—
Green DCF	0.4 wt%	—	—
CVL	—	4 wt%	—
Orange DCF	—	—	1.6-3.4 wt%
Red DCF	—	—	0.7-1.5 wt%

Note: ODB-II: 2-Anilino-6-dibutylamino-3-methylfluoran; Green DCF: 2'-(Dibenzylamino)-6'-(diethylamino) fluoran; CVL: Crystal Violet Lactone; Orange DCF: 6'-(Diethylamino)-1',3'-dimethylfluoran; Red DCF: 6'-(Diethylamino)-1',2'-benzofluoran.

8.2.2 Temperature control measurement

The thermochromic-coated self-sensing cementitious specimens (SSCS) were exposed to a high-intensity discharge (HID) xenon lamp, which enabled precise temperature control during testing. The lamp operates with xenon gas, producing light via arc discharge by converting electrical energy into ultraviolet and visible radiation. Owing to its ability to replicate the spectral characteristics of sunlight, the xenon lamp was used to simulate solar radiation (Liu et al., 2022). The temperature-control performance of the TC coatings was evaluated by monitoring changes in surface temperature under simulated sunlight. The experiment was conducted inside a dark box to eliminate external interference. Initially, an infrared thermometer was used to measure the surface temperature of the cement mortar specimens, recorded as T_1 . The xenon lamp was then directed onto the TC-coated surface. After 5 min of exposure, the specimen was removed and the surface temperature recorded as T_2 . After a further 10 min of exposure, the surface temperature was again measured and recorded as T_3 . The temperature rise was calculated from the differences between T_1 , T_2 , and T_3 , enabling a direct comparison between TC-coated specimens and uncoated control specimens (CC). The experimental setup for temperature control and water evaporation was designed to ensure uniform thermal exposure and controlled environmental conditions. Specimens were subjected to a predefined temperature range using a controlled heating system, while mass loss due to evaporation was monitored over time. This setup enabled the evaluation of thermally induced behaviour and moisture variation in the composite system.

8.2.3 Water evaporation test

The piezoresistive behaviour of self-sensing specimens is strongly influenced by their internal moisture content, with optimal performance typically observed when water content ranges between 80% and 95% (Deng et al., 2024). Piezoresistivity measurements

were therefore conducted after evaporating 5%, 10%, and 20% of the water from fully saturated samples. To ensure complete saturation, the specimens were immersed in deionised water for 72 h. Following saturation, the specimens were dried in an oven at temperature T_3 to achieve the desired moisture levels. During the drying process, the specimens were weighed every 10 min during the first 30 min, and subsequently every 30 min, to monitor moisture loss. Drying continued until the specimen weights stabilised, indicating that equilibrium moisture levels had been reached.

8.2.4 Other methodologies

Other methodologies were same as Chapters 3 and 4.

8.3 Hydration and compressive strength

Fig. 8.1 presents the XRD patterns of specimens prepared with 50% seawater (SW) and those without SW. Peaks corresponding to ettringite (Aft) and portlandite (CH) were identified, with the ettringite peak appearing at $9.1^\circ 2\theta$ and the portlandite peaks located at 18° and $34^\circ 2\theta$. In addition, the presence of Cl^- ions resulted in the formation of Friedel's salt ($\text{Ca}_4\text{Al}_2(\text{OH})_{12}\text{Cl}_2 \cdot 4\text{H}_2\text{O}$), which was detected in the 50% SW samples at 9.1° and $39^\circ 2\theta$. Previous study (Cheng et al., 2016) reported that Friedel's salt typically exhibits a plate-like crystal morphology. Consistent with this, Friedel's salt was observed in the microstructure of the hydrated cementitious matrix prepared with SW, as shown in Fig. 8.3. After 28 days of curing, however, the Friedel's salt signal appeared weak. This may be attributed to the preferential absorption of Cl^- ions by C–S–H gels, which are capable of binding substantial quantities of ions. As a result, many chloride ions may have been incorporated into the C–S–H structure rather than forming Friedel's salt (Hirao et al., 2005; Qiao et al., 2019), reducing the availability of free Cl^- for Friedel's salt precipitation at later hydration stages. A distinct peak was also observed at 45.7° in the XRD patterns of the SW specimens, which was stronger than that in the non-SW

specimens. This peak may be associated with the elevated chloride content introduced by SW. Furthermore, no characteristic gypsum peaks were detected in the hydrated paste samples, which may be explained by the rapid dissolution of gypsum followed by its reaction with C_3S and Cl^- ions (Zhang et al., 2022).

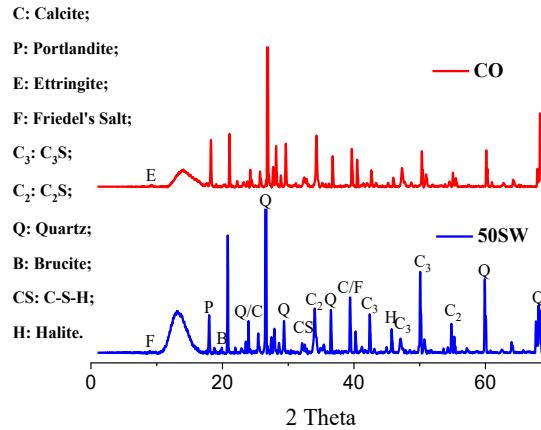


Fig. 8.2 XRD test results of specimens with/without SW after 28 days curing

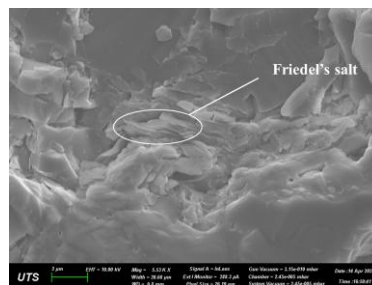


Fig. 8.3 SEM micrograph of Friedel's salt

The thermogravimetric analysis (TGA) results are shown in Fig. 8.4. In the 50% seawater (SW) specimens, the mass loss attributed to portlandite (CH) was approximately 1.83%, slightly lower than the 1.90% recorded for the specimens without SW. This reduction may be associated with the incorporation of Cl^- into Friedel's salt during hydration. Within the temperature range of 50–200 °C, Friedel's salt is known to release both interlayer and strongly bound water. The total mass loss in this range was 4.465% for the non-SW specimens and marginally higher at 4.569% for the 50% SW specimens. Although the

XRD results (Fig. 8.2) indicated only a weak Friedel's salt signal, the increased mass loss in this range provides additional evidence of its presence within the cementitious matrix. At higher temperatures (600–800 °C), the mass loss primarily associated with the decomposition of CaCO_3 , was 1.592% for the non-SW specimens and 1.572% for the 50% SW specimens.

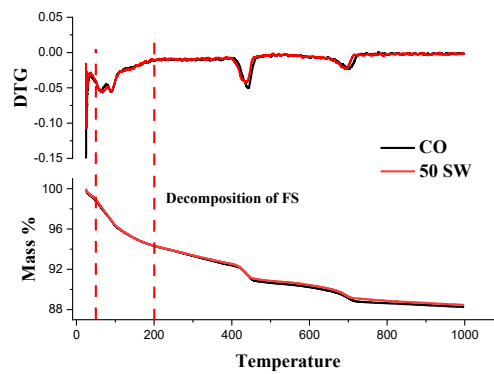


Fig. 8.4 TGA test results of cementitious mixtures with/without SW

Fig. 8.5 illustrates the influence of seawater (SW) on the development of compressive strength in cementitious specimens. After 28 days of curing, the specimens without SW achieved a compressive strength of 42.08 MPa, whereas the 50% SW specimens reached a significantly higher value of 57.08 MPa. These results indicate that the incorporation of SW enhances cement hydration, thereby improving mechanical performance. In addition, the absorption of Cl^- appears to contribute to microstructural modification, particularly through the reduction of macropore volume, as previously reported (P. Li et al., 2020). This matrix densification likely supports strength development by accelerating hydration reactions and facilitating chloride interactions within the cementitious system.

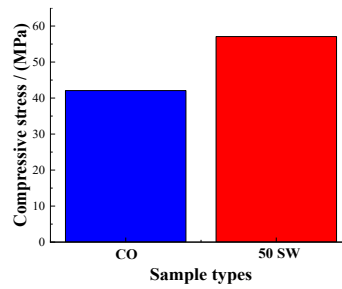


Fig. 8.5 Compressive strength of cementitious matrix after 28 days curing

8.4 Surface temperature control

The temperature-regulation performance of red, black, and blue thermochromic (TC) coatings applied to cementitious composites is presented in Fig. 8.6, together with the corresponding temperature changes and colour transitions under xenon lamp heating. All TC coatings initiated a visible colour shift when the surface temperature reached approximately 31 °C. During the initial heating stage (T_2), a gradual fading of colour was observed, indicating the onset of thermally induced chromatic transition. With further heating (T_3), the coatings became increasingly decoloured as the surface temperature stabilised, after which only minimal variation was recorded under continued exposure. As illustrated in Fig. 8.7, the uncoated specimen exhibited the most rapid rate of temperature increase, reaching 71.9 °C within the first 5 min. In contrast, TC-coated specimens displayed substantially lower surface temperatures, averaging approximately 55 °C during the same stage. By the end of the second heating phase, the uncoated specimen reached a peak temperature of 83.2 °C, corresponding to a total rise of 60.5 °C. Among the coated specimens, the red coating showed the highest final surface temperature (69 °C), while the black coating exhibited the greatest cooling efficiency. The black-coated specimen experienced a total temperature rise of 40.3 °C, yielding a surface temperature 20.5 °C lower than the uncoated specimen under identical photothermal conditions. These results confirm that TC coatings, particularly the black

variant, effectively reduce both thermal accumulation and the rate of surface temperature rise in cementitious composites, consistent with previous findings (Ma & Zhu, 2009). The microstructural features of TC coatings exposed to simulated solar heating are shown in Fig. 8.8. Small surface fissures were observed, especially in regions containing concentrated pigments. TC pigments were particularly prominent due to their tendency to agglomerate on the coating surface. Microscopic analysis further revealed that most microcracks originated near pigment clusters, suggesting a direct link between pigment aggregation and thermally induced surface damage.

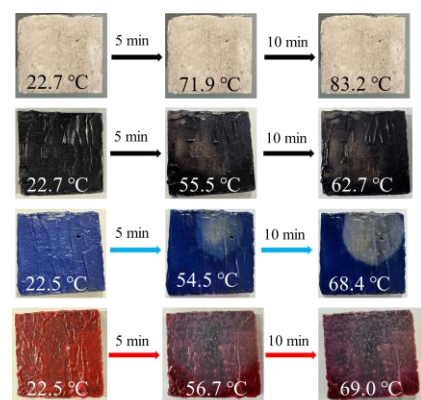


Fig. 8.6 The surface coating colour changes during the two stages of photothermal heating

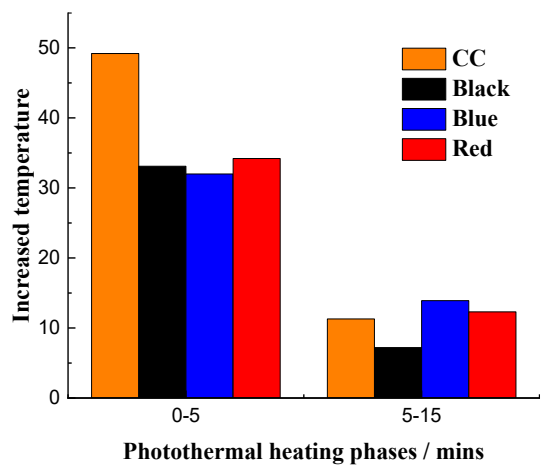


Fig. 8.7 Temperature raising efficiency in two photothermal heating periods

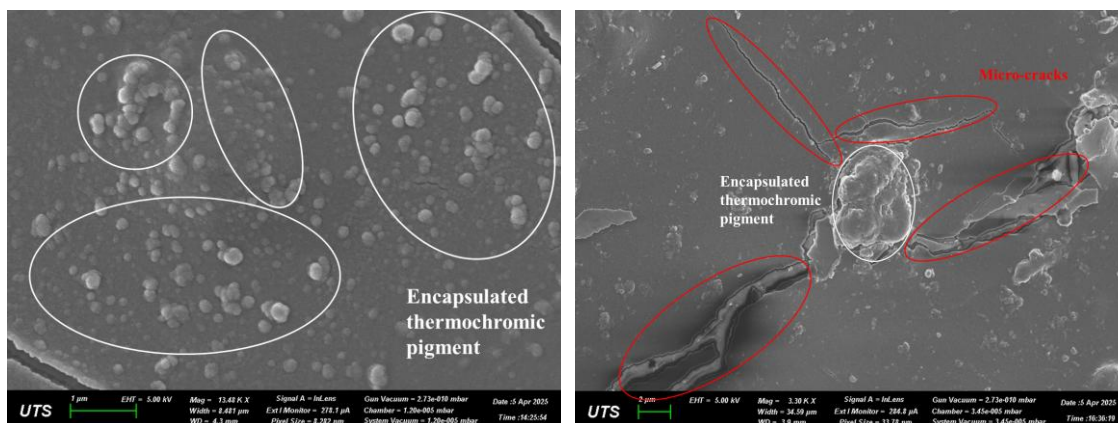


Fig. 8.8 SEM micrographs of thermochromic coating (a) encapsulated reversible thermochromic pigment; (b) microcracks on the surface of coatings.

8.5 TC coating analysis

Fig. 8.9 presents the infrared (IR) transmittance spectra of specimens coated with red, black, and blue thermochromic (TC) layers. All coatings exhibited distinct peaks at 720 cm^{-1} and 815 cm^{-1} . The peak at 720 cm^{-1} corresponds to $-\text{CH}_2$ rocking vibrations in long aliphatic chains, associated with methyl stearate and ethyl stearate components (Smith, 2021). The peak at 815 cm^{-1} is attributed to C–H bending vibrations, which may also be influenced by methyl stearate and ethyl stearate. Additional peaks appeared at 1170 cm^{-1} and 1270 cm^{-1} , assigned to C–O–C stretching vibrations related to CVL, orange DCF, and red DCF components in the coatings. According to the FTIR results, a band observed between $1340\text{--}1360\text{ cm}^{-1}$ corresponds to C–H bending in $-\text{CH}_2$ groups and C–N stretching vibrations, associated with polyoxymethylenemelamine (Pavia et al., 2015). A notable feature in all three coatings is reduced transmittance in the $1460\text{--}1560\text{ cm}^{-1}$ region, where C=C and C=N stretching vibrations and N–H bending typically occur. This behaviour may be attributed to polyoxymethylenemelamine in the coatings. Infrared absorption in this band is caused by dipole moment changes during bond vibrations (Zamadar et al.,

2013). Under the influence of dye molecules, stronger bonds can enhance dipole moment variations (Socrates, 2004). In particular, the highly polar C=N and N-H bonds produce significant dipole moment changes, explaining the relatively low transmittance in this region (J. Luo et al., 2015). At higher wavenumbers, all spectra exhibited transmittance of approximately 60% around 3050 cm^{-1} . This is consistent with aromatic =C-H stretching vibrations, typically occurring in the range 3020–3100 cm^{-1} , which are associated with weak to medium absorption due to limited dipole moment change during stretching (Socrates, 2004). In addition, two distinct peaks at 2850 cm^{-1} and 2920 cm^{-1} were observed in the red and blue TC coatings but were absent in the black coating. These peaks correspond to the symmetric and asymmetric C-H stretching vibrations of methylene ($-\text{CH}_2-$) groups. Their presence suggests that the red and blue coatings contain higher proportions of long-chain aliphatic compounds compared with the black coating. Consequently, the black coating exhibited greater reflectance, whereas the red and blue coatings showed stronger infrared absorption.

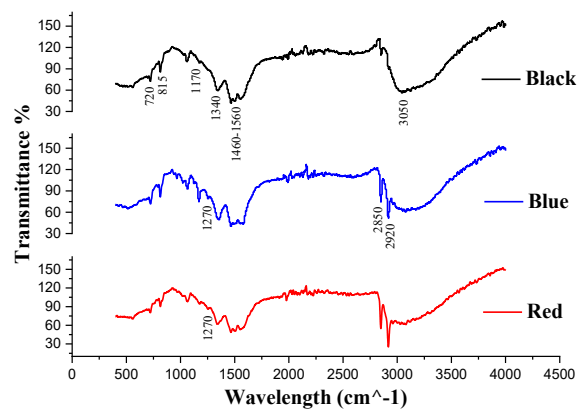


Fig. 8.9 FTIR test results of red, blue, and black coatings

Fig. 8.10 presents the UV-Vis-NIR absorption spectra of specimens coated with black, blue, and red thermochromic (TC) layers. Solar irradiation typically consists of approximately 5% ultraviolet (UV), 43% visible, and 53% near-infrared (NIR) energy (X.

Wu et al., 2013). The uncoated specimens exhibited consistently high absorbance across the entire 237–2000 nm spectral range. In the NIR region (800–2000 nm), their absorbance values exceeded 1.0, the highest among all specimens. Similarly, in the visible range (437–700 nm), uncoated specimens displayed the greatest absorbance, indicating that they absorbed more solar energy than the TC-coated specimens.

For the black TC coating, absorbance values exceeded 1.0 across the UV (200–400 nm) and visible (400–700 nm) ranges, particularly within 247–673 nm. This is consistent with the high light-absorption capacity of black surfaces under solar radiation. In the NIR range (700–2000 nm), the black coating exhibited absorbance values between 0.65 and 0.86. The blue coating displayed a distinct absorbance dip at 450–480 nm, corresponding to enhanced reflection in the blue–violet region of the spectrum (~450 nm) (Sharkey et al., 2020). Its primary visible-light absorption occurred between 600–650 nm (Cheng et al., 2015). The blue coating also exhibited relatively low absorbance in the NIR band compared with the black and red coatings. The red coating and the black coating both exhibited relatively low absorbance in the 245–580 nm range. Beyond 600 nm, however, the red coating showed diminished absorbance due to strong reflectance in the orange–red region of the visible spectrum. Nevertheless, the red coating absorbed more NIR light than either the black or blue coatings, suggesting a greater capacity to retain solar energy through enhanced NIR absorption.

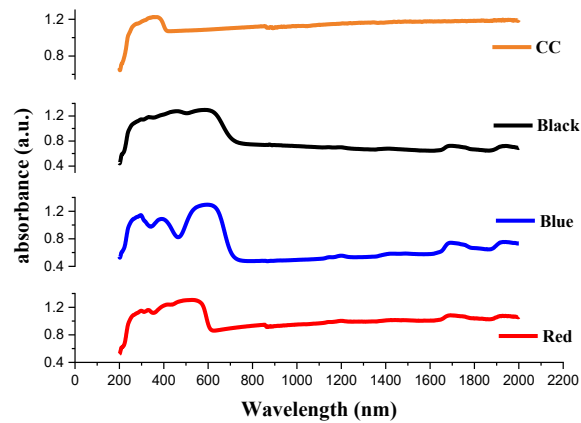


Fig. 8.10 UV test results of CC, black coated, blue coated, and red coated specimens

8.6 Water loss rate and correlation between water content and piezoresistivity

The specimens with black TC coating exhibited a steady maximum surface temperature of approximately 62 °C, the lowest among all samples (Fig. 8.6). In contrast, the uncoated control (CC) specimens reached a maximum temperature of 83 °C. To evaluate the influence of TC coatings on moisture loss, water evaporation rates were measured (Fig. 8.11) in parallel with the temperature-control assessment. The uncoated specimens displayed a considerably steeper slope in the water-loss curve during the first 12 h, indicating more rapid evaporation compared with the coated specimens. After 36 h of exposure, specimens subjected to 65 °C and 83 °C were tested for compressive strength. As shown in Fig. 8.12, elevated temperatures exerted a detrimental effect on mechanical performance. Prolonged exposure to 83 °C resulted in substantial reductions in compressive strength due to degradation of the cementitious microstructure, consistent with earlier findings (Care, 2008). By contrast, specimens with TC coatings retained markedly higher performance. After exposure to 80 °C and 105 °C, the coated specimens achieved compressive strengths of 70.5 MPa, approximately 1.5 times greater than those of uncoated specimens exposed to 83 °C. These results demonstrate that TC coatings

effectively mitigate heat-induced microstructural damage and help maintain mechanical integrity under thermal stress.

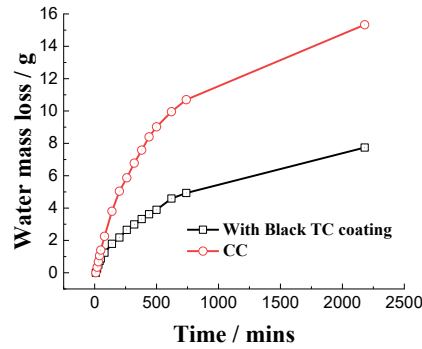


Fig. 8.11 Water evaporation efficiency of cementitious specimens with/without black TC coatings after 36 hours of heating

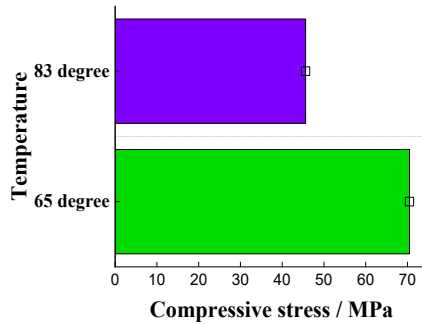
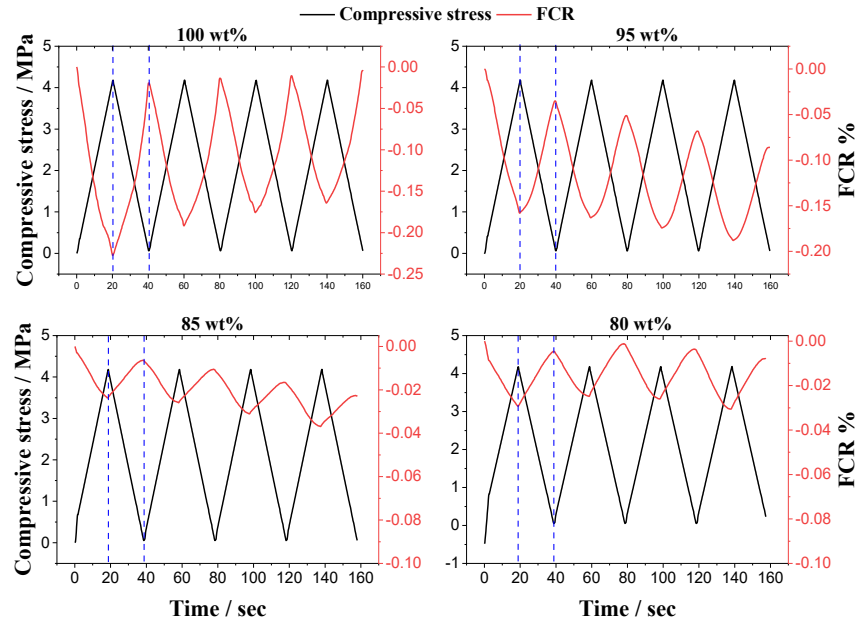


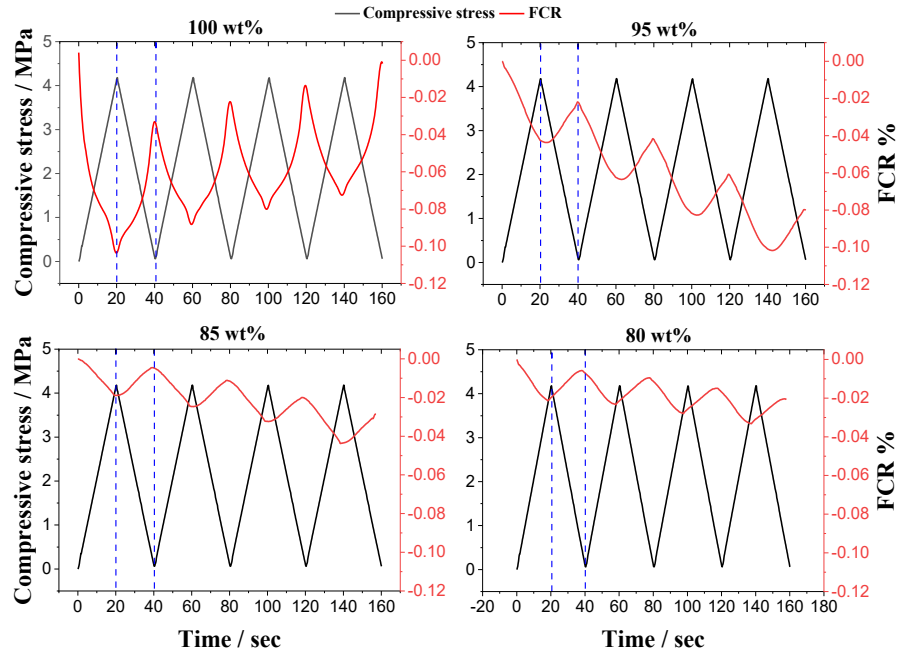
Fig. 8.12 Compressive strength of cementitious matrix at 65 °C and 83 °C

Two primary mechanisms enable self-sensing cementitious sensors to conduct electricity: electron conduction and ion conduction. The concentration of conductive fillers, such as MWCNTs, has the greatest influence on electron conduction. As illustrated in Fig. 8.13, the elongated morphology of MWCNTs embedded in the cementitious matrix facilitates electron transfer. In this investigation, the filler content was fixed at 0.5% for all samples, thereby ensuring comparable electron conduction across specimens.

By contrast, ion conduction plays a critical role in modulating the electrical response, depending on the concentration and mobility of ions within the pore solution. Moisture



(a)



(b)

Fig. 8.14 Piezoresistivity test results of (a) 50 SW and (b) CO samples under different water wt% content

TC coatings effectively mitigated the temperature rise of self-sensing cementitious specimens. The reduced surface temperature significantly decreased the rate of water evaporation (Fig. 8.11), thereby promoting greater retention of pore solution within the

matrix. The increased availability of pore solution enhanced ionic conductivity by supporting the formation of continuous ion-conductive pathways in the self-sensing specimens.

8.7 Summary

The encapsulated thermochromic (TC) coating effectively reduced the surface temperature of cementitious specimens by reflecting solar radiation. This temperature reduction limited water evaporation, enhanced ion conduction, and thereby stabilised piezoresistive performance. However, surface cracking caused by pigment aggregation highlights the need for improved pigment dispersion in future formulations. Furthermore, the TC-induced lower surface temperature ($\sim 65\text{ }^{\circ}\text{C}$) promoted continued hydration and increased compressive strength by facilitating denser C–S–H formation and the generation of additional hydration products.

The temperature-control effect of the TC coating also influenced the piezoresistivity of the specimens. The relationship between water mass loss and ion conduction is illustrated in Fig. 8.15. Ion transport occurs through the pore solution; however, when water content is insufficient, fewer conductive pathways are established within the cementitious matrix during loading. This condition can produce large variations in FCR but weakens the correlation between the FCR curve and the loading–unloading curve. For practical applications, self-sensing specimens must exhibit consistent and reliable responses to mechanical loading, with well-aligned pressure–FCR correlations.

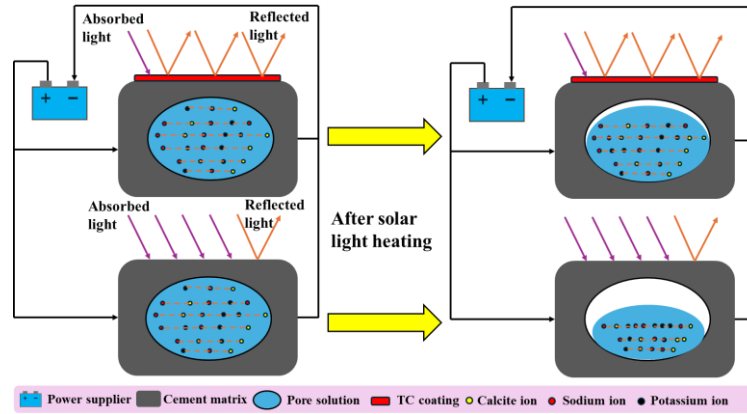


Fig. 8.15. The relationship between the water mass loss and ion conduction change

This chapter demonstrated that self-sensing functionality and TC materials can operate synergistically within cement-based specimens. By regulating specimen temperature and thereby preserving internal moisture, the contribution of ion conduction to piezoresistive performance was enhanced. These experimental findings confirm the feasibility of developing multifunctional pavements that integrate both structural health monitoring and thermal regulation.

Chapter 9: Multifunctional cementitious composites:

Integration of self-sensing and photocatalytic

9.1 Introduction

This section examines the interaction between self-sensing and photocatalytic functions. The following paragraphs provide the research background. The Earth's environmental condition has been profoundly affected by vehicular emissions. The transportation sector accounts for more than one-fifth of global CO₂ emissions, significantly contributing to global warming (Speizer et al., 2024). Freight transport alone produces approximately 2.9 Gt of CO₂ annually through trains, trucks, and related activities (Greene & Façanha, 2019). Between 2000 and 2022, road freight was responsible for nearly 55% of CO₂ emissions (Aryanpur & Rogan, 2024). These figures underscore the urgency of developing innovative strategies to mitigate transportation-related emissions. Photocatalysis has been introduced as a promising method for CO₂ reduction (Fang et al., 2023). TiO₂ is widely employed as a photocatalyst in this process. Modifications such as Ag doping have been shown to enhance photocatalytic efficiency, as Ag facilitates electron capture and suppresses recombination, thereby accelerating CO₂ reduction (Ban et al., 2024). Ultrathin TiO₂ nanosheets have also demonstrated superior catalytic activity owing to their increased surface area and reduced charge-transfer resistance (Yin et al., 2024). Furthermore, the brookite phase of TiO₂ exhibits favourable physicochemical stability, which can enhance photocatalytic performance (Chen et al., 2024). Nevertheless, the rapid recombination of photogenerated electron–hole pairs remains a major limitation, leading to low solar energy utilisation and insufficient catalytic efficiency (Zu et al., 2021). To overcome this challenge, the incorporation of suitable electron acceptors is critical for improving photocatalytic activity.

In this study, multifunctional cementitious mortars integrating self-sensing and photocatalytic functions were investigated. Multi-walled carbon nanotubes (MWCNTs) were employed as conductive fillers, while nano-TiO₂ served as the photocatalyst. The internal conductive networks formed by MWCNTs are expected to facilitate the transfer of photogenerated charge carriers. The objective of this research is to evaluate the functional synergy between self-sensing and photocatalytic capabilities and to assess the practical potential of such multifunctional composites. Analytical techniques utilised in this study include thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), UV–visible spectroscopy (UV–Vis), and ion chromatography (ICG).

9.2 Experimental program

9.2.1 Raw materials and coated sand preparation

Yang et al. (Yang et al., 2018) introduced a nano-TiO₂-coated sand (TS) preparation method based on a cyclic coating procedure. To enhance the density of surface hydroxyl groups, river sand was first treated with 0.1 M NaOH solution for 24 h. The residual alkali was then removed by rinsing the sand three times with deionised water. Subsequently, the cleaned sand was immersed in an anatase-phase nano-TiO₂ hydrosol suspension at room temperature for 1 h. After immersion, the sand was separated by filtration and dried at 105 °C for 1 h.

This immersion–filtration–washing–drying cycle was repeated three times to ensure a uniform and stable coating. The final product was subjected to drying at 105 °C for 24 h. The resulting TiO₂-coated sand was characterised, as shown in Fig. 9.1. Energy-dispersive X-ray spectroscopy (EDS) confirmed that the nano-TiO₂ coating was uniformly distributed and strongly adhered to the sand surface.

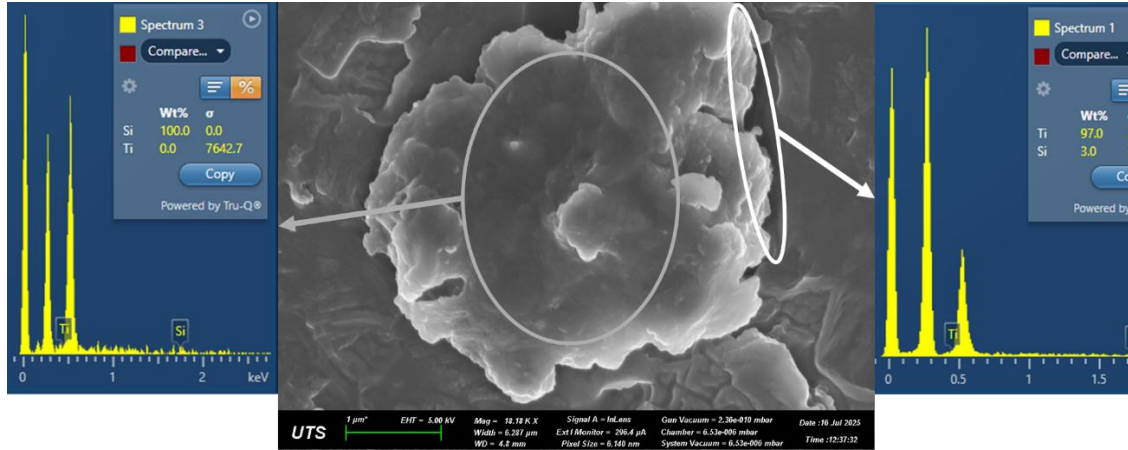


Fig. 9.1 SEM graphic of sand coated with nano- TiO_2 and EDS point scan

General Purpose (GP) cement conforming to AS 3792 (Standard, 2010) was used as the binder. Multi-walled carbon nanotubes (MWCNTs) were incorporated as conductive fillers to modify the electrical properties of the cementitious composites. To determine the optimal MWCNT content for piezoresistive performance, specimens were prepared with varying MWCNT dosages (0.1%, 0.3%, 0.5%, 0.7%, 1.0%, 1.5%, and 2.0% by weight of cement). The percolation thresholds were identified from electrical impedance measurements (Fig. 9.2). Among the tested dosages, 0.7% MWCNTs provided the most favourable balance between conductivity and dispersion and were therefore selected for subsequent experiments. The sand used in the mix was pre-coated with nano- TiO_2 . The detailed mix proportions are summarised in Table 9.1. The primary objective was to investigate whether the incorporation of an internal conductive network could enhance photocatalytic activity. To minimise the effect of ionic conduction within the pore solution, deionised water was used for mixing (Deng et al., 2024). The MWCNTs were first dispersed in deionised water using an ultrasonicator at a constant frequency of 40 kHz for 40 min to ensure uniform dispersion. The resulting suspension was then combined with a dry mixture of cement and TiO_2 -coated sand in a Hobart mixer and stirred for 5 min to obtain a homogeneous slurry. The fresh mortar was cast into $50 \times 50 \times 50 \text{ mm}^3$ moulds, with copper mesh embedded to serve as electrodes for subsequent electrical assessments.

After 24 h of initial curing, the specimens were demoulded and transferred to a curing chamber maintained at 24 °C and 95% relative humidity for 28 days.

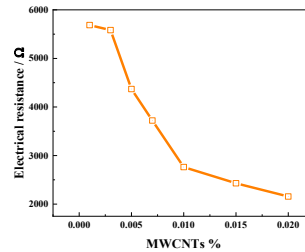


Fig. 9.2 Percolation thresholds of self-sensing cementitious specimens.

Table 9.1 Mixing design of the cementitious composites

Items	Cement/g	S/C	TS/C	M/C	W/C
Ph-1	275	--	2.0	0.7%	0.4
Ph-2				--	
CC	275	2.0	--	0.7%	0.4

Note: S/C: sand/cement ratio; TS/C: nano-TiO₂/cement ratio; M/C: MWCNTs/cement ratio; W/C: water/cement ratio; TS: mixed cementitious composites with/without MWCNTs (Ph-1/Ph-2); control group (CC).

9.2.2 Photocatalytic reaction

The photocatalytic performance of the specimens was evaluated using the experimental setup illustrated in Fig. 9.3. A CO₂ gas cylinder supplied high-purity carbon dioxide to the system. The reaction chamber, a sealed dark box, was equipped with two sensors positioned on either side to monitor and regulate CO₂ concentration. During testing, the concentration of CO₂ was stabilised at 5000 ppm, a level selected to ensure the production of detectable photocatalytic products while also representing a practically relevant concentration for low-grade CO₂ sources such as industrial exhaust gases (Aggarwal et

al., 2021). Once the CO₂ concentration reached 5000 ppm, the gas cylinder was deactivated, and the chamber was sealed to maintain a stable internal environment. The extraction port remained open during CO₂ injection to preserve ambient pressure and was sealed once the concentration had stabilised.

A control test was performed using an identical sealed dark box (reference box) without UV irradiation or specimen exposure to account for potential gas leakage while keeping all other conditions constant. The relative humidity inside the reaction chamber was maintained at approximately 89%. A UV lamp with a wavelength of 364 nm was employed, positioned 2 cm from the specimen surface. While previous studies typically placed UV lamps at ≥ 10 cm to balance irradiation intensity and heat dissipation, this study adopted a shorter working distance to enhance irradiation efficiency (Sharma et al., 2023). Maintaining the minimum 2 cm separation improved irradiance application efficiency, thereby allowing photocatalytic reactions to be measured within a reasonable timeframe. Each specimen was exposed to UV light for approximately 12 h. Following the photocatalytic reaction, the extraction port was reopened to flush residual gases from the system. The recorded results were then corrected by subtracting background values obtained from the reference box, which accounted for potential leakage or environmental variations. Eqs (1-3) illustrated the reduction reaction of CO₂ gas.



Where λ is the wavelength of UV light. After being activated by UV irradiation, electrons and holes that were generated from TiO₂ was explained by Eq (1). The oxidation of

moisture by photogenerated holes leads to the formation of molecular oxygen and protons (H^+) (Ismail & Bahnemann, 2014). Simultaneously, adsorbed CO_2 undergoes electron reduction by capturing a photogenerated electron, generating a carbon dioxide radical anion (CO_2^-). This intermediate subsequently undergoes successive protonation and reduction steps, ultimately yielding formic ions, as shown in Eq. (3) (Dhakshinamoorthy et al., 2012).

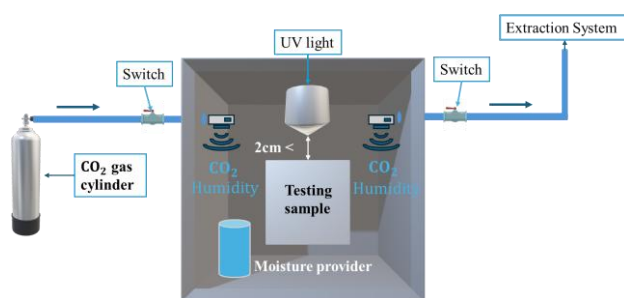


Fig. 9.3 Photocatalytic experiment reaction dark box

9.2.3 Ion chromatography (ICG) test

Ion chromatography (ICG) was employed to quantify low-molecular-weight ionic products generated during the photocatalytic reaction. Liquid samples were collected from the surface of the cementitious specimens by applying 3 mL of deionised water, which was left in contact with the surface for 3–5 min. Owing to the hydrophilic nature of formic ions, they were expected to diffuse readily into the aqueous phase (Rudolph & Irmer, 2022). The surface liquid was then extracted using a pipette, passed through a 0.22 μm syringe filter, and transferred into a clean vial for ICG analysis. For each test, 1 mL of the filtered solution was placed in an ICG sample bottle. Calibration was performed using standard formate solutions prepared in the range of 1–50 ppm. Each ICG run was completed within approximately 35 minutes.

9.2.4 Other methodologies

Other methodologies were same as Chapters 3 and 4.

9.3 Hydration and compressive strength

The hydration heat flow and cumulative heat release profiles are presented in Fig. 9.4 and Fig. 9.5. The maximum heat flow of the Ph-1 specimens reached 0.0414 W/g, nearly 3.9 times lower than the 0.1623 W/g recorded for the control cement (CC) specimens. Similarly, the total accumulated heat release of the Ph-1 specimens was 72.02 J/g, representing a reduction by a factor of 3.5 compared with the CC samples. These results demonstrate that nano- TiO_2 -coated sand significantly altered the early hydration behaviour of the cementitious matrix. This phenomenon can be attributed to a localised reduction in the water-to-cement ratio at the interfacial transition zone (ITZ), where the coated sand exhibits an initial tendency to absorb free water, thereby retarding the early hydration process (Y. Liu et al., 2024). Previous research has also reported that nano- TiO_2 dosages exceeding 0.01% may impede cement hydration due to the coating effect (Wang et al., 2020). The TiO_2 layer deposited on the sand surface can absorb and retain water around sand particles during the initial mixing phase. This localised retention reduces the availability of free water required for early hydration. As hydration progresses, however, the entrapped water is gradually released, functioning as an internal curing agent. The delayed water release regulates hydration kinetics in the early phase and reduces initial heat generation, which explains the lower cumulative heat observed during the early stages of hydration (Jiang et al., 2023; Sun et al., 2017).

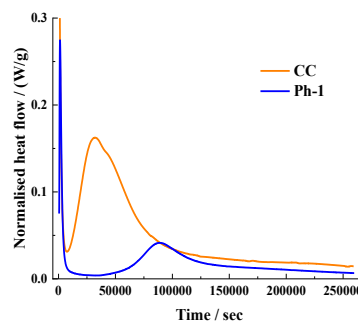


Fig. 9.4 Early hydration heat release flow curve during the initial 72 hours.

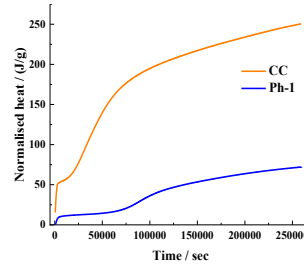


Fig. 9.5 Heat release accumulation during the initial 72 hours.

To evaluate the influence of TiO_2 -coated sand on the compressive strength of cementitious composites, specimens prepared without coated sand were used as the control group (CC). As shown in Fig. 9.6, the compressive strength of the Ph-1 specimens after 28 days of curing was 68.13 MPa, representing a 13.99% reduction compared with the CC specimens, which reached 79.21 MPa. This result indicates that the incorporation of TiO_2 -coated sand adversely affected compressive strength development. To elucidate the underlying mechanisms and phase changes after 28 days of hydration, XRD and TGA analyses were performed. According to the XRD results (Fig. 9.7), the principal crystalline phases detected in the cementitious specimens included C–S–H at 31° , quartz at 20.8° , 23.9° , 26.7° , 29.4° , 36.4° , and 42.4° , tricalcium silicate (C_3S) at 32.1° and 41.2° , portlandite (CH) at 18° , and dicalcium silicate (C_2S) at 32.5° , 54.9° , and 68.2° (Aly et al., 2012; Khaloo et al., 2016). Additionally, reflections corresponding to ettringite (9.1° , 15.8° , and 23°) and calcite (23.1°) were identified. The peak intensities at 9.1° and 15.8° showed no significant differences between the Ph-1 and CC specimens, suggesting that ettringite formation was not substantially influenced by TiO_2 -coated sand. Similarly, the characteristic peaks of C_3S , C_2S , and portlandite (CH) exhibited comparable intensities in both groups, indicating that the coated sand had little effect on the development of major hydration products over 28 days of curing. The presence of muscovite, a common

constituent of river sand, was confirmed by a distinct reflection at 25.4° , along with additional related peaks (Bish & Post, 2018). Anatase TiO_2 was also detected at 25.3° (Zhang & Banfield, 2000), which coincides with the muscovite reflection. To further verify the TiO_2 crystalline phase, additional reflections at 55.0° and 68.7° were examined. The intensity of these peaks in the Ph-1 specimens was higher than that observed in the CC samples, confirming the incorporation of TiO_2 in the coated-sand composites.

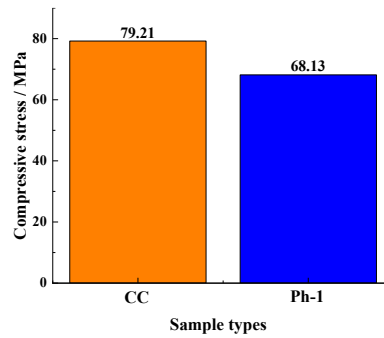


Fig. 9.6 Compressive strength of Ph-1 and CC specimens after 28 days of curing.

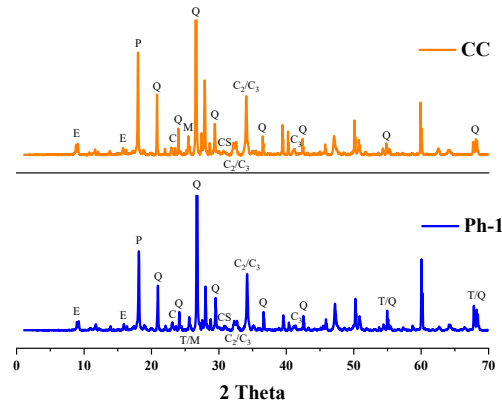


Fig. 9.7 XRD test result of Ph-1 and CC samples after 28 days of curing.

Fig. 9.8 presents the TGA and derivative thermogravimetric (DTG) curves of the cementitious matrix containing TiO_2 -coated sand (TS) after 28 days of hydration. In the temperature range of $50\text{--}200^\circ\text{C}$, the Ph-1 specimens exhibited a mass loss of approximately 3.67%, which was 1% lower than the 4.67% recorded for the CC specimens. Between $390\text{--}500^\circ\text{C}$, the Ph-1 specimens showed a mass loss of 1.61%,

compared with 1.95% for the CC specimens. In the range of 600–800 °C, mass loss for Ph-1 specimens was 1.21%, slightly lower than the 1.34% measured for CC specimens. The reduction in mass loss within 50–200 °C is primarily attributed to the evaporation of physically adsorbed water and the dehydration of C–S–H. These findings suggest that TiO₂-coated sand promotes the development of a denser microstructure by improving C–S–H packing density, thereby reducing water absorption and permeability (Akono, 2020). Previous studies (Jiang et al., 2023; Reddy & Chandana, 2024) have similarly reported that nano-TiO₂ hydrosol coatings on sand substantially decrease mortar porosity, with total porosity reduced by approximately 35%, while the interfacial transition zone (ITZ) becomes denser, exhibiting a 40% reduction in thickness. These microstructural improvements indicate that the coating significantly enhanced the density of the sand–mortar interface, reducing the water absorption capacity of interfacial pores. In the 390–500 °C and 600–800 °C ranges, the lower mass losses of the Ph-1 specimens indicate diminished portlandite production and reduced carbonation compared with the CC specimens. This outcome is consistent with the compressive strength results, confirming that Ph-1 specimens exhibited lower strength performance than the CC specimens.

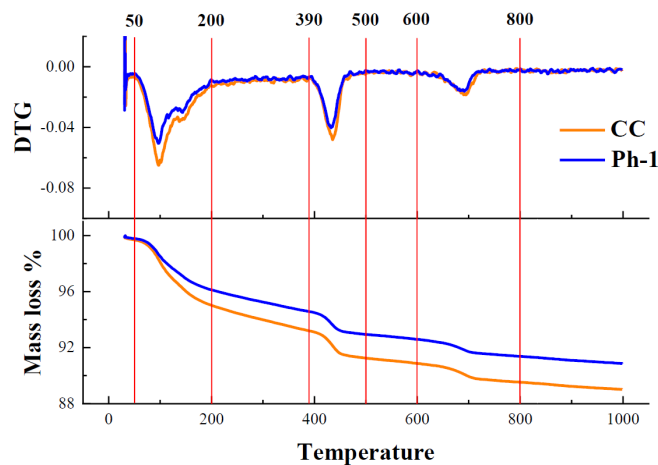


Fig. 9.8 TGA and DTG results of both Ph-1 and CC specimens after 28 days curing.

9.4 Piezoresistivity

The AC impedance test was employed to evaluate the electrical resistance of the cementitious specimens. As shown in Fig. 9.9, the application of nano-TiO₂ coatings significantly increased the electrical resistance of the composites compared with the CC specimens. Under ambient conditions, anatase-phase TiO₂ functions as a semiconductor with inherently low electrical conductivity (Rasheed et al., 2025). When applied as a coating on sand particles, TiO₂ increases interfacial resistivity between conductive phases in the cementitious matrix, thereby elevating overall electrical resistance (Ding et al., 2023). Furthermore, as indicated by the TGA results in Fig. 9.8, the Ph-1 specimens exhibited lower moisture loss than the CC specimens. Because ionic conduction in cementitious composites is strongly dependent on the conductivity of the pore solution, reduced moisture availability likely hindered ion transport, further contributing to the increased resistance. The piezoresistive behaviour of self-sensing specimens incorporating 0.7% MWCNTs was examined through cyclic loading tests. The electrical response, expressed as the FCR, was used to characterise this behaviour. FCR reflects internal pore-structure variations under compressive loading, where deformation induces rearrangement or re-establishment of conductive pathways within the matrix (Wang et al., 2025). Fig. 9.10 shows the electrical responses of the specimens under cyclic loading. The TS-modified samples achieved a maximum FCR of 0.314%, which was approximately 9.2 times greater than the 0.034% measured for the CC specimens. These results indicate that the introduction of TiO₂-coated sand substantially elevated the initial electrical resistance of the cementitious matrix. As suggested by the TGA results, the reduced internal moisture content in the TS specimens implies that ionic conduction had little influence on the baseline resistance. Consequently, the observed improvement in

FCR can be primarily attributed to microstructural modifications and the enhanced sensitivity of the conductive network to mechanical deformation.

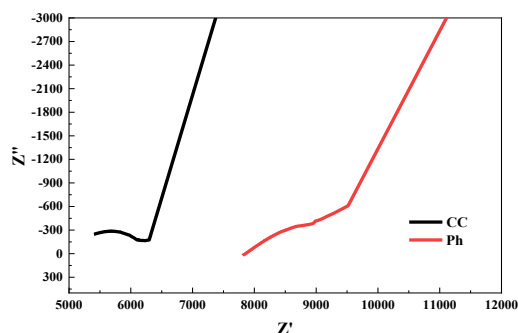


Fig. 9.9 Electrical impedance test of specimens

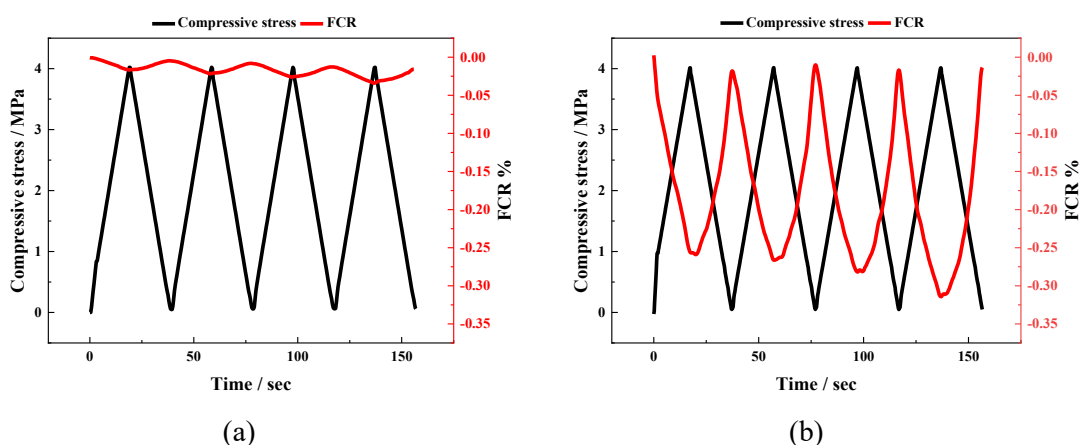


Fig. 9.10 Piezoresistive performance of (a) CC specimens, and (b) Ph-1 specimens

9.5 Photocatalytic performance

During the photocatalytic reaction, the CO_2 concentration initially remained constant and then gradually decreased as the reaction progressed until the UV light was switched off. Fig. 9.11 shows the time-dependent variation in CO_2 concentration recorded by the sensors. To verify the generation of photocatalytic products, FeCl_3 was added to the collected solution containing formate ions, initiating a complexation reaction. The resulting complexes were analysed using UV–Vis spectroscopy. As shown in Fig. 9.12, a pronounced increase in absorbance was observed in the 300–400 nm region, indicating

strong UV–light interactions. Beyond 400 nm, however, the absorbance intensity declined, reflecting reduced light-harvesting capacity in the visible spectrum.

This behaviour can be attributed to the ability of Fe^{3+} ions to associate with simple carboxylic acids such as formic, facilitating ligand-to-metal charge transfer (LMCT) transitions. The elevated UV absorption observed here is consistent with previous studies on Fe^{3+} –carboxylate complexes, particularly those involving acetate and oxalate ligands (Gallard et al., 1999). The detection of formic ions—supported by the reaction pathways shown in Eqs. (1–3) and the UV–Vis spectroscopy results confirm that photocatalytic CO_2 conversion occurred effectively under UV irradiation at 364 nm. ICG was further employed to evaluate the CO_2 conversion efficiency of the specimens. The results are presented in Table 9.2. Formic ions were clearly detected, exhibiting characteristic retention peaks within the 3.0–3.5 min interval (W. Wang et al., 2017). To calculate the transfer rate of CO_2 to HCOO^- , the first step is to calculate the moles of the formic ion, following Eqs. (4-5):

$$C_f = \frac{C_{ft}}{45.02} \quad (4)$$

$$n_f = C_f * V_t \quad (5)$$

Where C_f is the molarity of formic ion, C_{ft} is the tested concentration of formic ion, n_f is the mole of formic ion, and V_t is the tested liquid volume. The molar mass of formic ion is 45.02 g/mol. Given that the pressure in the reaction chamber was maintained at atmospheric conditions, the Clapeyron–Clausius equation was employed in this study to calculate the molar quantity of CO_2 , as presented in Eq (6).

$$PV_c = n_c RT \quad (6)$$

Where P represents the pressure of the reaction box, V_c represents the volume of CO_2 , n_c represents the molar amount, R represents the universal gas constant, and T represents the temperature in K. Lastly, the transfer rate (r) was measured by Eq. (7).

$$r = \frac{n_f}{n_c} \quad (7)$$

According to Eqs. (5–8), the CO_2 transfer rate for the Ph-1 specimens was calculated as 0.00666%, whereas the Ph-2 specimens exhibited a rate of 0.00096%. The incorporation of MWCNTs enhanced CO_2 transfer efficiency by nearly sevenfold. As shown in Fig. 9.11, specimens without MWCNTs displayed no measurable decrease in CO_2 concentration during the reaction, resulting in a markedly lower transfer rate. Although the absolute transfer rates remained below 0.01%, consistent with previous reports (J. Wang et al., 2022), the integration of MWCNTs substantially improved the photocatalytic performance of the composites.

Table 9.2 Formic ion concentration IC test results.

Items	Time / mins	Area / $\mu\text{S} \cdot \text{min}$	Amount / ppm
Ph-1	3.287	1.2599	16.5049
Ph-2	3.257	0.1100	2.3872

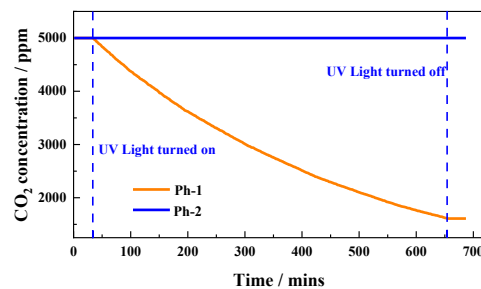


Fig. 9.11 CO_2 concentration change curve during the photocatalytic reaction process of Ph-1 and Ph-2 specimens

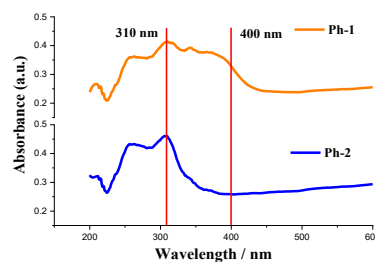


Fig. 9.12 UV-vis spectroscopy test result of formic and $FeCl_3$ reaction products of Ph-1 specimens

9.7 Summary

This study demonstrated that the incorporation of nano- TiO_2 , enhanced the self-sensing performance of the specimens by increasing the initial electrical resistance, a result of the denser internal microstructure. In parallel, the addition of MWCNTs provided efficient electron-conduction pathways. Because the MWCNTs were ultrasonically dispersed in water prior to mixing, they facilitated the formation of continuous internal conductive networks, thereby enabling efficient electron transport. The working principle is illustrated in Fig. 9.13.

As discussed above, the synergistic combination of self-sensing and photocatalytic functions improved catalytic efficiency, underscoring the promising application potential of multifunctional cement-based composites. Future investigations should aim to further enhance CO_2 conversion efficiency through optimisation of nano- TiO_2 activity and by developing more effective strategies for its integration into cementitious systems.

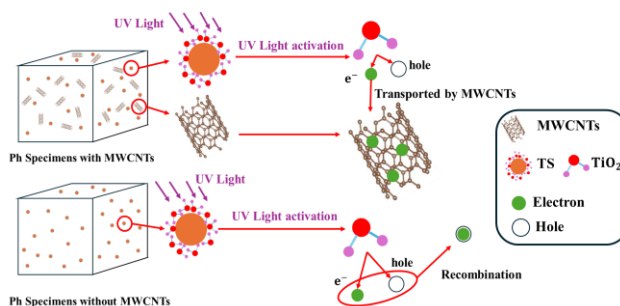


Fig. 9.13 Photocatalytic performance of Ph specimens with/without MWCNTs

Chapter 10: Conclusions and Recommendations

10.1 Enhancement of self-sensing cementitious specimens by bitumen emulsion

1. The incorporation of bitumen emulsion enhanced the water-retention capacity of self-sensing cementitious specimens, thereby enabling an internal curing process.
2. At binder-to-cement (B/C) and water-to-cement (W/C) ratios of 0.25 and 0.30, respectively, the specimens achieved their highest compressive strength. Although the bitumen-containing mixtures exhibited slower early-age hydration, as evidenced by heat evolution data, long-term compressive strength results, supported by microstructural analyses (NMR and TGA), confirmed delayed strength development attributable to continuous internal curing.
3. For the same MWCNT content, the fractional change in resistivity (FCR) values of bitumen–cement (BC) specimens were consistently higher than those of the control cement (CC) specimens. This demonstrates that BC self-sensing sensors are more sensitive to loading variations and, when applied in pavements, can provide superior performance in traffic monitoring compared with CC sensors. Thus, in addition to its conventional role, bitumen emulsion also enhances the piezoresistive properties of BC-based self-sensing sensors.
4. At a 0.25 B/C ratio, the bitumen–cement mixtures exhibited excellent chloride diffusion resistance, particularly when cationic bitumen emulsion was used. The total charge passed was limited to only 356.29 Coulombs, and the corresponding D_{nssm} value was the lowest among ASS, CSS, and CO specimens.

5. The subsurface of the cementitious matrix incorporating cationic bitumen emulsion displayed a smoother surface with fewer pores than that of the ASS specimens. By contrast, the high porosity of the ASS matrix facilitated chloride-ion penetration, leading to greater diffusion compared with CSS specimens.

10.2 SW effects on the piezoresistive performance, self-healing performance, mechanical property with different ion concentrations and multifunctional composites performance

1. The 1:1 blend of seawater (SW) and tap water (50 SW) exhibited the steepest percolation-threshold curve among all mixtures tested. The ion concentrations in the pore solution were measured as 41.25 mmol/L for Na^+ , 26.27 mmol/L for K^+ , and 1.02 mmol/L for Ca^{2+} . These variations in ion concentration directly influenced the percolation thresholds of the piezoresistive specimens.

2. Inductively coupled plasma (ICP) analysis revealed notable differences in Na^+ concentrations across specimen types. In this study, a Na^+ concentration of approximately 41 mmol/L enhanced the piezoresistivity of multifunctional specimens. However, excess Na^+ increased overall electrical conductivity, which in turn exerted a negative effect on piezoresistive sensitivity.

3. The incorporation of SW improved the hydration process of self-sensing specimens, leading to higher compressive strength after 28 days of curing. In addition, SW reduced the water absorption rate, likely due to a reduction in pore volume within the cementitious matrix.

4. Elevated Ca^{2+} and Mg^{2+} concentrations accelerated the self-healing process when crystalline admixture (CA) was used as a healing agent. Energy-dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analyses of the self-healing products confirmed that

ions provided by SW enhanced the self-healing efficiency of multifunctional cementitious specimens.

5. The integration of a self-healing agent promoted the formation of denser composites, thereby increasing the electrical resistance of specimens. Under the combined influence of seawater and crystalline admixture, the self-sensing composites not only retained their semiconducting characteristics but also demonstrated improved sensitivity to resistance changes under applied stress. Consequently, the combined use of self-healing and self-sensing technologies in seawater-based systems presents significant potential for practical applications of multifunctional cementitious composites.

10.3 Influence of pore structure on piezoresistivity

The results showed that specimens containing 9.5 mm coarse aggregates achieved the highest FCR values. This outcome is attributed to the optimal combination of coarse (575 g) and fine (625 g) aggregates, which produced a compact microstructure with an increased proportion of small pores (equivalent diameter 0.1–0.2 mm). These fine pores facilitated the formation of conductive pathways under applied stress. In contrast, specimens incorporating 13.2 mm aggregates contained larger pores, which diminished electrical sensitivity to loading variations. These findings indicate that pore structure exerts a critical influence on self-sensing behaviour, with a higher proportion of small-sized pores contributing to greater sensitivity under mechanical loading.

10.4 Integration of thermochromic and self-sensing function

1. The encapsulated TC coating exhibited excellent solar reflectivity, leading to a reduction in specimen temperature under solar exposure. However, surface cracks were observed as a result of pigment aggregation. Maintaining a lower temperature (<65 °C) promoted the formation of denser C–S–H, thereby enhancing compressive strength.

2. By reducing surface temperature, the TC coating also decreased the rate of water evaporation. The additional retention of pore solution improved ionic conduction, which in turn stabilised the piezoresistive performance of the cementitious specimens.

10.5 Integration of self-sensing and photocatalytic functions

1. The incorporation of TS in the cementitious specimens densified the interfacial transition zone (ITZ), thereby reducing aggregate moisture absorption but simultaneously hindering complete hydration. As a result, the compressive strength after 28 days was lower compared with the control specimens.

2. The presence of nano-TiO₂ also reduced the availability of free water and restricted ion conduction, which increased the initial electrical resistance. This effect, however, enhanced both the stability and the amplitude of piezoresistive responses under cyclic loading.

3. Furthermore, the synergistic role of MWCNTs improved photocatalytic efficiency by facilitating electron transport and suppressing charge recombination. This, in turn, led to greater CO₂ reduction and a higher yield of formic ions.

10.6 Recommendations for future work

1. The commercial deployment of cement/asphalt-based self-sensing sensors remains limited. Future research should carefully consider the influence of ion concentration, moisture content, and pore structure, as these factors critically affect sensing sensitivity and the accuracy of piezoresistive data. Greater emphasis should be placed on developing straightforward and practical methods to regulate these parameters and thereby facilitate field applications.

2. This thesis has demonstrated the potential synergy between self-sensing, self-healing, thermochromic, and photocatalytic functions. The individual components of each

function (conductive fillers, healing agents, thermochromic materials, and photocatalysts) not only operate independently but also mutually reinforce one another. Future investigations should explore more integrated combinations of these functionalities to further improve pavement serviceability and durability.

3. Future research should explore the feasibility of integrating multiple functionalities within a single material system. Achieving simultaneous piezoresistive sensing, self-healing, thermal responsiveness, and photocatalytic performance requires careful optimisation to balance potential synergies and trade-offs. This represents a key step toward the development of next-generation smart pavement materials.

4. Beyond the development of multifunctional composites, the integration of computational algorithms, particularly machine learning approaches, offers significant potential to enhance the efficiency and accuracy of self-sensing systems. Future work should therefore focus on coupling multifunctional materials with advanced algorithms and digital models, thereby enabling more effective real-world applications.

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