Construction Industry Council

Development of Ultra-Ductile Cementitious Waterproofing Rendering by using Recycled Plastic

Research Summary

CONSTRUCTION
INDUSTRY COUNCIL
建造業議會

DEVELOPMENT OF ULTRA-DUCTILE CEMENTITIOUS WATERPROOFING RENDERING BY USING RECYCLED PLASTIC

RESEARCH SUMMARY

Authors

Dr Ivan M.L. SHAM
Nano and Advanced Materials Limited

Prof. Christopher K.Y. LEUNG
The Hong Kong University of Science and Technology

Prof. Kaimin SHIH
The University of Hong Kong

Published by

Construction Industry Council,
Hong Kong SAR

DISCLAIMER

The information given in this report is correct and complete to the best of knowledge of the authors and publisher. All recommendations are made without guarantee on the part of the authors or publisher. The authors and publisher disclaim any liability in connection with the use of the information given in this report.

Enquiries

Enquiries on this research may be made to the CIC Secretariat at:

CIC Headquarters
38/F, COS Centre, 56 Tsun Yip Street, Kwun Tong, Kowloon

Tel.: (852) 2100 9000

Fax: (852) 2100 9090

Email: enquiry@cic.hk

Website: www.cic.hk

FOREWORD

Water seepage problems in buildings have been a concern for owners and governments worldwide. In Hong Kong, over 25,000 complaints were received in 2010 and the common causes of water seepage are found to be the leakage of pipes and seepage through roofs or external walls. For reinforced concrete structures, applying waterproof coating is the most common mitigation measure to prevent water seepage but this method is limited by weather and the conditions of concrete surface.

Development of a new generation waterproof material become an urgent task. Hence, the Construction Industry Council (CIC) engaged Nano and Advanced Materials Institute (NAMI) to conduct a research on the topic.

The research team has successfully developed a waterproof rendering made of engineered cementitious composite. The rendering made use of polyethylene terephthalate (PET) which is recycled from plastic bottles. It is well known that there are hundreds of tons of plastic bottles disposed of as landfill in Hong Kong each year. Recycling these plastic bottles is significant for the sustainable development of the city. The innovative rendering demonstrates good tensile capacity and a remarkable water repelling effect when the cementitious matrix is well designed.

The research work presented in this report was funded by the CIC Research Fund, which was set up in September 2012 to provide financial support to research institutes/construction industry organizations to undertake research projects which can benefit the Hong Kong construction industry through practical application of the research outcomes. CIC believes that research and innovation are of great importance to the sustainable development of the Hong Kong construction industry. Hence, CIC is committed to working closely with industry stakeholders to drive innovation and initiate practical research projects.

The research team is led by Dr Ivan SHAM. The project cannot succeed without the dedicated effort of the research team. I would like to give thanks to all who took part in this valuable work. The CIC would also like to collaborate with NAMI and industry stakeholders to commercialize the innovative product.

Ir Albert CHENG
Executive Director of Construction Industry Council

PREFACE

This research project focused on the development of an ultra-ductile cementitious rendering for waterproofing. The developed waterproof rendering can repel water and has ultimate tensile strain up to 2%. Water seepage is a common issue of buildings in Hong Kong. There are several types of waterproof coatings. Polymeric waterproof coatings perform low water permeability but they are either brittle or limited on dry concrete substance surface during application. Some of high quality polymeric waterproof coatings require a primer to improve bonding between concrete substance and coating, incurring additional time and disruption to occupants. Cementitious-based waterproof coating allows water vapour to move in and out and reduce the possibility of bubble formation. However, the allowable elongation is limited. In view of the above, this research on an innovative waterproofing material that can combine the advantages of high water permeability resistance and high ductility can meet the urgent demand in the industry.

According to the report from the government of Hong Kong SAR, about 206 tonnes of plastic bottles are dumped to landfills every day in 2014. The plastic bottles are mostly made of polyethylene terephthalate (PET), which is thermoplastic that can be melt and reformed to other shapes. One potential application of the recycle plastic bottles is to replace the man-made polyvinyl alcohol (PVA) fibre, which is commonly used in engineered cementitious composite to form the ultra-ductile cementitious rendering for waterproofing.

In this project, a waterproofing rendering made of Engineered Cementitious Composites (ECC) has been developed. Recycled polyethylene terephthalate (PET) fiber and polyvinyl alcohol (PVA) fiber are incorporated in the ECC. PVA fibers have been widely studied in the literature while PET fibers which are made from extrusion of plastic bottle have not been examined for ECC. Therefore, the mechanical properties of recycled PET fiber were optimised. Furthermore, advanced surface modification techniques were adopted to improve the fibre dispersion into the cementitious matrix and form a uniformly distributed network. The bond strength between the fibre/matrix interfaces was optimised to achieve the multiple-cracking ultra-ductile behaviour.

For the test results, the tensile capacity of ECC is over 2%, and the maximum crack width is less than 300 μm at 1% tensile strain. The ECC also presents water repelling effect and achieves a coefficient of water permeability as low as 4.46×10^{-10} cm/s. These results suggest ECC can be very useful for waterproofing applications. Furthermore, by modifying the mix design, ECC can achieve proper workability and good adhesion with concrete substrate, which can facilitate its application on walls. Both lab trial and field trial are conducted to demonstrate that the ECC rendering can work in practical situations, and it has large potential market as they are cost effective.

This project not only developed a new generation of waterproof rendering material, but also created a local market for the recycle plastic bottles in Hong Kong that can relief the pressure on the limited landfill space. With the successful completion of the project, it demonstrated to the industry about the practical application of waste utilisation.

Dr Ivan M.L. SHAM
Nano and Advanced Materials Limited

RESEARCH HIGHLIGHTS

An ultra-ductile cementitious waterproofing rendering reinforced with recycled PET fibers has been comprehensively developed in this project. The characterization and surface treatment of the PET fibers were conducted. Tensile performance, water contact angle, drying shrinkage, and water permeability were studied, followed by the parametric study and optimization of matrix formulation. The properties and dosage of PET fibers were fine-tuned to increase alkaline resistance and bond strength. Bending behavior, workability and pull-off adhesion were also evaluated. The key findings are highlighted in the following paragraphs.

The first section of the study focused on the development of surface modification techniques of recycled PET fibers and their emerging applications have paved the way to the substantial usage of PET waste in cementitious materials. Therefore, a literature review on various surface modifications was firstly conducted as summarized below.

i. The surface roughness of fiber has significant positive effects on the fiber pullout behaviour. Surface physical indentation of recycled PET fiber is one of the viable methods to improve the mechanical performance of fiber-reinforced cementitious composite. Recycled PET fiber with embossed surface exhibits better adhesive performance compared to the smooth one. Other tailor-made surface geometries of PET fibers may be explored to evaluate their effects on the performance of cementitious composite.

ii. Chemical modification methods, including (a) grafting of the fiber surface by alkali or plasma treatment; and (b) deposition of small particles on the fiber surface such as maleic anhydride grafted polypropylene (mPP) or silica fume may improve the wettability of recycled PET fiber surface and its alkaline resistance in the cement matrix. The mechanism is surface hydrophilization, surface activation and the introduction of surface polar functional groups. Investigation of the appropriate degree of chemical treatment such as the concentration of reagents and the treatment time is needed for achieving the required alkaline resistance and desirable interfacial bond between PET fiber and cementitious matrix.

iii. The incorporation of recycled PET fiber in cementitious materials is advantageous. The merits of recycled PET fiber-reinforced cementitious composites include high ductility, enhanced fracture toughness, increased tensile strength and deformation capacity, good crack control and crack bridging properties. The surface modifications of recycled PET fiber can contribute significantly to the physical and mechanical performances of the cementitious composites. Further detailed research work to formulate the optimum application strategy of recycled PET fiber in cementitious materials is urgently needed.

The next section of the study is to address the development of matrix mixture in terms of water contact angle, drying shrinkage, tensile performance and water permeability. Key findings are summarized as follows:

i. A matrix with very good waterproofing ability and low drying shrinkage is obtained by introducing 0.15%, 0.3% and 0.45% water waterproofing agent (WPA). The contact angle changed from 61 degrees to 120 degrees or higher. In addition, test results indicate that WPA has no adverse influence on the compressive strength.

ii. The drying shrinkage is controlled by adding shrinkage-reducing agent (SRA). The shrinkage value could be reduced by 18% and 29% respectively with the addition of 1.0% and 2.0% of SRA. Beside using SRA, replacing a small percentage of the Portland cement (OPC) by calcium sulfoaluminate (CSA) cement is also applied to reduce the drying shrinkage. The lowest shrinkage was achieved with the blended ratio of SAC/OPC at 1/49.

iii. The effects of sand-to-binder ratio and SAC/OPC ratio on the tensile performance of the mortar were investigated. Results showed the optimized blended ratio of sand-to-binder was 0.2 and SAC/OPC at 1/49

iv. Water permeability test has been conducted, which indicated that the introduction of very small amount of WPA could effectively improve the water permeability resistance, and excellent water permeability resistance was observed with the WPA content no less than 0.30%.

Having developed the matrix mixture, the next focus would be on the mechanical and alkaline resistance properties of PET fiber used in the mortar. Key findings are summarized as follows:

i. PET fibers with varied diameter ranging from 12.5 to 38 μm were investigated in details with respect to the mechanical properties, dispersity in cementitious matrix and alkaline resistance. Finally, 38 μm PET fiber was chosen.

ii. The surface modification with alkaline treatment and silane coating was applied on PET fiber, and surface morphologies of untreated and treated PET fibers were studied by SEM, their surface compositions and functional groups were investigated by FTIR spectroscopy and XPS.

iii. The bonding adhesion was measured by single fiber pill-out tests. The results suggested that the suitable surface modification method for PET sheets was pre-treated with 5 wt% NaOH solution for 3 hours, and then treated with 3 wt% KH570 silane solution for 4 hours. The ultimate bond strength of treated PET fiber was more than three time of plain PET fiber, which would further improve the property of rendering.

In addition, the mechanical properties of rendering reinforced with PVA and recycled PET fibers have been comprehensively studied. The total fiber volume was set at 2%, and parametric study on various proportions of PVA and PET fiber was conducted.

i. The compressive strength of all the mixtures achieved over 30 MPa at 28-day age, and 48 MPa or above after accelerated aging curing, the influence of different fiber combination was insignificant.

ii. Tensile performance of rendering with pure PVA fiber was the best but satisfactory mechanical performance was achieved with up to 50% of PVA fibers replaced by recycled PET fibers, in which the matrix provided tensile capacity(strain) of 2.16% and tensile strength of 3.63 MPa. When pure PET fibers were used, the ductility of the mixes with treated PET fibers was 30% higher than those with untreated PET fibers.

iii. Bending test results showed similar trend to the direct tensile performance, that when more PVA fibers were replaced by PET fibers, the bending behavior became poorer. From the test results, the specimens consisting of 50% of PVA fibers and 50% PET fibers, had comparatively high bending ductility, i.e. 2.50×10^{-3}/cm, which was only 25% less than that of pure PVA fibers, revealing that the amount of PET fibers could be further increased to replace PVA fibers

In the last section, workability and pull-off adhesion between the rendering and concrete substrate were analyzed. The matrix mixture was further modified to achieve a sufficient flow table diameter around 150 mm and bond strength larger than 1 MPa.

CONTENTS

1 INTRODUCTION

1.1 Background

Water seepage is a common problem in reinforced concrete structures. There are a dozen of reasons of water seepage. For example, crack formation in concrete, inferior concrete quality, poor joint connection of water pipes, deterioration of pipe material and many other reasons. It is more critical for basement retaining structures and the like, because there is always a driving force by water head difference. Nowadays, the design of those structures is through limiting the crack size according to the serviceability limit state. However, excessive steel reinforcement is used more than the requirement of ultimate limit state to limit the crack size under service load. The consequences are that (i) the cost of the structures is significantly increased and (ii) it creates difficulty during construction since excessive reinforcement makes the concrete compaction difficult.

The most common mitigation measure is to apply waterproof coating to prevent water seepage. Typically cement-based waterproof rendering is brittle and can be cracked easily by any crack propagation in the concrete substrate or any differential displacement at the joint. For epoxy based waterproof coating, the volatile organic compound level inside is relatively high and is too brittle to be cracked as the cementitious counterpart. The high-end waterproof product, such as polyurea/polyurethane based waterproof coating, has excellent tensile strength and ductility, but it cannot be applied on wet surface. Hence, the application of such waterproof coating is limited by weather and the conditions of concrete surface. In most cases, primer is also necessary for good quality of waterproof coating and the manpower cost is therefore increased further.

An ultra-ductile ECC has been proposed for decades (Li, 2002; Li & Leung, 1992; Li & Wu, 1992). At the ultimate state under uniaxial tension, the strain of particular ECC can reach 3% to 7%, with crack width around 60 μm (Li, 1993). With properly designed matrix, even tighter crack widths, as low as 20 μm, have been achieved (Yang & Li, 2007). In conventional ECC, only polymeric fibers such as polyethylene (PE) or PVA have been employed, as high ductility can be achieved with a relative low fiber volume fraction (about 2%). Although ECC possesses many good properties, the high cost of PE or PVA fibers limits the wide applications of ECC in civil engineering. Therefore, it is essential to have an alternative approach to reduce the overall materials cost for more applications.

According to a report from the government of Hong Kong (Hong Kong Environmental Protection Department, 2015), about 206 tons of plastic bottles are dumped to landfills every day in 2014, which has created imminent burden on the limited space of landfill sites. More than two third of the plastic bottles in Hong Kong are made of PET, which is thermoplastic that indeed can be melt and reformed to other shapes. It is desirable to figure out an economical way for the recycle plastic bottles through creating local demands. Hence, the Construction Industry Council (CIC) commissioned Nano and Advanced Materials Limited (NAMI) to launch a research project entitled “Development of Ultra-Ductile Cementitious Waterproofing Rendering by using Recycled Plastic”.

1.2 Aims and Objectives

The objective of this project is to explore the potential application of the recycled PET fibers to replace the PVA fiber in the ultra-ductile cementitious rendering for waterproofing.

1. To develop a fiber reinforced cementitious matrix for waterproofing rendering. Formulation of matrix is developed to achieve water contact angle larger than 90^o, reduce the shrinkage and enhance the water permeability resistance.
2. To characterize and test the mechanical properties of various fibers, including recycled PET from different manufactures and PVA fibers. And investigate the durability of recycled PET in alkaline environment.
3. To propose a suitable surface treatment of recycled PET fibers that can enhance the fiber/matrix bonding and improve the alkali resistance.
4. To conduct single fiber pull-out test of the treated recycled PET and PVA fiber to examine the adhesion between fiber and cementitious matrix.
5. To evaluate the mechanical properties of the hybrid PVA/PET fiber ECC with totally 2% fiber volume fraction, including direct tension test, bending test and compression test. Mechanical properties with standard curing and accelerated ageing curing are reported. Digital Image Processing method is adopted during the direct tension test to determine the average crack width at various strain levels, which are of relevance to durability.

1.3 Scope

This study aimed at developing an ultra-ductile cementitious rendering for waterproofing application. Recycled PET fibers made from waste bottles were incorporated into the rendering to replace PVA fibers. A literature review was carried out on surface modification of PET fiber used in construction materials. Mechanical properties of the PET fibers were characterized, and a suitable surface modification method was applied to the PET fiber to improve the matrix/fiber interface.

The cementitious matrix was developed to achieve high ductility, high water resistance, low dry shrinkage and water repelling. Therefore, compression, tension and bending tests, contact angle measurement, shrinkage measurement and water permeability tests were conducted. Moreover, to demonstrate the applicability of the developed ECC rendering, two field trials were conducted.

2 RESEARCH METHODOLOGY

An ultra-ductile ECC has been proposed for many years. Instead of the brittle behaviour as convention cementitious material, for which a single large crack is formed, ECC can spread large tensile strain into many fine hair cracks with the size of less than 0.06 mm (Figure 1). Figure 2 shows typical stress-strain relationship from the direct tensile test of ECC. The ultimate tensile can be up to 1 to 3% when 1~2 % volume fraction PVA fibers are used. However, the cost of PVA fibers is relatively expensive and thus limits its applications in the industry.

Figure 1 Example of multiple crack behaviour of ECC from the Hong Kong University of Science and Technology (HKUST)

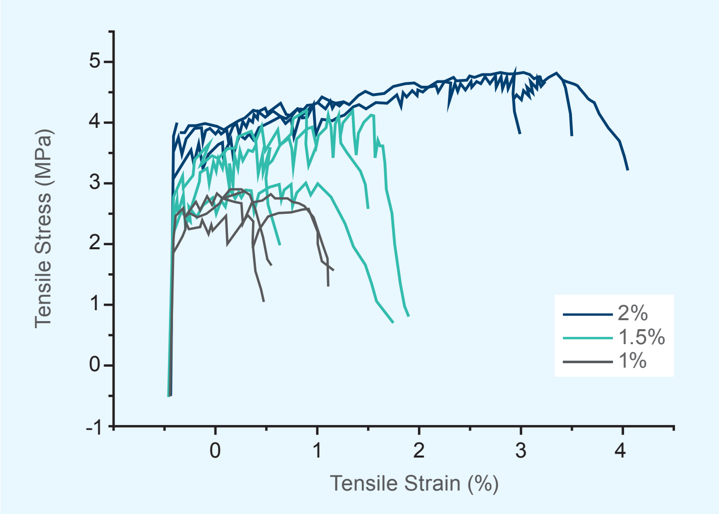


Figure 2 Typical stress-strain relationship of the direct tensile test of ECC with different volume fraction of PVA fibre from the HKUST

According to the report from the government of Hong Kong SAR, about 206 tonnes of plastic bottles are dumped to landfills every day in 2014. The plastic bottles are mostly made of PET, which is thermoplastic that can be melt and reformed to other shapes. One of the potential applications of the recycle plastic bottles is to replace the expensive PVA fibre to form the ultra-ductile cementitious rendering for waterproofing. To develop the ultra-ductile cementitious rendering for waterproofing application, the following techniques are explored.

2.1 Optimization of PET Fibre by Recycled Plastic Bottles

The collected waste plastic bottles, which are made of PET, are cleaned and sorted. Then, they are crushed into small pieces. The crushed PET is melt and extruded into continuous filament as shown in Figure 3. The continuous filament is chopped into discrete fibre with prescribed length. To improve the mechanical properties of the recycle PET filament, the extrusion process should be iterated.

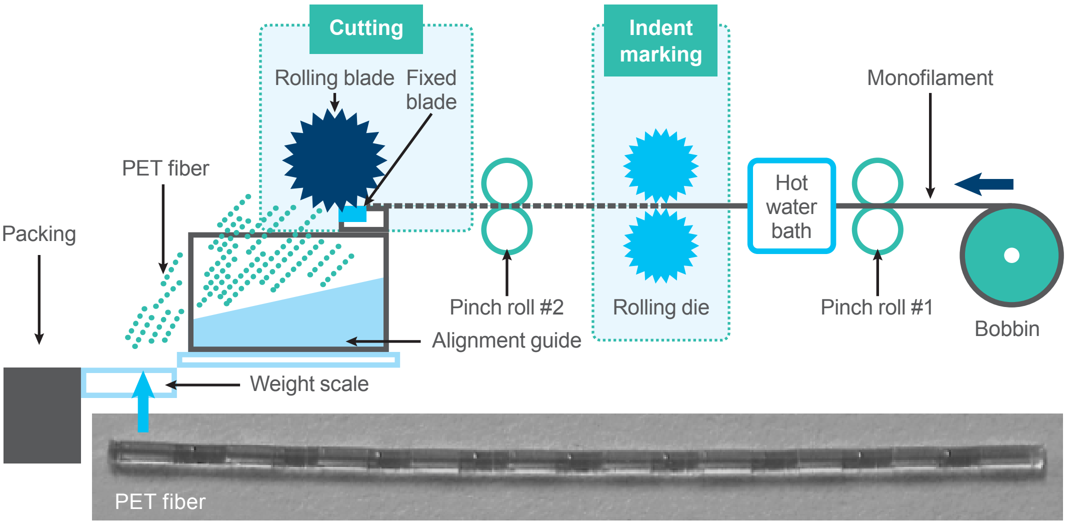


Figure 3 Apparatus of the indent marking and cutting of PET fiber (Okubo, & Fukui, 2007)

It is important to disperse the discrete PET fibres into cementitious matrix evenly. Hence, surface treatment is needed to improve the dispersion of the discrete PET fibre and to enhance the bond strength at the fibre/matrix interface. If the bond strength is too weak, the frictional force is not large enough to stabilise and bridge the crack. On the contrary, if the bond strength is too strong, the fibre cannot slide relative to the matrix before breakage and hence, reduce the matrix ductility. The bond strength is dependent on the fracture toughness of the cementitious matrix as well as the mechanical and the geometrical properties of the discrete PET fibres. The basic principle is to absorb the energy release by crack propagation from frictional force (Leung, 1996). Hence, the key objective of this task is to investigate the conditions of extrusion on the fibre geometry and how the surface treatment affects the bond strength.

2.2 Design of the Mix of Ultra-ductile Cementitious Rendering

The cementitious matrix consists of ordinary Portland cement, calcium sulfoaluminate cement, limestone powder, quartz sand and fly ash. By replacing PVA fibre by PET fibre, for which the mechanical properties are poorer than PVA fibre to achieve multiple-crack ultra-ductile behaviour, the fracture toughness of the cementitious matrix has been reduced so that the fibre can stabilise crack propagation. The reduction of fracture toughness can be achieved by two approaches. The first approach is to increase the water-to-binder ratio. However, it increases the porosity of the cementitious matrix and has detrimental effect on the cohesiveness, and causes significant segregation and bleeding. The second approach is to reduce the binder content, i.e. higher inert filler content. It can reduce the cementitious content and hence the cost. In addition, it helps reduce the dry shrinkage.

When the fracture toughness of the cementitious matrix is reduced, the cementitious matrix becomes weaker and affects the water transportation inside the matrix. As the proposed ultra-ductile cementitious rendering is used for waterproofing, it is necessary to reduce the water permeability. This would be the balance between water permeability and fracture toughness. One possible solution is to change the cementitious matrix from hydrophilic to hydrophobic. Figure 4a shows water spreads on the hydrophilic conventional cementitious matrix, surface. When the cementitious matrix is hydrophobic, water is kept at droplet form and moves away easily (see Figure 4b). Hence, changing the cementitious matrix from hydrophilic to hydrophobic, can prevent water spreading, out and minimise the contact surface area with cementitious matrix. A silane based waterproofing agent is applied to achieve this goal. The dosage of the waterproofing agent is optimized in terms of hydrophobicity of the surface and water permeability.

(a) Normal (b) Hydrophobic surface

Figure 4 Example of hydrophilic (left) and hydrophobic (right) cementitious matrix

The hydrophobicity of the cementitious matrix can be quantified by water contact angle. A syringe pump is used to squeeze a prescribed quantity (1 μL) of distilled water on top of the smoothed cementitious surface. The angle between the tangent of water droplet and the substrate surface that is inside the water droplet is measured. If the water contact angle is larger than 90°, the surface is hydrophobic as shown in Figure 5, for which the contact angle is about 105°.

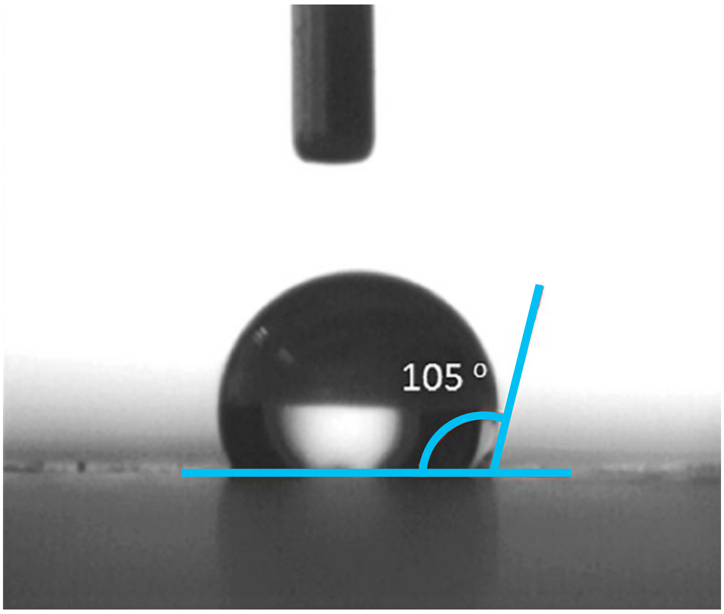


Figure 5 Measurement of water contact angle

To quantify the water permeability, falling head test is performed. Sample is oven dried to remove all moisture inside and it put into an apparatus as shown in Figure 6. The water in the small diameter pipe provides head difference to drive water to penetrate into concrete. The coefficient of water permeability (k) can be estimated using the equation (Wang *et al.,* 1997):

$$k_w = \frac{a}{A}\frac{L}{t}\ ln\ \frac{h_0}{h_f}$$

where a is the diameter of the small pipe, L is the height of the sample, A is the cross-sectional area of the sample, h_0 is the initial head difference, h_f is the final head difference and t is the time taken. The coefficient of water permeability is measured and compared with different mix proportion of the cementitious matrix from hydrophilic to hydrophobic.

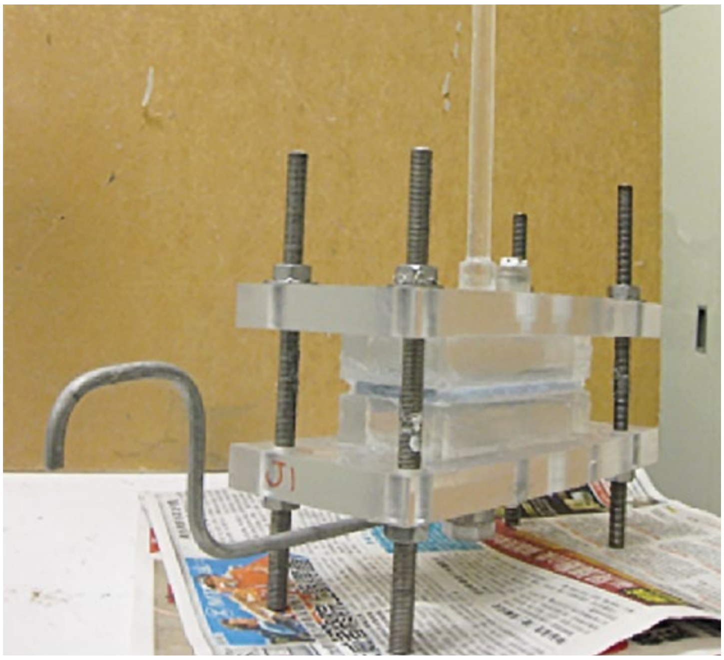


Figure 6 Experimental set up of water permeability test

The ductility of the proposed ultra-ductile cementitious rendering is verified by direct tensile test in MTS-810. The experimental set up and the specimen are shown in Figure 7. To prevent crushing at the gripped region in the testing machine, the specimen is protected by carbon fibre reinforced polymer sandwiched by two aluminium plates. The displacement is measured by two linear variable differential transformers (LVDTs) at two sides. Any significant discrepancy between the two LVDTs indicates eccentricity and the result will be discarded.

Figure 7 Experimental set up of direct tensile test

Dry shrinkage can induce crack and dramatically increase the coefficient of water permeability. By (Zhang *et al.,* 2009), ECC may lose the ultra-ductility due to dry shrinkage. The dry shrinkage of the proposed ultra-ductile cementitious rendering is examined. The sample is stored in a room with regulated temperature and humidity at 23^{o}C and 55%RH, respectively (Figure 8).

(a) Samples for dry shrinkage test (b) Test room for dry shrinkage test

Figure 8 Experimental set up of dry shrinkage test

3 RESEARCH FINDINGS AND DISCUSSION

3.1 Matrix Development of Cementitious Rendering

The matrix for the waterproofing ECC was optimized as listed in Table 1. The matrix materials used in this study included ordinary Portland cement (OPC), calcium sulfoaluminate cement (SAC), fly ash (FA), lime stone powder (LSP), silica sand, waterproofing agent (WPA), shrinkage-reducing agent (SRA) and super-plasticizer (SP). Using high volumes of fly ash results in the reduction of steady-state crack width, which is beneficial to waterproofing as well as the long-term durability of the structure. The mix was modified for the waterproofing application in terms of water repelling effect and shrinkage reduction.

Table 1 Mix proportions for studying matrix design (by weight)

Mix ID	Binder (B)	S	W	SRA/(B+S) [%]	WPA/(B+S) [%]	SP/B [%]	Fiber [vol.%]	
							PVA	PET
M1	1	0.2	0.3	0	0	0.22	0	0
M2	1	0.2	0.3	0	0.15	0.22	0	0
M3	1	0.2	0.3	0	0.3	0.22	0	0
M4	1	0.2	0.3	0	0.45	0.22	0	0
M5	1	0.2	0.3	0.5	0.3	0.22	0	0
M6	1	0.2	0.3	1	0.3	0.22	0	0
M7	1	0.2	0.3	1.5	0.3	0.22	0	0
M8	1	0.2	0.3	0	0.3	0.22	0	0
M9	1	0.2	0.3	0	0.3	0.33	0	0
M10	1	0.2	0.3	0	0.3	0.44	0	0
M11	1	0.2	0.3	0	0.3	0.55	0	0
M12	1	0.2	0.3	0	0.3	0.37	2	0
M13	1	0.2	0.3	0.5	0.3	0.37	2	0
M14	1	0.2	0.3	1	0.3	0.37	2	0
M15	1	0.2	0.3	1.5	0.3	0.37	2	0
M16	1	0.4	0.3	1	0.3	0.37	2	0
M17	1	0.8	0.3	1	0.3	0.37	2	0
M18	1	0.2	0.3	1	0.3	0.48	2	0
M19	1	0.2	0.3	1	0.3	0.59	2	0
M20	1	0.2	0.3	1	0.3	0.7	2	0
M21	1	0.2	0.3	1	0	0.37	1	1
M22	1	0.2	0.3	1	0.15	0.37	1	1
M23	1	0.2	0.3	1	0.3	0.37	1	1
M24	1	0.2	0.3	1	0.45	0.37	1	1

Note:
OPC - Ordinary Portland cement CEM I 52.5N; **SAC** - Calcium sulfoaluminate cement; **FA** - Fly ash (Class F); **LSP** - Limestone powder with the nominal diameter of 58 μm; **S** - Silica sand with the nominal diameter from 120 μm to 212 μm; **W** - Water; **SRA** - MUNZING Metolat P872 shrinkage-reducing agent; **WPA** - WACKER Powder D silane based waterproofing agent; **SP** - GRACE AVDA 105 superplasticizer; **PVA** - KURARAY K-II REC15 polyvinyl alcohol fibers; **PET** - recycled Polyethylene terephthalate fibers.

Water repelling effect

A silane-based WPA was select to enhance the hydrophobic characteristics of the rendering; various amount of WPA, 0.15%, 0.3% and 0.45% were studied. The contact angles of mortars with water on flat non-casting surfaces were measured and the results were listed in Table 2. The results show that 0.15% can already result in high contact angle. 0.30% is used to make sure hydrophobicity can be achieved.

Table 2 Contact angles of mortar specimens with water

Dosage of S-WPA (%)	0	0.1	0.3	0.5
Contact angle (degree)	63°	116°	118°	121°

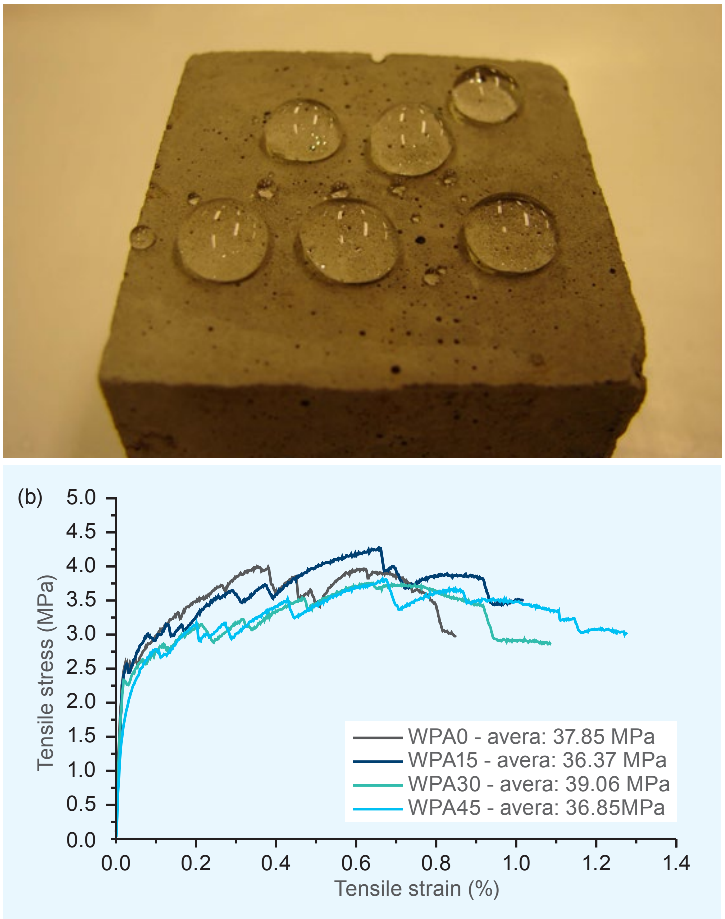
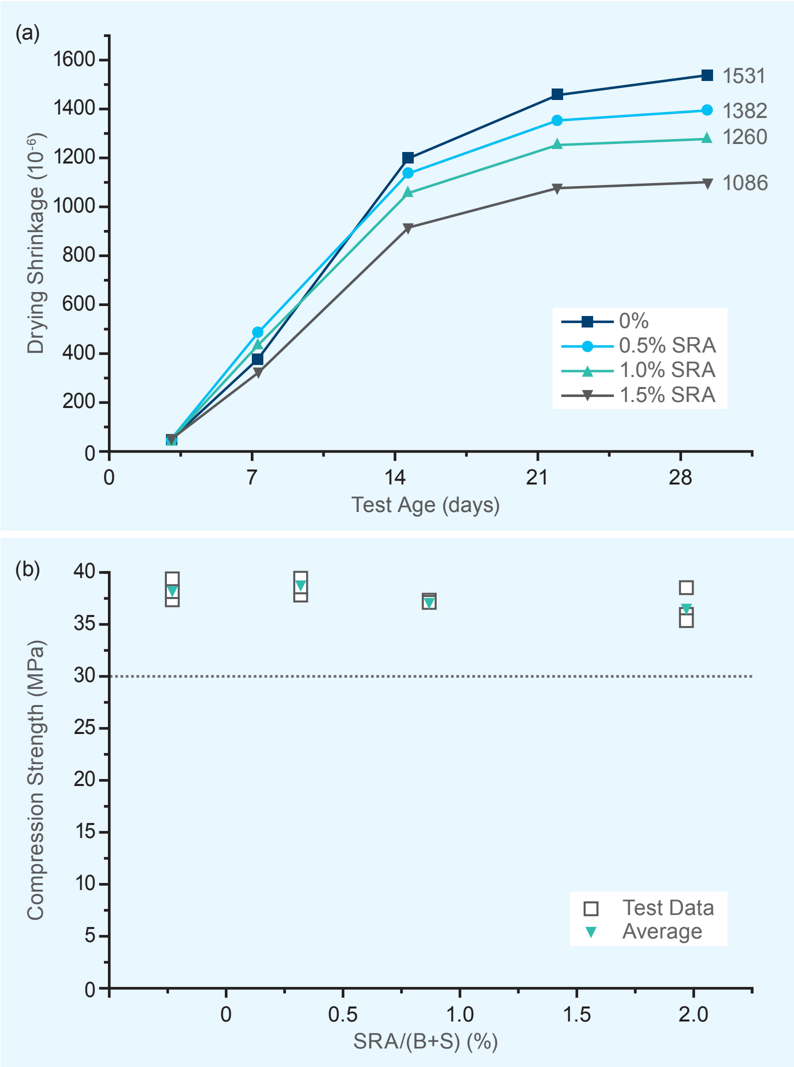


Figure 9 (a) Water repealing effect of ECC. (b) Influence of the WPA content on the tensile performance of the rendering

Drying shrinkage control

Both expandable SAC and SRA were used in reducing the shrinkage, and their effectiveness was systematically studied as shown in Figure 10. The shrinkage value can be reduced by 18% and 29% respectively with the addition of 1.0% and 2.0% of SRA. Figure 10b shows high amount of SRA would significantly decrease the compressive strength. As shown in Figure 10c, replacing some OPC with SAC is beneficial as the lowest shrinkage was achieved with the blended ratio of SAC/OPC of 1/49. Figure 10d shows that SRA still works well at the blended ratio of SAC/OPC of 1/49. The addition of SRA at 1.0% can significantly reduce the shrinkage value, while further increase of the SRA amount will not have significant effect. Balancing among compression strength, shrinkage-reducing effect and cost, a SRA-to-binder and sand ratio of 1.0% is adopted.



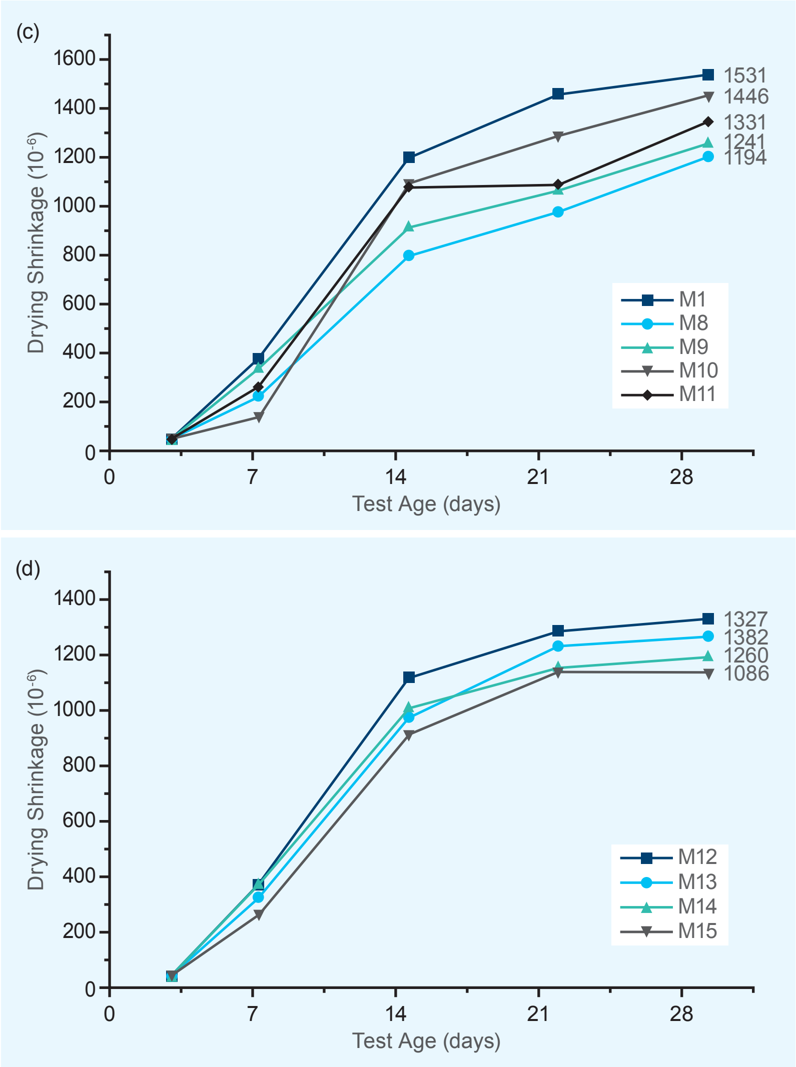


Figure 10 Effects of SRA content on (a) drying shrinkage and (b) 28-day compressive strength of mortars.
(c)(d) Effects of SAC and SRA content on drying shrinkage of composites

Tensile performance

The influences of the sand-to-binder ratio on the tensile performance at the 28-day age were investigated in Figure 11a. Stable tensile performance with the tensile ductility more than 4% is achieved with a sand-to-binder ratio of 0.2. Further increasing the sand-to-binder ratio would result in poor tensile ductility, a sand-to-binder ratio of 0.2 is chosen for further study. In addition, the influences of the SAC/OPC ratio on the tensile performance were investigated. As shown in Figure 11b, all the mixes show similar first cracking strength, ultimate tensile strength and excellent tensile ductility. However, further investigation on the crack pattern indicates that more SAC results in a wider crack opening under tension as shown in Figure 12. This finding confirms the selection of the blended ratio of SAC/OPC of 1/49.

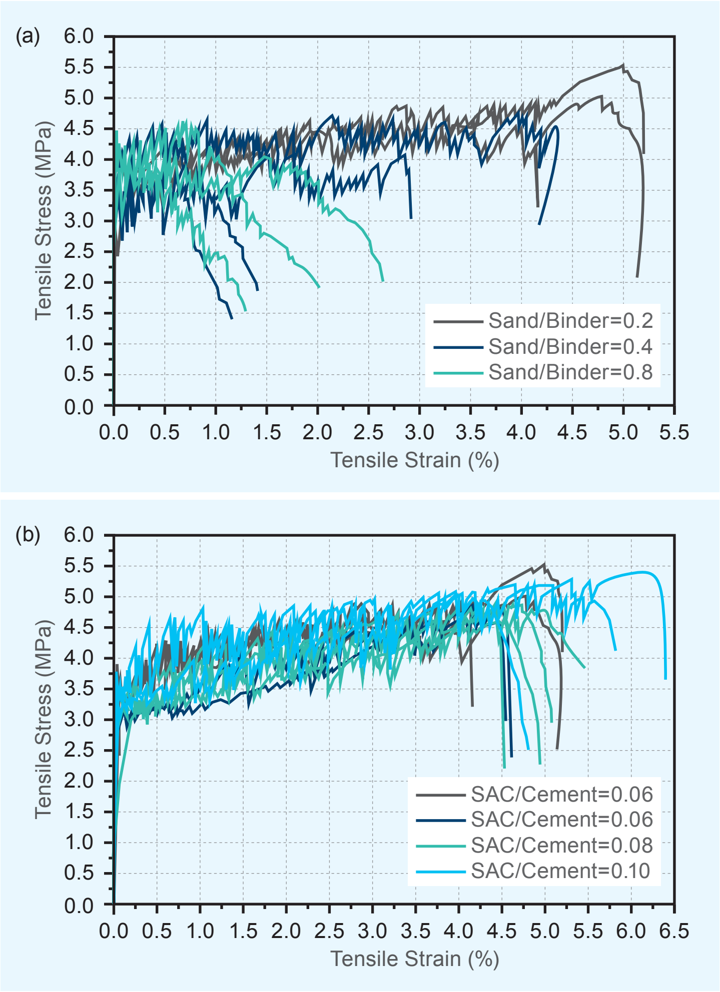


Figure 11 Effects of (a) sand-to-binder ratio and (b) SAC/Cement ratio on tensile performance of composites

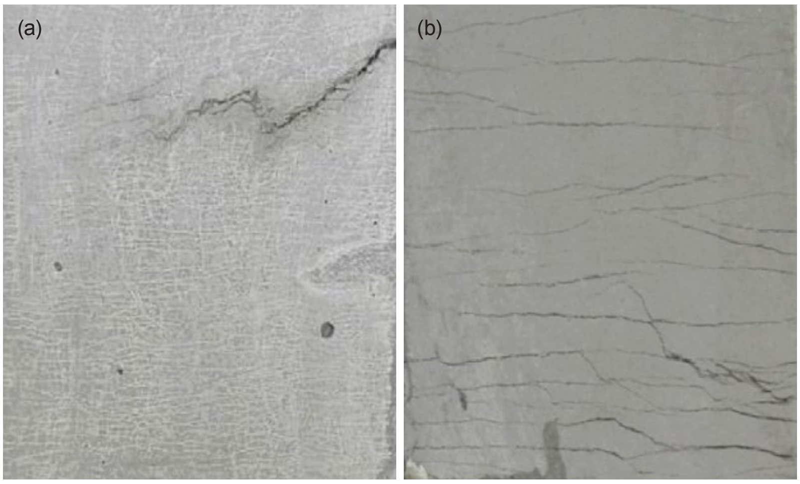
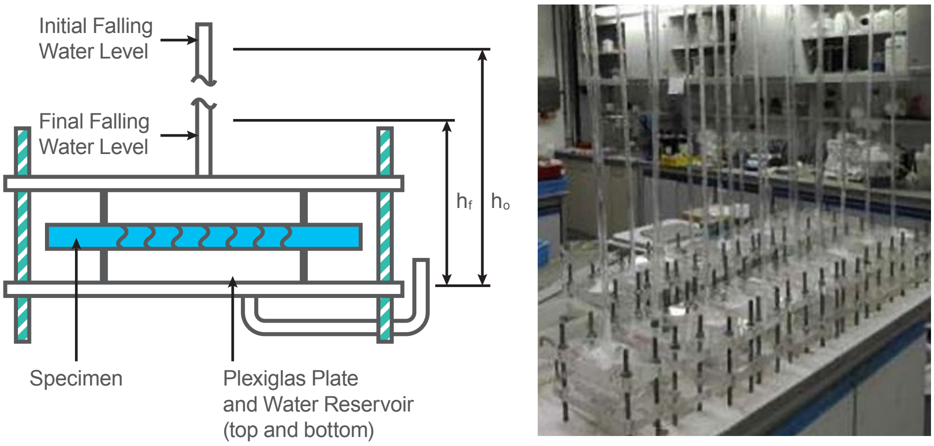


Figure 12 Crack pattern of (a) SAC/Cement =0.02; (b) SAC/Cement =0.1

Water permeability

Water permeability test was conducted. The test setup was shown in Figure 13b using the falling head designed by Lepech & Li, 2009. The coefficient of water permeability (k_w) of the ECC rendering was measured to be 4.46x10^{-10} cm/s. Compared with normal ECC, the ECC developed in this study demonstrated effective water resistance characteristics and the ability for waterproofing application.



(a) Falling head setup (b) Testing in laboratory

Figure 13 Water permeability test

3.2 Characterization and Modification of Fibers

In order to reinforce the cementitious matrix, the recycled PET fibers must be uniformly distributed, have suitable mechanical properties, and must be durable in the contact with alkaline cementitious matrix. The fiber geometry was first determined, and then the hydrophobic surface was modified to improve the alkali resistance and achieve a suitable bonding between fibers and matrix.

Physical properties of various fibers

Recycled PET fibers of various diameters ranging from 12.5 μm to 38 μm were tried to mix into the matrix (Figure 14). The length of all PET and PVA fiber is 12 mm. Generally, a small aspect ratio results in less agglomeration and better dispersion of the fibers. So the dispersion of fiber in the cementitious matrix largely depends on the fiber diameter. As shown in Figure 15, 2 vol.% 16 μm PET fibers exhibit poor dispersity that fiber balls are observed, the maximum content of 16 μm PET fiber to achieve uniform dispersion is 0.7 %. On the contrary, 38 μm PET fibers demonstrate quite good dispersity, and achieve a similar dispersion to the PVA fiber.

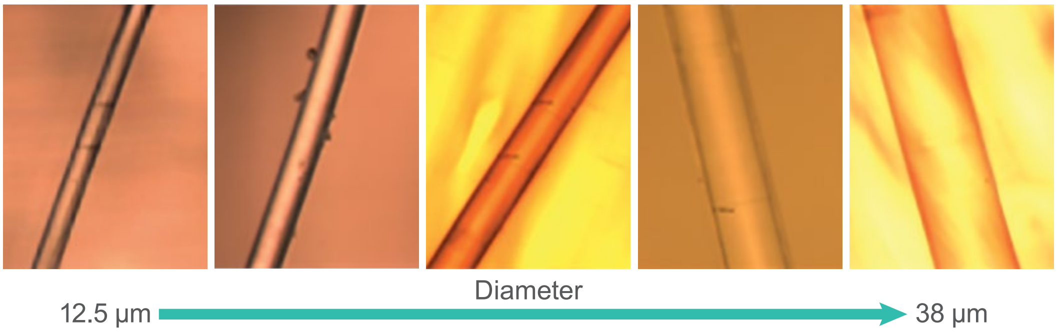


Figure 14 Optical microscope images of PET fiber of diameter ranging from 12.5 μm to 38 μm

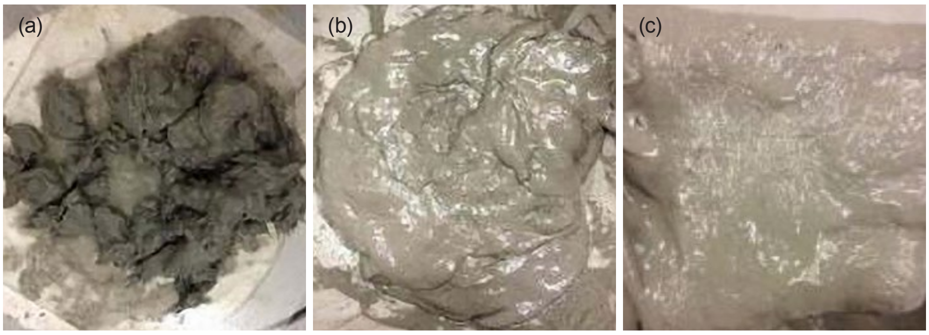


Figure 15 Fresh composite with 2 vol.% of a) 16 μm PET fibers, b) 38 μm PET fiber and c) 40 μm PVA fiber

Tensile stress-strain curves of PET fibers are shown in Figure 16 and compared to PVA fibers which are of diameter 40 µm as listed in Table 3. From the results,it is noticed that the 38 µm PET fibers exhibits competitive strength and larger ultimate tensile strain than PVA fiber. Since PET fibers are cheaper than PVA fibers, the hybrid utilization of the two fibers is desired.

Table 3 Physical properties of PVA and PET fibers (lab test)

Fiber	Length (mm)	Diameter (µm)	Aspect ratio	Modulus of elasticity (GPa)	Fiber strength (MPa)	Density (g/cm³)
PVA	12	39	308	16.9	1275	1.30
PET	12	38	318	10.7	1095	1.37

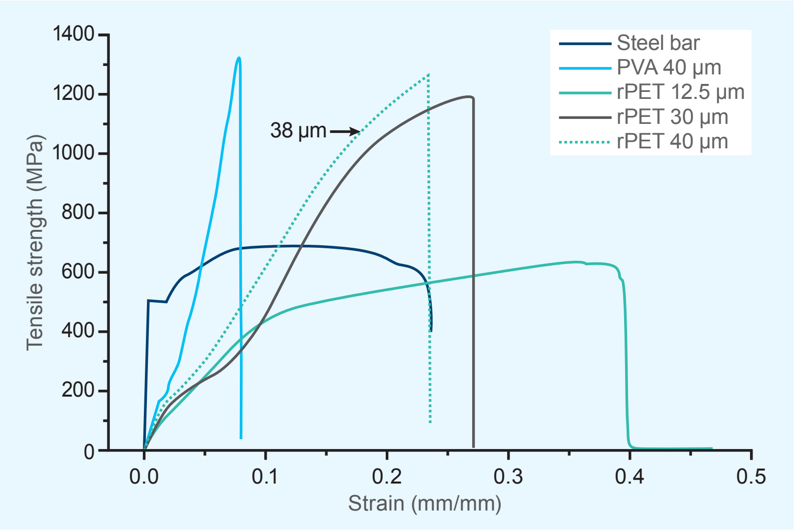


Figure 16 Comparison between different fibers materials used in cementitious material

Surface modification methods

Surface modification was applied to improve the adhesive strength between cement matrix and PET fiber. Comprehensive studies on the modification methods and condition were conducted, finally NaOH and silane KH570 treatment were selected. The FTIR spectra in Figure 17 indicated surface compositions and functional groups of as-manufactured PET fibers. A small peak at 1120 cm^{-1} representative of Si-O groups was observed, showing that the silane was effectively coated on the PET fibers.

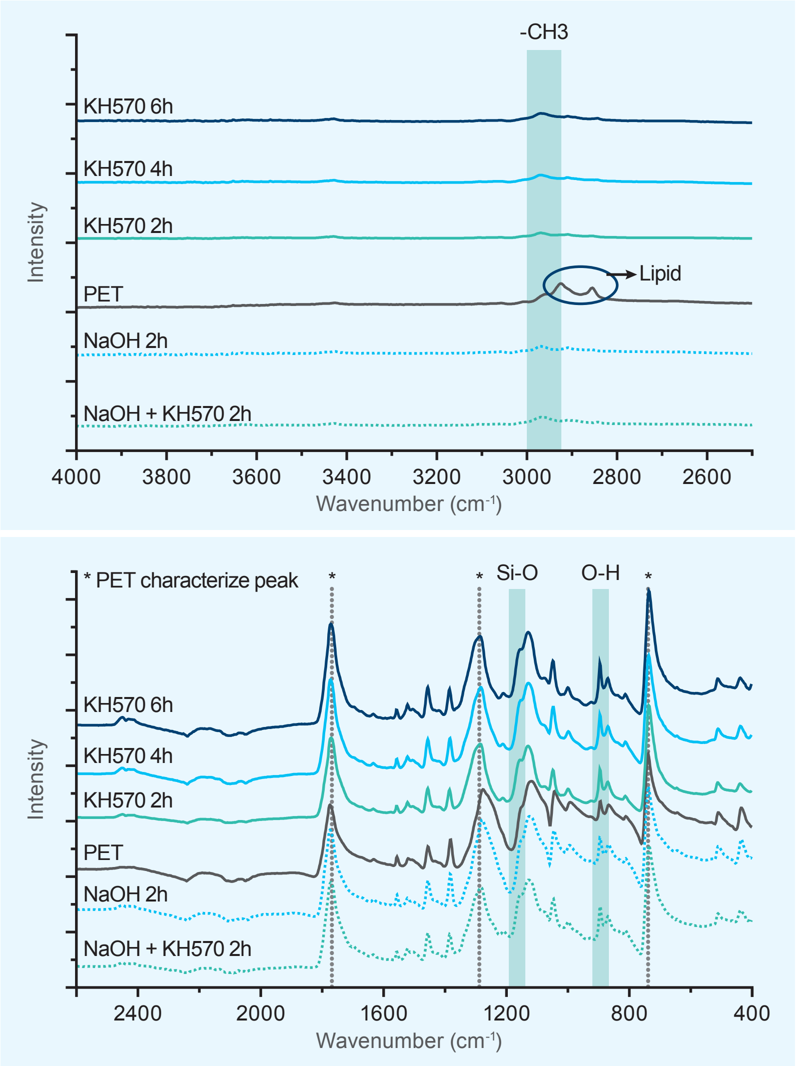


Figure 17 FTIR spectra of PET fiber after treatment

Single fiber pull-out test

The pull-out test was performed on plain PET, treated PET fiber and PVA fiber to determine the bond strength of various fibers with cementitious matrix. The results were shown in Figure 18, indicating that after the treatment the bond strength is lightly increased.

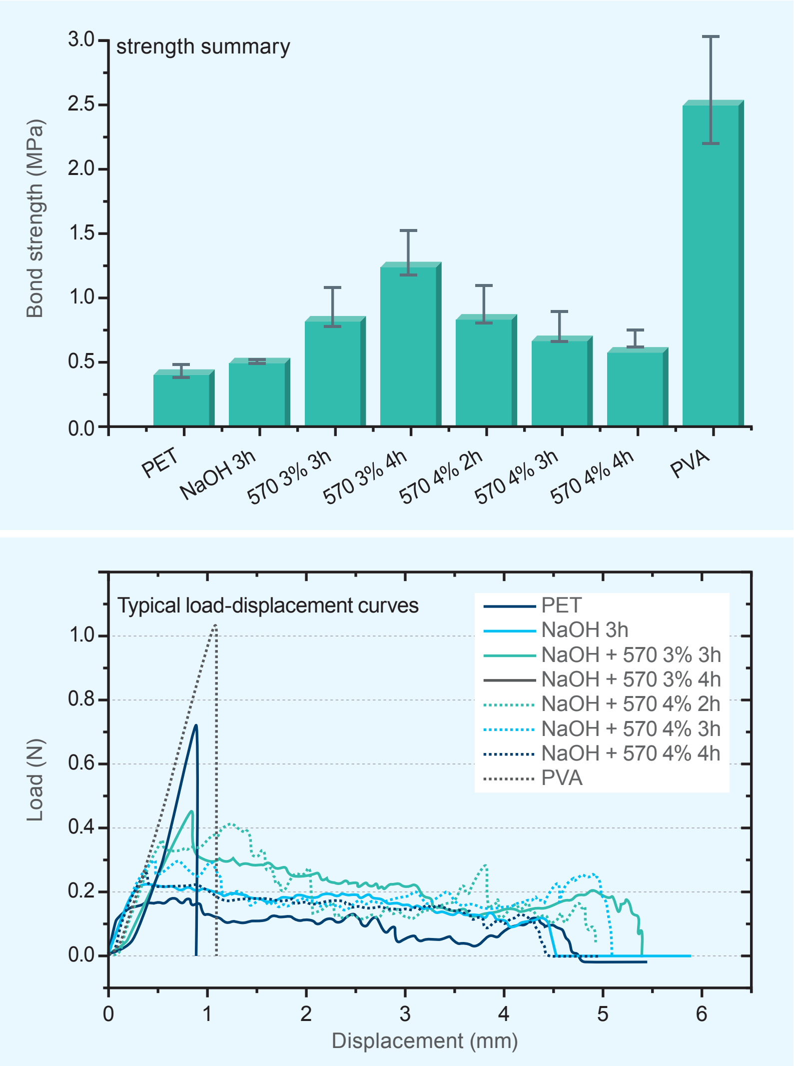


Figure 18 Single fiber pull-out test results

3.3 Mechanical Properties of Cementitious Rendering

Sample preparation and test procedures

The IDs for different mix proportions are based on the fiber combination, as shown in Table 4.

Table 4 Mix proportion of hybrid PVA/PET fiber ECC

Mix ID	Binder (B)	S	W	SRA/(B+S) [%]	WPA/(B+S) [%]	SP/B [%]	Fiber [vol.%]		
							PVA	PET-U	PET-T
P20	1	0.2	0.3	1	0.3	0.37	2	0	0
P15U05							1.5	0.5	0
P15T05							1.5	0	0.5
P10U10							1	1	0
P10T10							1	0	1
P05U15							0.5	1.5	0
P05T15							0.5	0	1.5
U20							0	2	0
T20							0	0	2

Note:
PET-U – Fresh recycled Polyethylene terephthalate fibers;
PET-T – Treated recycled polyethylene terephthalate fibers as discussed above.

Compression test

Based on the results shown in Table 5, the compressive strength of all the mixtures achieved 30 MPa or higher at 28-day age, and 48 MPa or higher after accelerated aging curing. The compressive strength is sufficient for waterproofing application. Generally speaking, the samples with untreated PET fibers show a little bit higher compressive strength than those with treated PET fibers. For the influence of different fiber combination, no obvious regular pattern can be concluded from the current results.

Table 5 Mechanical properties of rendering

Mix ID	Standard 28-day curing					Accelerated aging curing		
	Comp strength [MPa]	Tensile capacity [%]	Tensile strength [MPa]	Bending capacity [1x10^{-3}/cm]	Bending strength [MPa]	Comp. strength [MPa]	Tensile capacity [%]	Tensile strength [MPa]
P20	36.02	4.63	5.17	3.10	11.40	49.16	3.73	6.15
P15U05	37.50	3.27	4.44	2.86	10.20	63.54	2.62	4.60
P15T05	37.23	3.90	4.35	2.92	10.35	59.14	3.14	4.53
P10U10	34.46	1.83	3.59	2.45	10.19	60.35	1.20	3.76
P10T10	34.37	2.16	3.63	2.53	10.10	54.58	1.35	3.86
P05U15	37.22	0.97	3.20	1.82	7.52	58.29	0.53	3.60
P05T15	35.41	1.05	3.26	1.81	7.80	58.23	0.64	3.41
U20	32.62	0.66	2.82	0.76	6.63	50.06	0.54	2.76
T20	33.31	0.85	2.63	1.29	6.89	48.15	0.99	2.63

Uniaxial tensile test

The tensile stress-strain curves of the mixtures are shown in Figure 19, the mechanical properties of the mixtures are summarized in Table 5, for the samples under both 28-day standard curing and accelerated aging curing. ECC with pure PVA fibers (P20) shows the best performance in terms of tensile strength and ductility (Figure 19a) for both standard 28-day curing and accelerated curing. As indicated in Table 5, for the hybrid fiber systems, when more PVA fibers are replaced by PET fibers, the tensile behavior becomes poorer. Generally speaking, the samples hybridized with treated PET fibers show higher strength and improved ductility over those with untreated PET fibers. These results indicate that the fiber surface treatment is effective in enhancing the alkali resistance of recycled PET fibers, as discussed in the previous section. From the test results, even when 50% of PVA fibers are replaced by recycled PET fibers, the tensile ductility of P10T10 (and P10U10) is over 1.0% even after accelerated aging curing. This should be sufficient for waterproofing applications as strain during the serviceability state of structures rarely reaches such a high value.

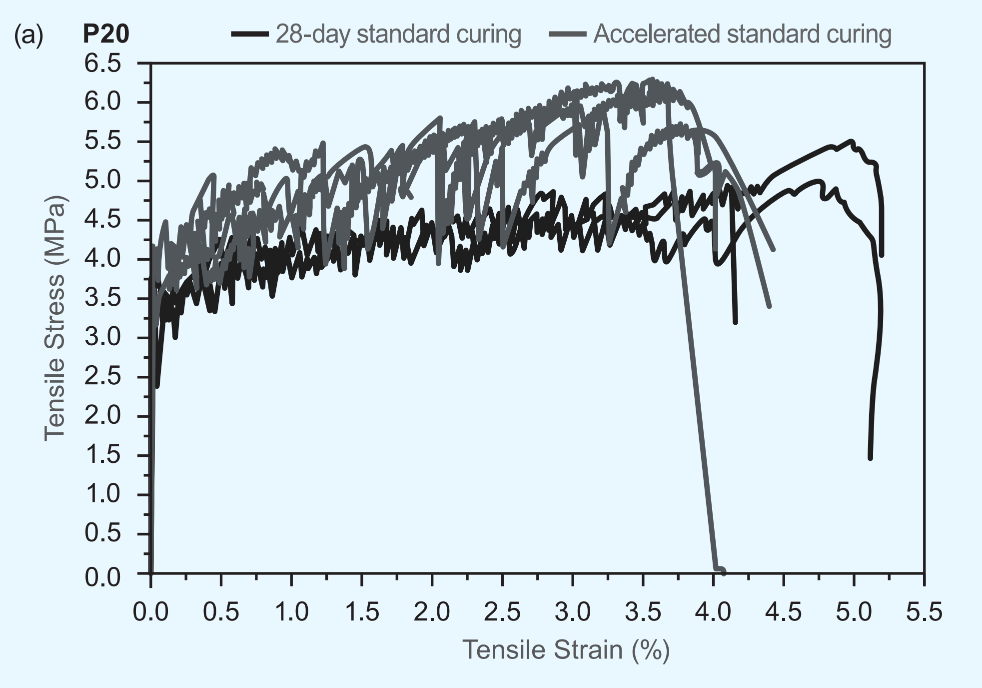


Figure 19 Tensile response of (a) P20, after both standard 28-day curing and accelerated aging curing

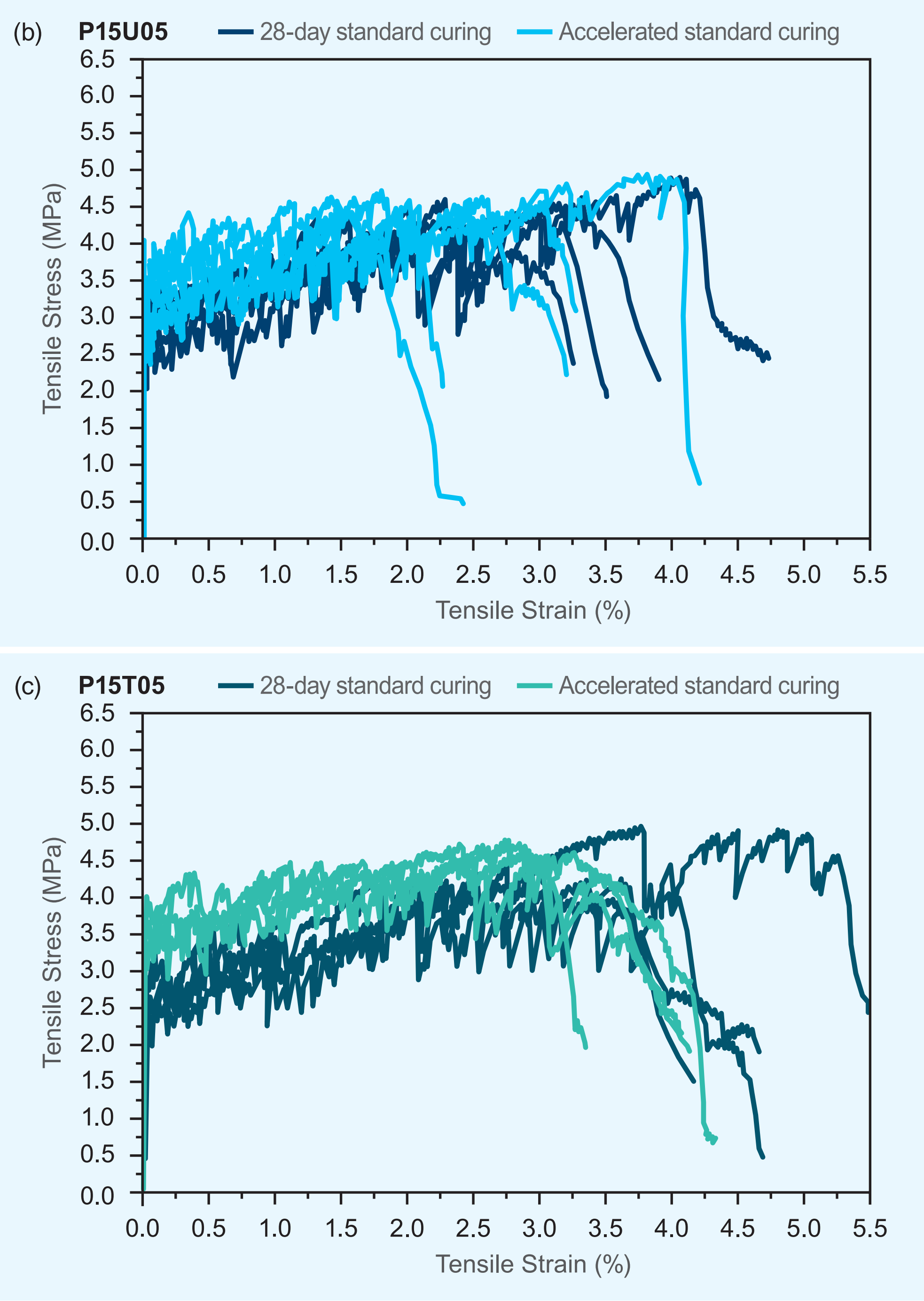


Figure 19 Tensile response of (b) P15U05; (c) P15T05, after both standard 28-day curing and accelerated aging curing

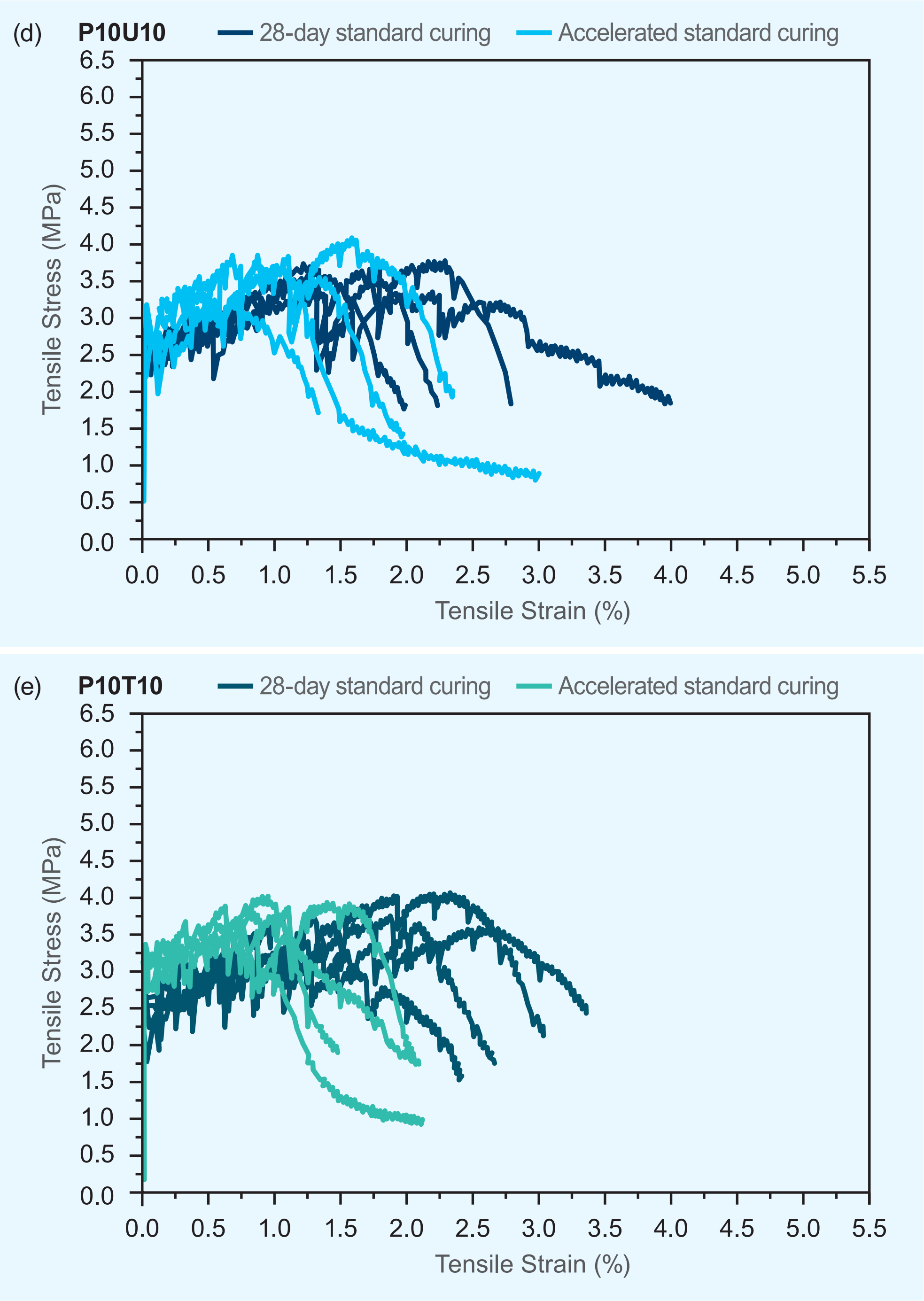


Figure 19 Tensile response of (d) P10U10; (e) P10T10, after both standard 28-day curing and accelerated aging curing

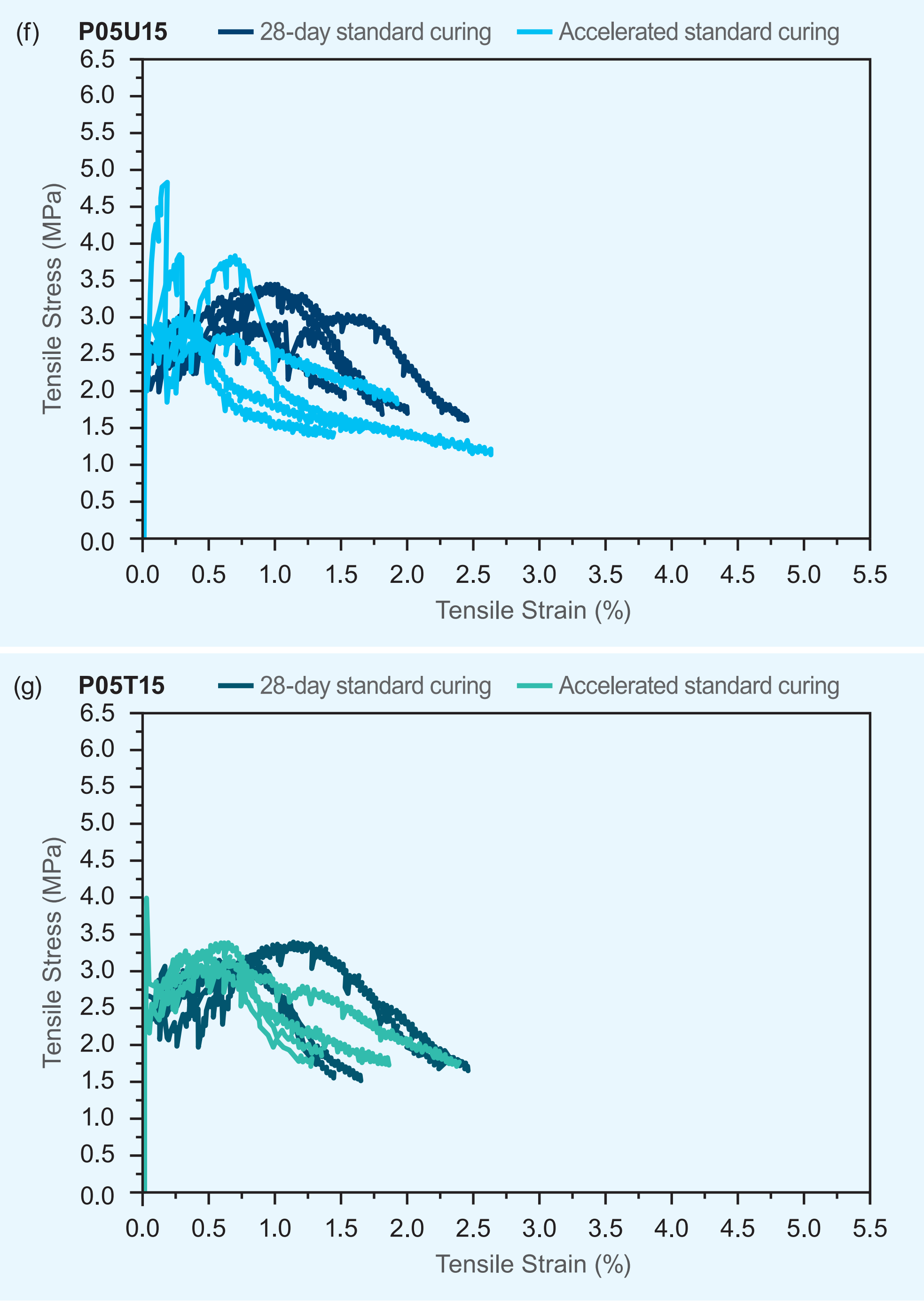


Figure 19 Tensile response of (f) P05U15; (g) P05T15, after both standard 28-day curing and accelerated aging curing

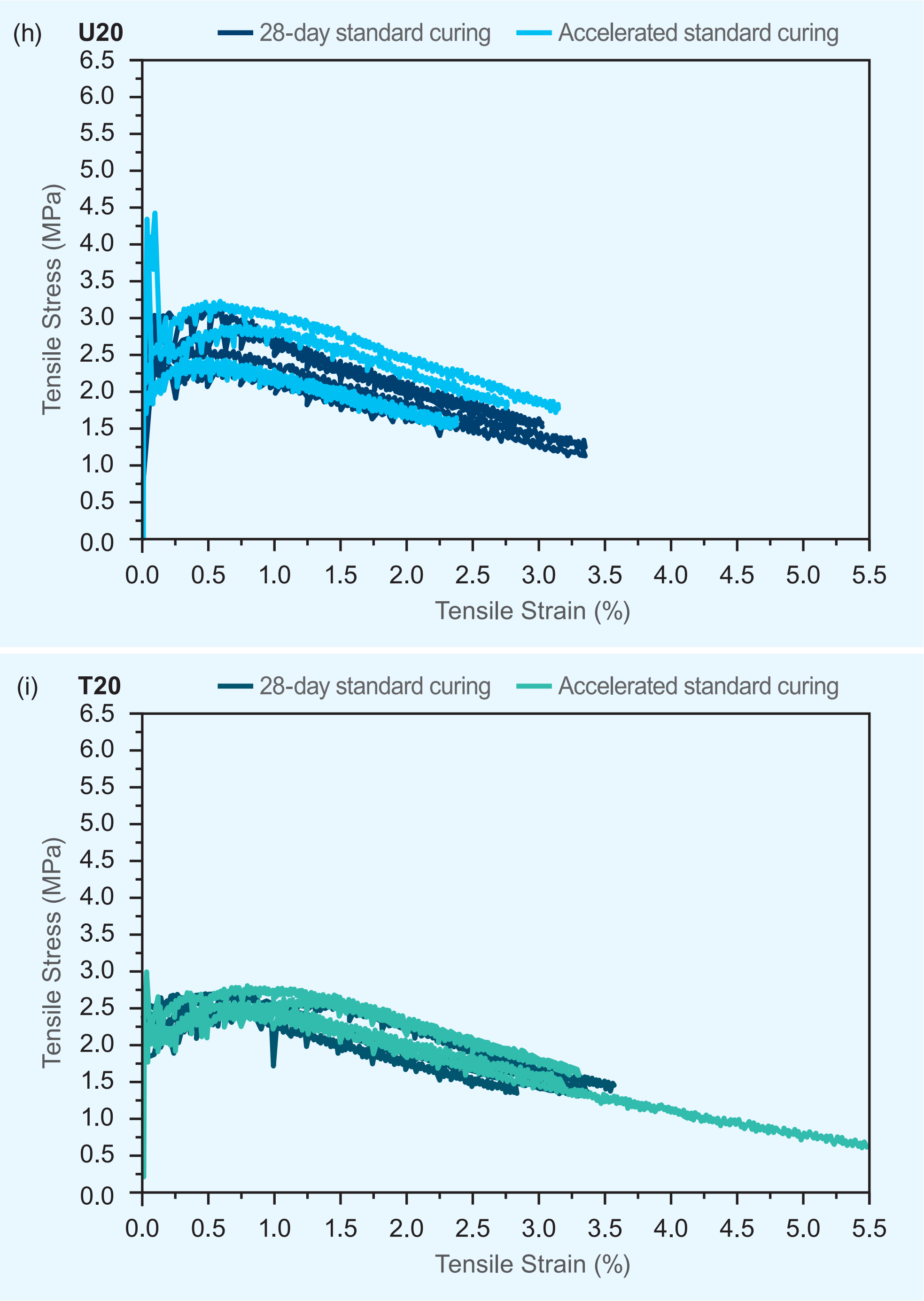


Figure 19 Tensile response of (h) U20 and (i) T20, after both standard 28-day curing and accelerated aging curing

Crack width analysis

According to the Hong Kong Code of Practice for Structural Use of Concrete (2013), maximum crack width is limited to 300 μm for normal structures and 200 μm for water retaining structures. Therefore, it is important to control the crack width of cementitious materials after first cracking. Crack width development of all specimens is indicated in Figure 20 and Figure 21, in which maximum crack width and average crack width against tensile strain are observed under direct tension at 28-day age and accelerated aging, respectively. With the present matrix and interface, PVA fibers control the crack very well. P20 shows an average crack width under 60 μm and the maximum crack width under 100 μm up to 4%. Specimens with treated fibers have generated better control on the average crack width than the untreated fibers. Under standard 28-day curing condition, for P10U10, the average crack width is under 100 μm at 1.0%, and the maximum crack width is under 150 μm at 0.625%, while for P10T10, average crack width is under 100 μm at 1.75%, and the maximum crack width is under 150 μm at 1.25%. In addition, T20 is observed to have a comparable average crack width to P05U15. In summary, the mix P10T10 would be suitable for many practical applications.

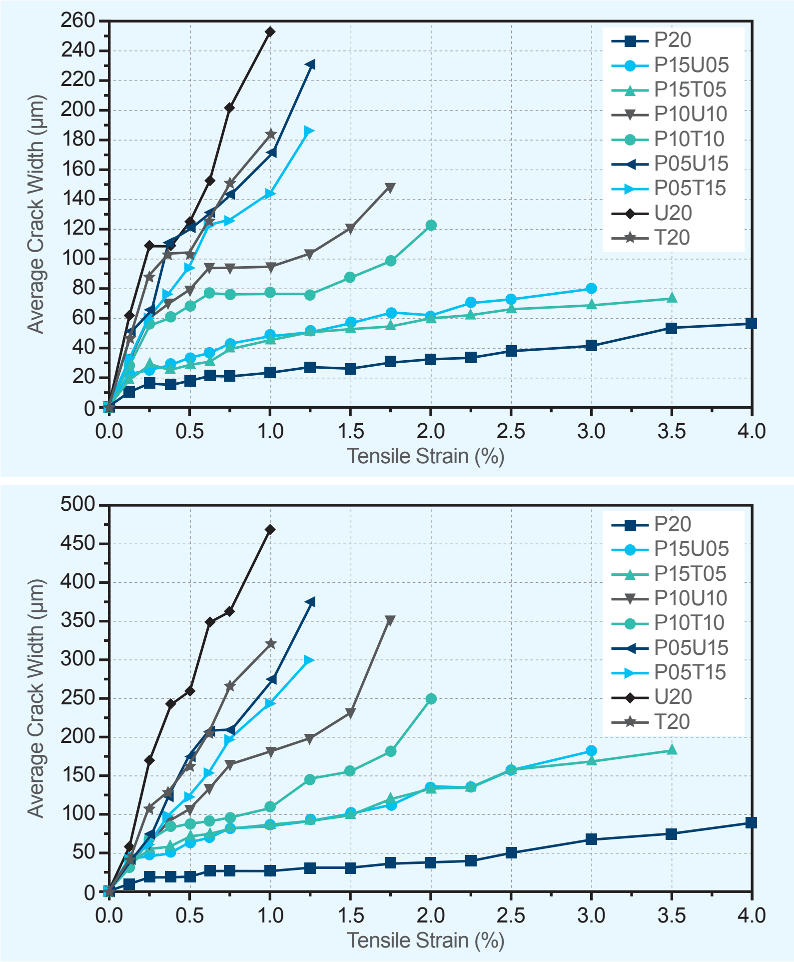


Figure 20 Crack width development of hybrid PVA/PET fiber ECC under direct tension at 28-day age

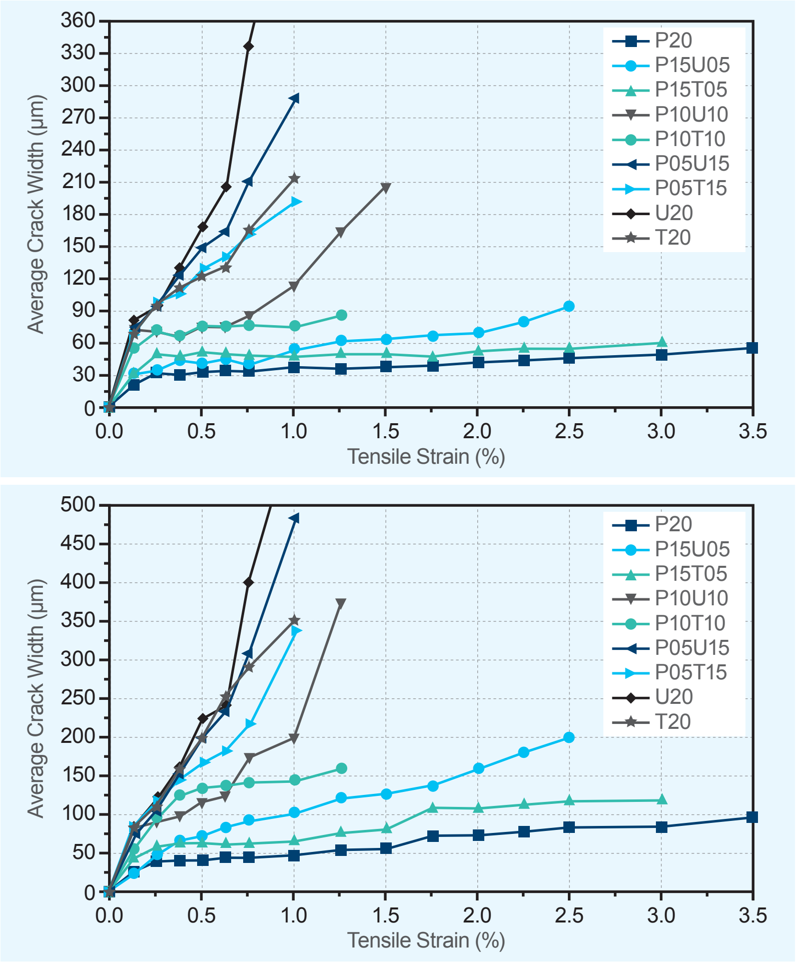


Figure 21 Crack width development of hybrid PVA/PET fiber ECC under direct tension after accelerated aging curing

Four-point bending test

The bending response of the mixtures is shown in Figure 22, and the major results are summarized in Table 5, for the samples under 28-day standard curing.

Multiple cracking was observed in all the samples, and similar first cracking strength was recorded. The ECC with pure PVA fibers (P20) showed the best performance in terms of both bending strength and bending ductility (Figure 22). As indicated in Table 4, for the hybrid fiber systems, when more PVA fibers were replaced by PET fibers, the bending behavior became poorer, which was similar to the direct tensile performance. Generally speaking, the samples hybridized with treated PET fibers showed almost the same bending strength and ductility as those with untreated PET fibers. For ECC with pure PET fibers, the bending ductility for mix with treated PET fibers (T20) was 70% higher than that with untreated PET fibers (U20). From the test results,it is observed that even when 50% of PVA fibers were replaced by recycled PET fibers, the bending ductility of P10T10 (and P10U10) was around 2.50×10^{-3}/cm, which was only 25% less than that of P20. This should be sufficient for waterproofing applications as the strain during the serviceability state of structures rarely reaches to such a high value.

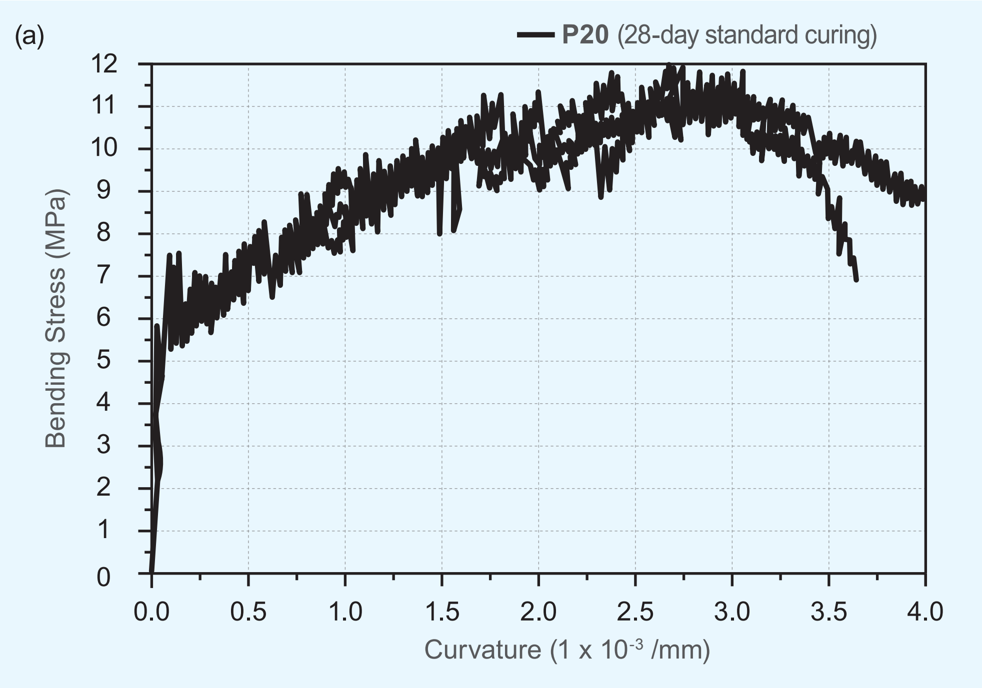


Figure 22 Bending response of (a) P20 after 28-day standard curing

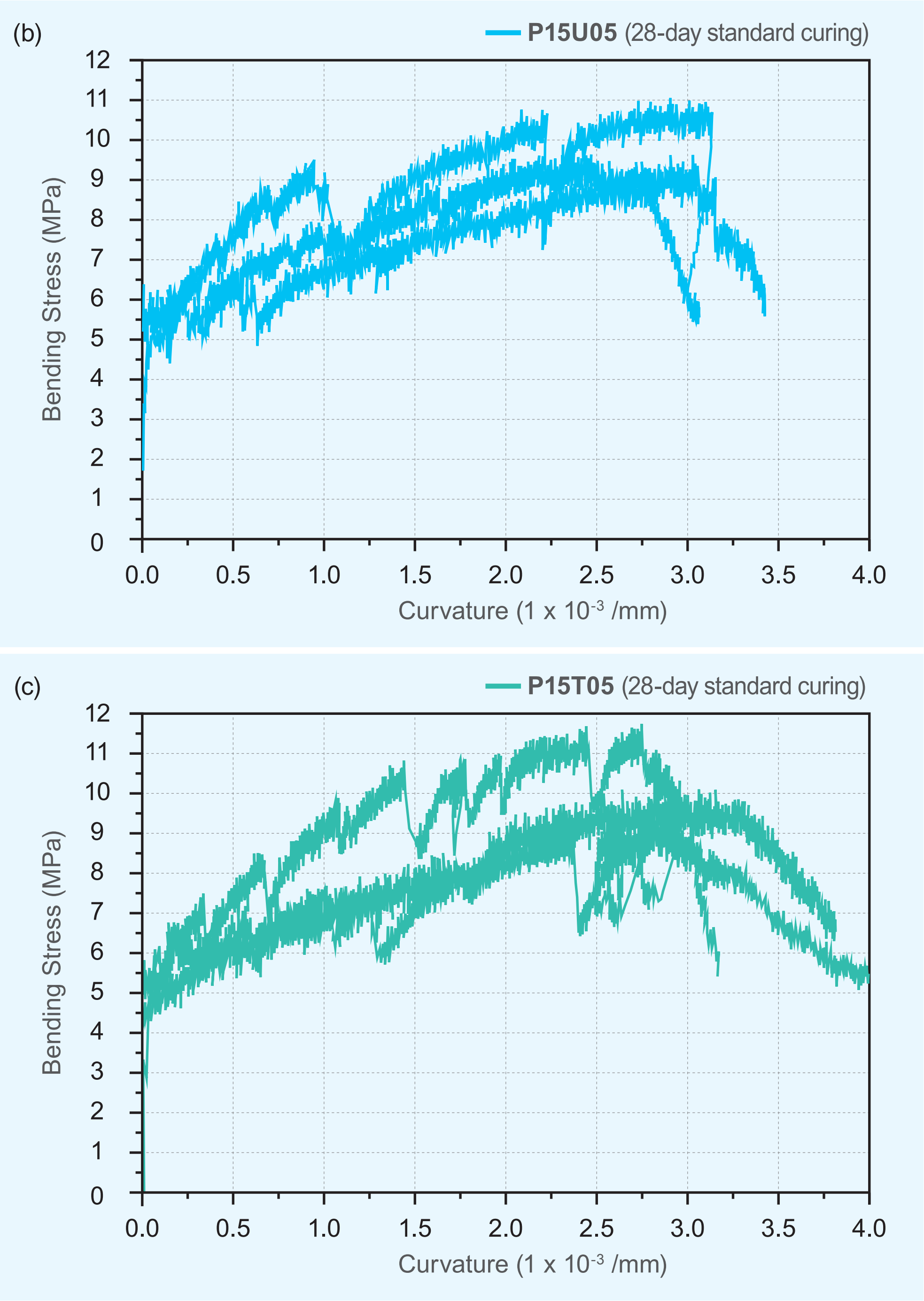


Figure 22 Bending response of (b) P15U05; (c) P15T05 after 28-day standard curing

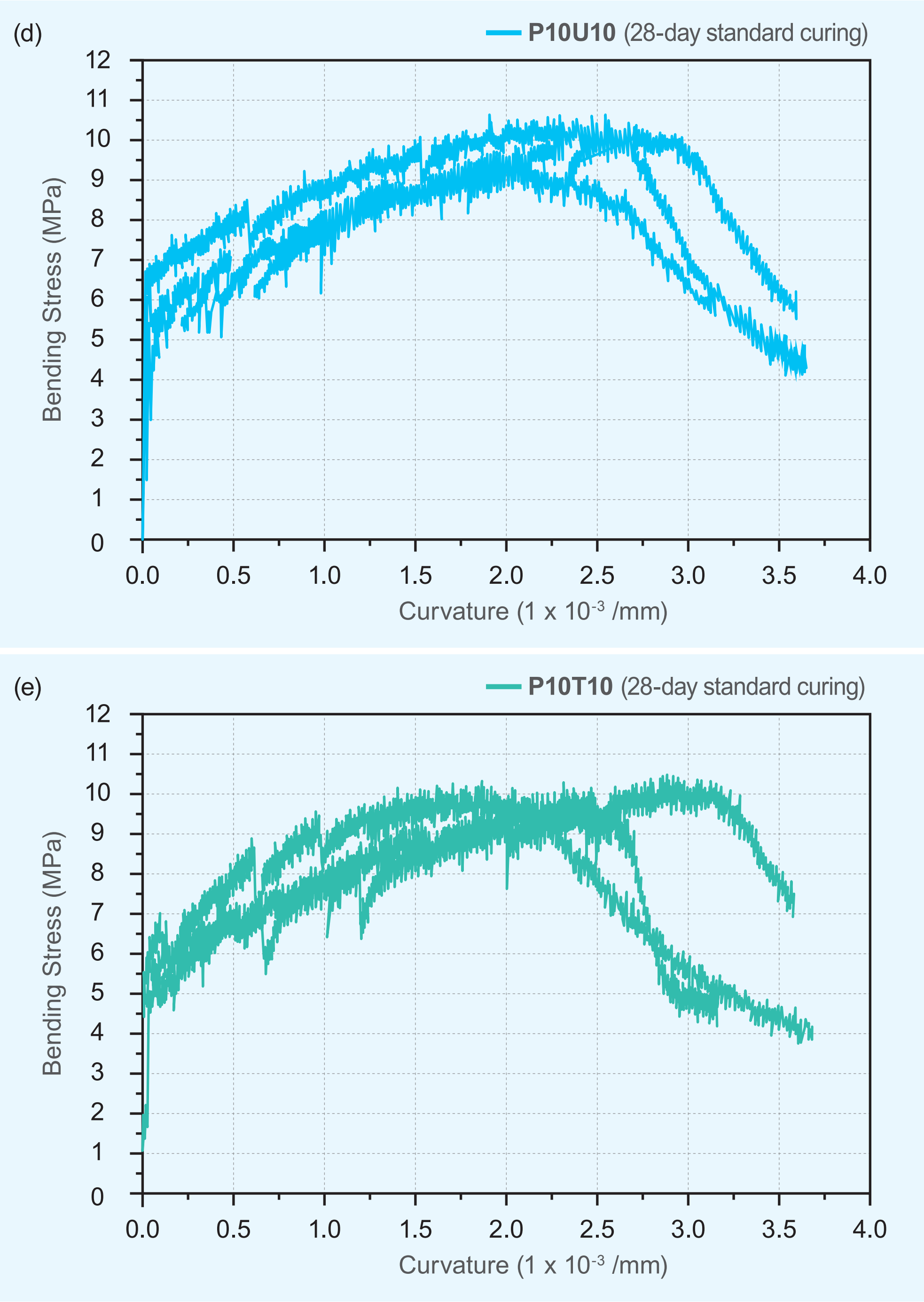


Figure 22 Bending response of (d) P10U10; (e) P10T10 after 28-day standard curing

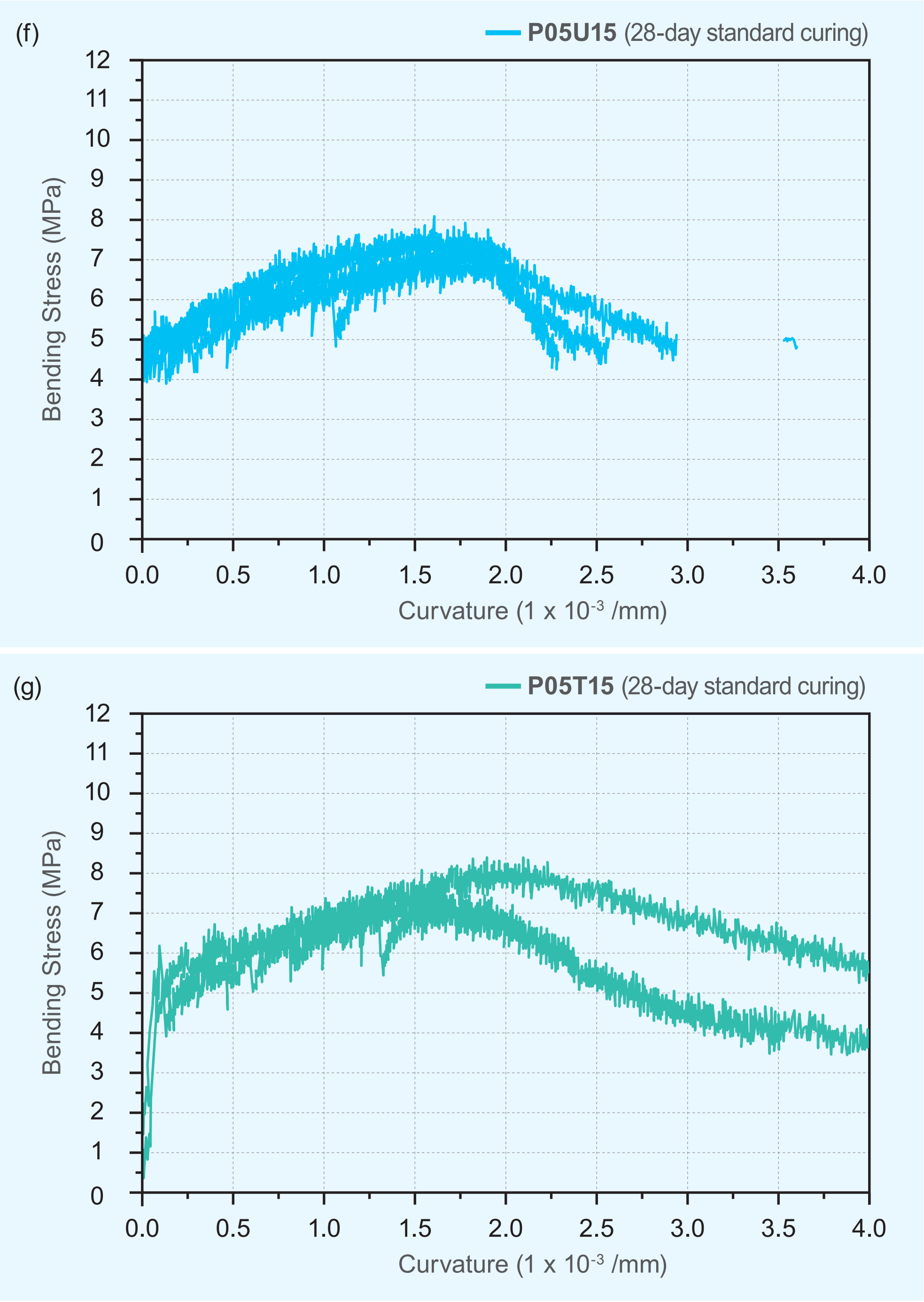


Figure 22 Bending response of (f) P05U15; (g) P05T15 after 28-day standard curing

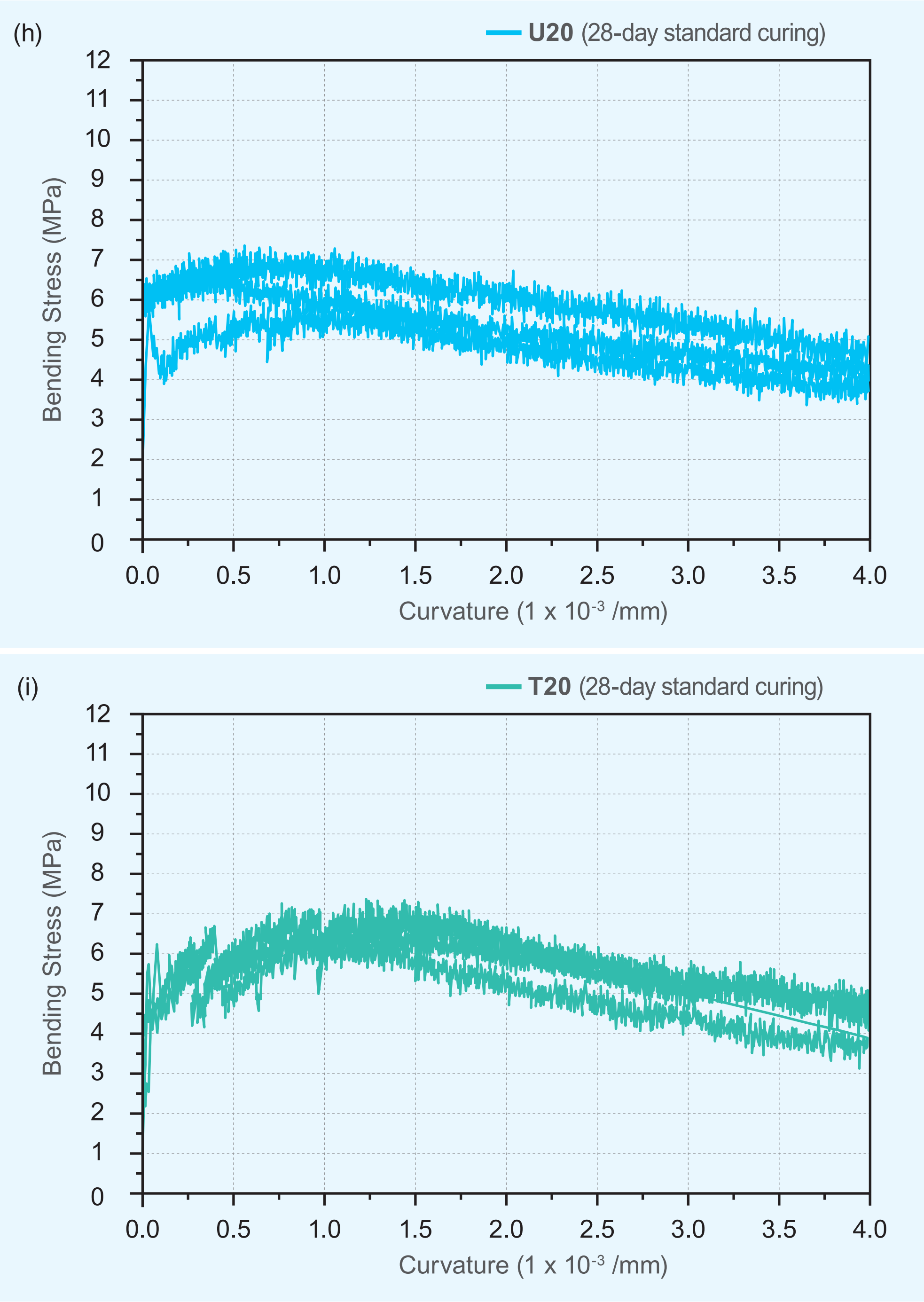


Figure 22 Bending response of (h) U20 and (i) T20 after 28-day standard curing

3.4 Workability Tests of Cementitious Rendering

Pull-off tests

Pull-off tests were carried out to assess adhesion strength between ECC and concrete substrate in tension in accordance with (ASTM D7234-12, 2012). 50 mm diameter dolly is attached on ECC by epoxy. Four possible failure modes are identified as shown in Figure 23.

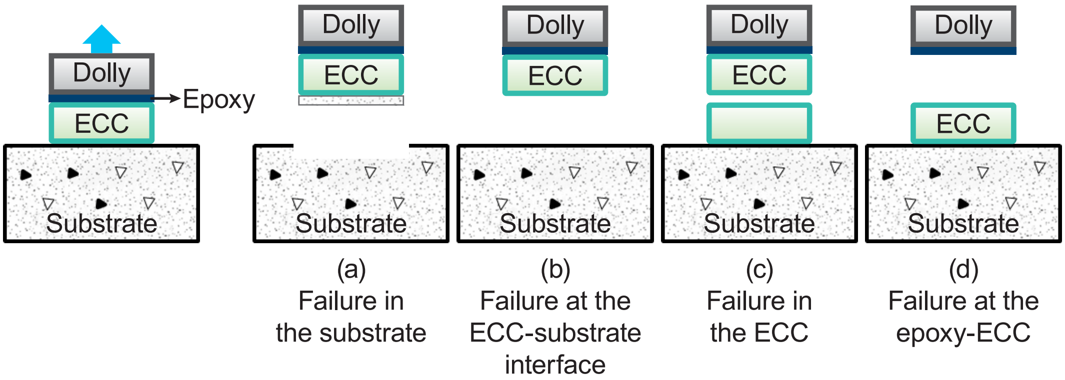


Figure 23 Possible modes of failure

Results of pull-off tests are shown in Table 6. Control sample was prepared on wet substrate surface and subject to standard curing for 7 days (test 1). The adhesion strength was 0.852 MPa. The adhesion strength on dry substrate surface after air curing (test 2) was 0.306 MPa, which was much lower than the control test. This indicates that water on the parent concrete substrate will not affect the adhesion between ECC rendering and concrete substrate, while proper curing could enhance the adhesion. Test 3 studied the compatibility of ECC rendering with commercial top coat. The adhesion strength was similar to test 1 and failure also occurred at the ECC-substrate interface, which indicated the good compatibility of ECC-Top coat (ECC-TC) system. To further improve the bonding, polyvinyl acetate (PVAc) was added to the mix. After 7 day curing, the adhesion strength of ECC with PVAc was only 0.463 MPa with the failure surface within ECC, indicating that the mechanical properties had been affected. However, after 28 day of curing, adhesion strength of ECC with PVAc (Test 6) increased to 1.23 MPa which was almost 50% higher than ECC without PVAc (Test 5). This finding suggested that for long-term performance, PVAc should be added.

Table 6 Adhesion strength between ECC and substrate under different condition

Test (3 specimen)	Testing condition	Adhesion strength (MPa) Ave.	Adhesion strength (MPa) Std	Failure mode
1. Control	7 day curing	0.853	0.039	b
2. Dry surface		0.306	0.054	b
3. With commercial top coat		0.833	0.09	b
4. With 1.5% PVAc		0.463	0.062	c
5. Control	Test after 28 day curing	0.865	0.005	b
6. With 1.5% PVAc		1.23	0.12	b

Flow table test of rendering

The flow table test in accordance with BS EN 1015-3 was conducted to evaluate the consistency of rendering. The flow diameter of rendering was measured immediately after mixing. In order to apply onto the wall; the ECC workability was adjusted to suit vertical application. With w/b ratio of 0.3, a large diameter is obtained, and the ECC falls off the wall. To solve this problem, w/b ratio was reduced to 0.28. If the w/b ratio was lower than this value, fiber could not mix well in mortar. Also, PVAc was added at 1.5% into fresh ECC mortar to achieve the right consistency of ECC as shown in Figure 16. Based on the results, it is suggested to use the w/b ratio of 0.28 and 1.5% PVAc.

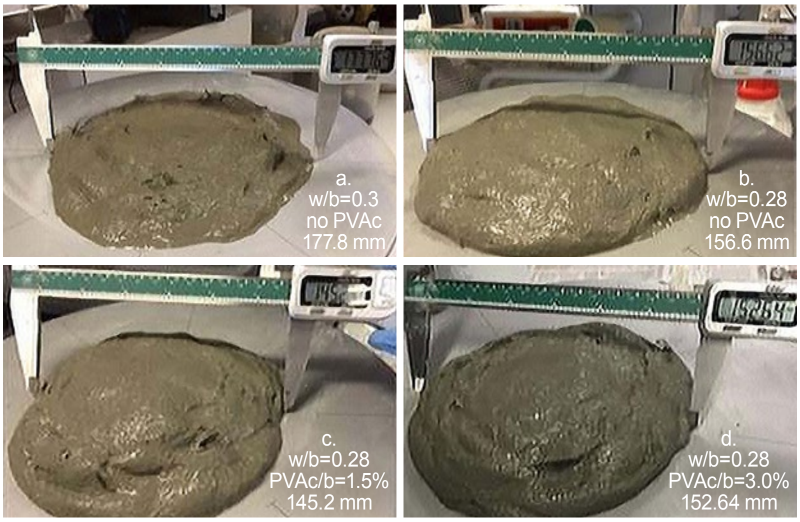


Figure 24 Workability of ECC with different mixes

Bending flexibility test of ECC-TC

To study the bending behavior of ECC-TC system, four-point bending test was conducted as illustrated in Figure 17. Four 150 mm × 400 mm × 50 mm (thick) concrete specimens were prepared, in which sample a was directly coated with 1 mm thick commercial top coat; samples b and c were coated with 15 mm thick ECC and 1 mm thick top coat. Sample b was loaded to the failure deflection of sample a, and sample c was loaded to failure; sample d was coated with conventional 15 mm thick cement-sand mortar without fiber, and then with 1 mm top coat.

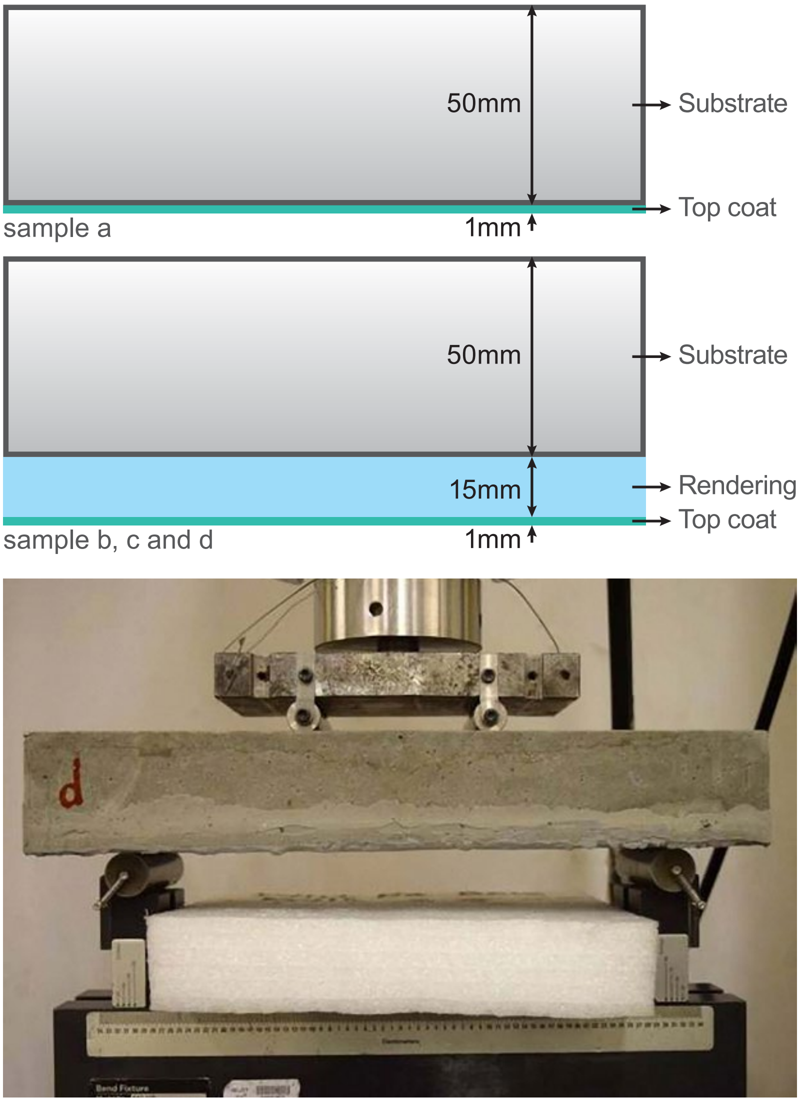


Figure 25 Experimental design for bending flexibility test

The results are shown in Figure 26. Sample a and d exhibit a peak load around 5.5 kN, and the ultimate deflection is less than 0.5mm. A single major crack is observed. With ECC rendering, the peak load of sample c increases significantly to 10 kN, and the ultimate deflection is up to 2mm. When the single crack propagates from substrate into ECC, multiple cracking can be observed in the ECC.

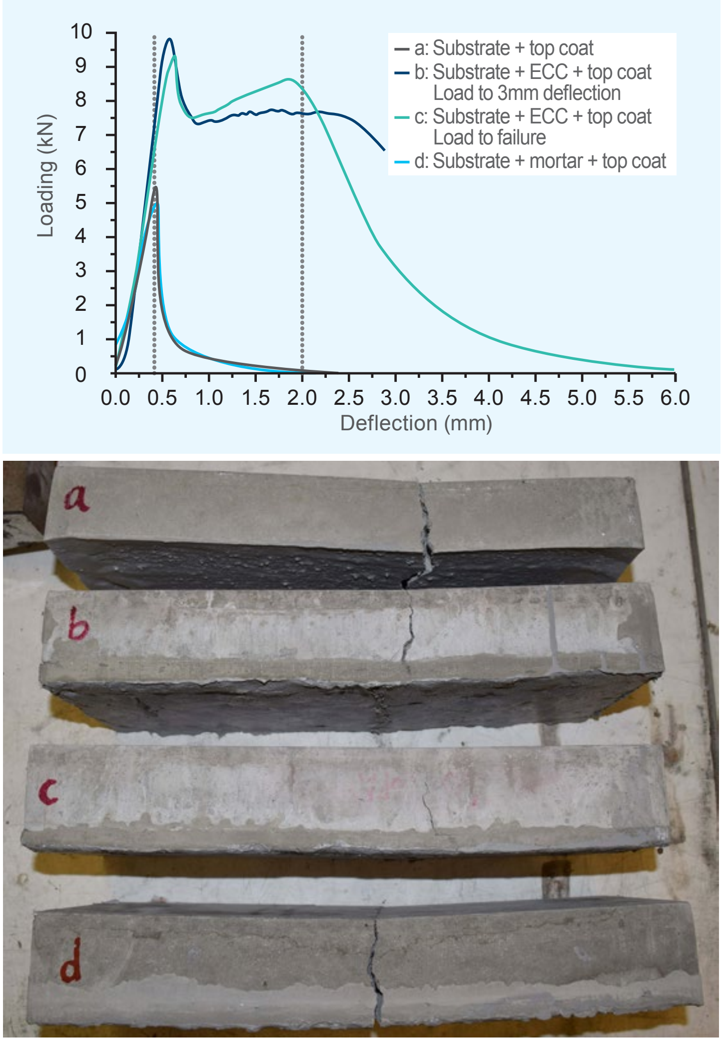


Figure 26 Bending flexibility test results

4 RECOMMENDATIONS

An ultra-ductile cementitious rendering for waterproofing application has been developed. The cementitious matrix and recycled PET fibers were comprehensively studied in the laboratory. Future field trials should be conducted to evaluate the applicability of the rendering. It is suggested to investigate the following aspects:

1. Proper material preparation and mix method for large scale production – Dry powder materials and fiber would be pre-mixed before adding water. And a proper mixing machine should be used to uniformly disperse the fibers.
2. Preparation of the concrete substrate – In order to realize the expected performance of rendering, the concrete substrate should be artificial chiselled by mechanically roughening or high pressure water-jet.
3. Optimum flowability of rendering for straight wall application – The flowability of rendering could be adjusted according to circumstances.
4. Durability of the rendering – durability tests should be carried out to estimate the service life of the rendering.
5. To deal with different levels of water seepage, fast setting materials and polymeric membrane integrated into the rendering to form a waterproofing system is recommended.

5 REFERENCES

ASTM D7234-12. (2012). *Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers* (Vol. 06.02). West Conshohocken, PA: ASTM International. http://doi.org/10.1520/D7234-12

Buildings Department. (2013). *Code of Practice for Structural Use of Concrete.* Hong Kong: The Government of the Hong Kong Special Administrative Region.

Hong Kong Environment Protection Department. (2015). *Monitoring of Solid Waste in Hong Kong Waste Statistics for 2014.* Hong Kong.

Leung, C.K.Y. (1996). Design criteria for pseudoductile fiber-reinforced composites. *Journal of Engineering Mechanics, 122*(1).

Lepech, M. D., & Li, V. C. (2009). Water permeability of engineered cementitious composites. *Cement and Concrete Composites, 31*(10), 744–753. http://doi.org/10.1016/j.cemconcomp.2009.07.002

Li, V. C. (1993). From micromechanics to structural engineering - the design of cementitious composites for civil engineering applications. *Doboku Gakkai Rombun-Hokokushu/ Proceedings of the Japan Society of Civil Engineers,* (471 pt 1-24), 1–12.

Li, V. C. (2002). Advances in ECC Research. *ACI Special Publication on Concrete: Material Science to Applications, SP 206-23,* 373–400.

Li, V. C., & Leung, C. K. Y. (1992). Steady State and Multiple Cracking of Short Random Fiber Composites. *Journal of Engineering Mechanics, 118*(11), 2246–2264. Retrieved from http://deepblue.lib.umich.edu/ handle/2027.42/84882

Li, V. C., & Wu, H.-C. (1992). Conditions for Pseudo Strain-Hardening in Fiber Reinforced Brittle Matrix Composites. *Applied Mechanics Reviews, 45*(8), 390. http://doi.org/10.1115/1.3119767

Ochi, T., Okubo, S., & Fukui, K. (2007). Development of recycled PET fiber and its application as concrete-reinforcing fiber. *Cement and Concrete Composites, 29*(6), 448–455. doi:10.1016/ j.cemconcomp.2007.02.002

Wang, K., Jansen, D. C., Shah, S. P., & Karr, A. F. (1997). Permeability study of cracked concrete. *Cement and Concrete Research, 27*(3), 381–393. doi:10.1016/S0008-8846(97)00031-8

Yang, E. H., Yang, Y., & Li, V. C. (2007). Use of High Volumes of Fly Ash to Improve ECC Mechanical Properties and Material Greenness. *ACI Materials Journal, 104*(6).

Zhang, J., Gong, C., Guo, Z., & Zhang, M. (2009). Engineered cementitious composite with characteristic of low drying shrinkage. *Cement and Concrete Research, 39*(4), 303–312. http://doi.org/10.1016/j.cemconres.2008.11.012

Members of CIC Task Force on Research

Mr. Jimmy TSE
Prof. Christopher LEUNG
Ir Joseph MAK
Prof. James PONG
Prof. Sze-chun WONG
Ir Chi-chiu CHAN
Ir Tommy NG
Mr. Shu-jie PAN

Research Team

Project Coordinator

Dr Ivan M.L. SHAM

Deputy Project Coordinator

Prof. Christopher K.Y. LEUNG
Prof. Kaimin SHIH

Project Team Members

Dr Xiuyi LIN
Mr. Jing YU
Dr Jingjing JIA
Dr Hedong LI
Mr. Jie YAO

Research Manager

Dr Jeffery Y.K. LAM

建造業議會
Construction Industry Council

Address 地址 38/F, COS Centre, 56 Tsun Yip Street, Kwun Tong, Kowloon
九龍觀塘駿業街56號中海日升中心38樓

Tel 電話 : (852) 2100 9000

Fax 傳真 : (852) 2100 9090

Email 電郵 : enquiry@cic.hk

Website 網址 : www.cic.hk

cic_hk

HKCIC

Construction Industry Council

CICHK

HKCIC

Construction Industry Council Hong Kong

2018

CIC Research Report No. CICRS_017