EVALUATING THE SELF HEALING BEHAVIOR OF THE FIBER-REINFORCED CEMENTITIOUS COMPOSITE INCORPORATING THE INTERNAL CURING AGENTS

by

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ABSTRACT

The formation of the cracks in concrete materials can shorten the service life of the structure by exposing the steel rebar to the aggressive substances from the external environment. Self-healing concrete can eliminate the crack automatically, which has the potential to replace manual rehabilitation and repairing work. This thesis intends to develop a self-healing fiber-reinforced cementitious composite by the use of internal curing agents, such as lightweight aggregate, zeolite and superabsorbent polymer (SAP). This study has evaluated the crack width control ability of three different types of fiber, polyvinyl alcohol fiber (PVA), Masterfiber Mac Matrix and Strux 90/40 fiber. Mechanical performance and flexural stress-strain behavior of the fiber-reinforced cementitious composite were tested and compared. In order to investigate the feasibility of using internal curing aggregate to enhance autogenous healing performance, two types of porous aggregates, zeolite and lightweight aggregate (LWA), were used as internal curing agents to provide water for the autogenous healing. The pore structure of the zeolite and lightweight aggregate was examined by the scanning electron microscopy (SEM). Two replacement ratios of sand with internal curing aggregates were designed and the healing efficiency was evaluated by the resonant frequency measurement and the optical microscopic observation. To further understand the influence of the internal curing on the designed material, water retention behavior of the bulk sample and the internal curing aggregates was evaluated. Moreover, to study the self-sealing effect of the superabsorbent polymer (SAP), the robustness of the SAP under various environmental conditions was first evaluated. The influence of the superplasticizer, hydration accelerator and fly ash on the absorption behavior of the SAP was investigated by the filtration test and void size analysis. Afterward, the self-sealing performance of the SAP in cement paste was evaluated by a water flow test.

The evaluation of three types of fiber indicated that the use of PVA fiber could produce a cementitious composite with stronger mechanical strength and crack width control ability. The result of the autogenous healing evaluation showed that the incorporation of the internal curing aggregates increased the self-healing recovery ratio from 12.6% to over 18%. The internal curing aggregate could absorb and store water during the wet curing and release it when the external water supply is unavailable. The comparison between the two types of internal curing aggregates indicated that finer pores in the internal curing aggregate can lead to a slower water release rate that is capable of continuously supplying water for the autogenous healing. In addition, the SAP was proved to be robust when various content of the additives and fly ash were used. And the self-sealing effect of the SAP is found to be effective in regaining the water tightness of cement paste. The result of this thesis can assist in the design of the fiber-reinforced cementitious composite with self-healing performance in civil engineering.

1. INTRODUCTION

1.1 Background and Motivation

Concrete is widely used in construction projects. It can be designed to have a service life as long as several decades. However, the cracking in concrete can lead to the deterioration of the structure. The formation of cracks can be attributed to a variety of reasons. For example, freezing and thawing, high salinity environment, and the early age shrinkage, etc. Even though reinforced rebar or fibers have been used to restrain the propagation of the crack by improving the post-cracking behavior of the concrete structure, the formation of the crack in the cementitious matrix is still inevitable. Although most of the cracks are initially small and would not directly affect the structural integrity, they can expose the inner part of the concrete structure to the aggressive liquids or chemical agents and cause the corrosion of the embedded reinforcement. These processes may also include volume expansion due to the chemical reaction, which further enlarges the crack width and depth. The existence of cracks, therefore, can negatively affect the sustainability of the concrete materials. Thus, timely repairs and rehabilitation which can prevent further deterioration are necessary for the concrete structure. However, those works could be extremely expensive, not to mention infrastructures such as bridges, tunnels and pavement require continuous service that can further increase the difficulty of the repair or rehabilitation.

To address the above-mentioned issues, concrete with the ability to self-heal the crack has been proposed and studied. The idea for self-healing concrete is that after the formation of cracks, concrete itself can re-generate solid substances that can block the cracks and even recover its mechanical strength. The phenomenon of self-healing of concrete was firstly reported by the French Academy of Science in 1836 [1]. The mechanisms for that are the secondary hydration of cementitious materials or carbonation and crystallization of the calcium hydroxide, so-called autogenous healing [2]. Since these processes are limited by water availability, mostly only small cracks can be self-healed [3]. Research shows that by providing sufficient water supply, the autogenous healing capacity can be enhanced [4]. However, for the in-field application, it is unrealistic to continuously supply external water for the concrete structure. Therefore, it is critical to avoid water limitation in order to improve the autogenous healing performance.

Internal curing is a method that provides internal water for the early age hydration, its goal is to reduce internal stress generated by self-desiccation. As a result, it has the potential to prevent the shrinkage and the formation of pre-mature cracks [5]. In this method, pre-wetted internal curing agents such as lightweight aggregate, superabsorbent polymer (SAP) or pre-wetted fibers, etc., are typically used as internal water reservoirs. During the hydration process, as the internal relative humidity reduce, the water absorbed by the internal curing agents can be gradually released and used for cement hydration [6]. It has been proved that the incorporation of the internal curing agent can effectively increase the internal humidity even at the age of 28 days. In Han’s study, the sample with internal curing agents exhibited an internal curing humidity of over 90% after 28 days curing under dry condition [7]. Based on the concepts of the internal curing method and autogenous healing, it is possible that the pre-wetted internal curing agent can provide water for the autogenous healing process. Due to the lack of experimental evidence, a systematic study of the influence of the internal curing method on the self-healing behavior is therefore necessary to be conducted.

1.2 Scope of this thesis

In this thesis, a hypothesis that the incorporation of the internal curing agent can improve the self-healing performance of the designed concrete is proposed. Two types of internal curing agents, internal curing aggregates and SAP, are used and evaluated. In the meanwhile, the mechanical performance of the designed concrete, and the robustness of the SAP in the concrete mixture are also investigated.

In order to limit the propagation of the crack during the loading process, synthetic fibers with crack width control ability were used to control the crack width. Three types of fibers were separately used as reinforcement, and the mechanical performance of the sample was evaluated and compared. Afterward, to verify the hypothesis that the internal curing aggregates can improve the autogenous healing, two types of pre-wetted porous aggregates, zeolite and expanded shale lightweight aggregate, were incorporated as internal curing agent to act as internal water reservoirs for the hydration of the cementitious materials. The autogenous healing performance of the designed material was then evaluated by two types of non-destructive tests. In the meanwhile, to investigate the influence of the porous internal curing aggregate on the mechanical performance of the cementitious composite, compressive, flexural and uniaxial tensile tests were performed. Moreover, to elucidate the underlying mechanism of the influence of internal curing agent on self-healing performance, an absorption test was performed on the concrete sample, and the desorption test and SEM analysis were conducted on the internal curing aggregates. In addition, apart from the porous aggregate, SAPs have been widely used as internal curing and self-sealing agent. Filtration test and the void size analysis were conducted to evaluate the robustness of the SAP. Afterward, the self-sealing effect of the SAP in cement paste was tested by the water flow test.

1.3 Organization and content of the thesis

This thesis has been divided into five chapters. Chapter one presents an overview of the existing problem in the concrete industry. A brief discussion of using self-healing concrete as a solution to solve the above-mentioned problem is provided. And a hypothesis about improving the self-healing performance is proposed. Chapter two corresponds to the literature review with regards to the design of a fiber-reinforced cementitious composite with self-healing ability. The concept and classification of the self-healing concrete are first introduced. The mechanisms and the pros and cons of each type of self-healing concrete are comprehensively reviewed. Moreover, in order to limit the crack propagation and improve the self-healing efficiency in concrete material, the design of the fiber-reinforced cementitious composite is discussed. In addition, since water is of vital importance in the autogenous healing process, the concept and the application of the internal curing method are also provided. Chapter three presents the design of the experimental works, including the mixture design of the fiber-reinforced cementitious composite, sample preparation, and the experiments conducted in this study. A flow chart about the experimental design is also provided. Chapter four presents the result of the experimental works. Firstly, the comparison between four types of fiber is discussed based on the result of the mechanical tests. Secondly, the evaluation of the influence of internal curing agent on the autogenous healing performance of the designed materials is presented. Two types of internal curing aggregates used in this study are compared based on the result of the mechanical tests and self-healing evaluation. Bulk sample absorption capacity, SEM analysis and the desorption behavior of the internal curing aggregates are presented, and the underlying mechanisms of the influence of internal curing on autogenous healing behavior are discussed. Afterward, the robustness and self-sealing effect of the SAP are presented. Finally, chapter five provides a conclusion of the work.

2. LITERATURE REVIEW

2.1 Self-healing in concrete materials

Self-healing concrete has been studied worldwide in recent years. The self-healing of concrete has the potential to extend the service life of the structure, reduce the maintenance cost and environmental pressure [8]. Based on the mechanisms of the healing, self-healing in concrete can be classified into two categories: autonomous healing and autogenous healing [9].

2.1.1 Autonomous healing

Autonomous healing of concrete is based on the artificially incorporated healing agents that enable the concrete to self-heal after cracking [10]. In general, the cracking process in concrete structure expose or release the embedded healing agent, the agent may then be activated upon it contact with air, water or specific chemical [9]. Different strategies to endow or enhance the autonomous healing performance of concrete materials have been proposed and investigated. Researchers used bacteria that can promote the precipitation of solid substances such as calcium carbonate ($CaCO_3$) through metabolism as self-healing additive to endow concrete with self-healing ability [11-15]. The embedded bacteria can be triggered by external elements such as water and oxygen [6]. Among different types of bacteria with various metabolic pathways, one with the ability to hydrolyze urea has been widely studied. In the metabolism process of bacteria, the urea is catalyzed by means of urease, and then degraded to carbonate and ammonium, as shown in the Equation (1~4) [16]. The newly produced carbonate can then combine with calcium ions from the cementitious matrix and form the calcium carbonate.

$CO(NH_2)_2 + H_2O \Rightarrow H_2COOH + NH_3$ (1)

$NH_2COOH + H_2O \Rightarrow NH_3 + H_2CO_3$ (2)

$2NH_3 + 2H_2O \Leftrightarrow 2NH_4 + 2OH-$ (3)

$2OH^{-} + H_2CO_3 \Leftrightarrow CO_3^{2-} + 2H_2O$ (4)

Another effective strategy is the incorporation of the capsules with healing agents into the concrete materials [17]. The release of the healing agent can be triggered when the new-form crack intersects and breaks the capsules [18]. The healing mechanism of various types of healing agents can be different. In Cailleux et al.'s work, tung oil and $Ca(OH)_2$ were separately incorporated into the capsule with a diameter of around 50μm [19]. Once upon the breakage of the capsules, the tung oil gets harden when it contacts with air, while the carbonation of $Ca(OH)_2$ with CO_2 produce $CaCO_3$ crystals. The physical and chemical reactions between the healing agents and the air are responsible for the closing of the crack. Similarly, expansive powder minerals such as magnesium oxide, bentonite and quicklime have been used as an encapsulated healing agent. The experimental result shows that due to the moisture in the air, the hydration of those mineral powders within the crack mouth can effectively recover the water tightness of the damaged area, and considerably improved the mechanical properties of the concrete [19]. In addition to the reaction of the healing agent with the external environment, chemical compounds that can react with the cementitious matrix can also be used as self-healing agent [3]. Huang et al. embedded sodium silicate solution into the cementitious composite as a healing agent. The sodium silicate solution was captured by the sponge with a diameter of about 5mm and then sealed by wax. Once the capsules break, sodium silica can react with the calcium cations in the cementitious matrix, which promotes the sealing of the crack [20].

With proper design, autonomous healing allows the healing of cracks larger than several hundreds of micrometers [21]. However, concerns about these techniques also exist. For the application of bacteria-based self-healing concrete, the survival of bacteria inside the harsh environment of concrete is questionable [22]. Not only the lack of space, water and oxygen, but also the high alkalinity may lead to the inactivation of the bacteria [11]. In order to ensure the vitality of the bacteria, extra protection is needed. Therefore, the alkaliphilic spore-forming strain has been used. The spores, which is the dormant state of the living bacteria, generally require less amount of nutrient and possess higher resistance to the harsh environment [23]. In addition, the mixing process of the fresh concrete may also apply damage to the bacteria. To solve this problem, various types of carriers, such as sol-gel and glass capillaries [14, 24], for the protection of bacteria have been designed and studied. It has been proved that the use of the carrier can effectively protect the bacteria, which enhances the healing performance of the concrete materials. However, the fabrication of the carrier can be complex and require further improvement. A more practical carrier technique is still needed.

As for the use of capsules, the activation of the healing mechanism can be triggered automatically once the capsules break, therefore, a relatively stable healing performance can be expected. However, some limitations still exist. First of all, the dosage of the capsule can be hard to determine. When a relatively low dosage is used, due to the random distribution of the capsule in the cementitious matrix, only a small portion of them will be triggered when a crack form. Therefore, a limited amount of the healing agent will be released to heal the crack [25]. On the other hand, when a higher dosage of the capsule is used, the inert capsule may act as flaws and negatively affect the mechanical strength of the concrete. The mechanical properties of the concrete material will then be a concern [26, 27]. Therefore, optimization of the mixture is necessary for the application of this strategy. In addition to the dosage, the stiffness of the capsule shell should also need to be well designed. Research has shown that whether a newly forming crack can intersect and break a capsule depends on the mechanical properties of the shell of the capsule [24]. When shell with high stiffness is used, the stress that causes the cracking of the cementitious matrix may not be sufficient to break the embedded capsule, thus, a low healing efficiency may be expected. On the contrary, capsule shells with low stiffness may not be able to survive the mixing process of the concrete. One of the most used capsules, glass capillaries, have exhibited a low survival rate after the mixing process. Various pre-treatment methods for the capsule have been studied. For example, protect the capsule by coating a layer of cement paste before the mixing [18]. Hilloulin et al used polymer with low glass transition temperature as the raw material for the capsule. A pre-heating process that transfers the polymer into the rubbery state is required before the mixing, which makes the capsule capable of surviving the mixing process [28]. Based on the literature review, it can be observed that most of the above-mentioned methods largely increase the difficulty and expense of concrete construction. It still requires effort to develop a capsule that is well-designed and easy to manufacture for the application of self-healing concrete.

2.1.2 Autogenous healing

Autogenous healing of the concrete is associated with the physical or chemical processes that occur in the cementitious matrix. By analyzing the healing product in the crack mouth, newly formed C-S-H gel [29] and calcium carbonate were found [30], which implies that the main mechanisms of the autogenous healing are the secondary hydration, carbonation of calcium hydroxide and the crystallization of portlandite ($Ca(OH)_2$) [2, 31]. Solid healing products can block the cracks and prevent the entrance of aggressive agents. As a result, the further deterioration of the concrete can be prevented, which ensures the service life of the structure [32]. Among the mechanisms of autogenous healing, the crystallization of calcium carbonate ($CaCO_3$) contributes the largest portion of the healing product [33]. Researchers have noticed that the whitish solid calcium carbonate can be observed within and outside the crack mouth [34]. Various studies have proved that $CaCO_3$ is the most efficient in terms of crack sealing and the regaining of water tightness. [3, 35]. The reason is that the carbonation process can happen as long as the calcium ions (Ca^{2+}) and the alkaline water is available. Due to the chemical composition of the concrete matrix, even if the water on the crack mouth is initially neutral or acidic, the pH value will increase because of the dissolution of the sodium hydroxide, potassium hydroxide and calcium hydroxide [21]. Moreover, since the cementitious matrix is rich in calcium ions, once upon the moisture is available, the carbonation process can be triggered.

The other autogenous healing mechanism, the secondary hydration has been proved to be highly responsible for the recovery of the mechanical properties of the concrete structure. Especially for the younger concrete with a larger portion of un-hydrated cementitious material that can be triggered by the external moisture. The newly formed healing products from secondary hydration possess a strength property that is able to re-connect the crack face and concrete debris together [36]. However, it is also worth noting that the nucleation of the secondary hydration products in the crack faces is not the same as the early age hydration in the bulk cement matrix. In Huang et al.'s study, the result of the differential thermogravimetric analysis (DTG) shows that a larger amount of bound water can be detected in the secondary hydration products [37]. This phenomenon may be due to a larger amount of water is available in the crack faces, compared with the hydration that takes place in the bulk cementitious matrix. Compared with the C-S-H gel in the bulk cementitious matrix, the mechanical property of the secondary hydration products is therefore weaker.

One of the most important factors that can affect the efficiency of the self-healing is the selection of cementitious materials. Previous research indicates that the partially replace of cement by fly ash and/or blast furnace slag can significantly improve the healing performance of the concrete [38]. The underlying mechanism is that compared with cement clinker, fly ash and slag possess a lower reactivity which can lead to a decrease of the early age hydration rate. Therefore, a larger amount of unreacted cementitious materials can be reserve for secondary hydration. It is also worth noting that the pozzolanic reaction of the fly ash and slag requires the existence of the calcium hydroxide. And it is known that calcium hydroxide is one of the hydration products of the cement. Thus, the replacement of the cement by the fly ash or slag should be controlled and limited to ensure that a sufficient amount of the cement will be hydrated to produce calcium hydroxide which can be used for secondary hydration of the fly ash or slag [2, 39].

Different exposure conditions have been studied by various researchers [40-45]. Hung et al. evaluated the healing performance of cementitious composites under three different curing conditions: lab-controlled dry, water submersion and natural weathering with high humidity. The result shows that the samples cured in a lab-controlled environment exhibited limited healing performance, compared with the other two curing environments with water supply [46]. Similar conclusion was also found in other researches [42, 47-49]. Thus, it can be seen that in order to achieve sufficient autogenous healing in cementitious material, water supply is of vital importance. During the autogenous healing process, water can act as carrier for the fine particles and ions, it is also indispensable for the chemical reactions. Previous researches found out that the healing efficiency in wet/dry cycles is higher than that in complete water submersion. This may be due to the wet curing can provide water for the secondary hydration, while the CO_2 is available for the precipitation of the calcium carbonate during the dry condition. Therefore, the combination of the secondary hydration and the carbonation during the wet/dry cycles can lead to a higher autogenous healing efficiency [47].

In addition to the mixture design and curing condition, the crack width is another factor that can significantly influence the healing efficiency. The width of the crack can directly determine the amount of healing products that are required to plug the crack [50]. It can be expected that smaller volumes of solid substances will be needed to heal a crack with narrow crack width. In general, concrete material can be used to sustain high compressive loading level, however, due to its intrinsic brittle nature, it is rather weak in tension [51]. The internal tensile stress caused by the shrinkage, external loading or thermal expansion may lead to the intensive propagation of the crack width. If the crack width can be controlled during the loading process, the autogenous healing performance can be improved. Therefore, fibers have been introduced to the development of self-healing concrete [32]. The fiber can act as bridges inside the concrete material and limit the propagation of the crack mouth. In addition to the crack width control, the reinforced fiber can also act as nucleation sites for the secondary hydration and the carbonation of calcium hydroxide. According to the microscopic observation, the healing products were found on the bridging fiber across the crack [52].

Based on the mechanisms of the autogenous healing, it is an intrinsic ability of cementitious material, with proper design, the concrete material can possess self-healing ability without aggressively increasing the cost and construction difficulty [2].

2.2 Fiber-reinforced concrete

Concrete is a complex composite material which composed of fine and coarse aggregate bonded with cement mortar. The border between different particles, especially the mortar-aggregate interface, is typically the weak spot of the concrete material [53]. The crack may therefore tend to generate at the weak spot during the service life of the concrete. Without timely repair, those cracks may propagate and cause further deterioration of the concrete structure. As mentioned in section 2.1.2, it can be expected that a crack with a larger width requires a larger amount of solid products in order to reach completed sealing [54]. Therefore, the control of the crack width has the potential to further improve the efficiency of the autogenous healing [35]. To address this issue, researchers have been used fiber as reinforcement to control the crack width in concrete material [33, 55, 56]. Generally, the tensile strength of the fiber used in the concrete industry is much higher than that of the hardened concrete matrix. The fiber can improve the post-cracking behavior of the concrete material by transferring the stress between crack faces and controlling the increase of the crack width [57, 58]. In addition, previous studies indicated that fiber can be used to produce concrete material with increased toughness and durability performance [59, 60].

2.2.1 Effect of the fiber parameters

In fiber-reinforced concrete, upon the formation of the crack, embedded fibers with random distribution can act as bridges to limit the propagation of the crack and transfer the stress across the cracks mouth [61, 62]. As a consequence, the strength and durability of the concrete with the incorporation of fiber as reinforcement generally higher than the plain concrete [63]. Nowadays, various types of fiber such as steel fiber, glass fiber, carbon fiber and PVA fiber, etc., have been developed and used in the concrete industry. Different parameters of the fiber, such as length and aspect ratio, can significantly affect the performance of the fiber-reinforced concrete [64].

The fiber geometry can intensively influence the concrete properties. In Doo-Yeol Yoo et al.'s study, the increase of the fiber length effectively enhanced the flexural deflection capacity of the cementitious composite [65]. In fiber-reinforced concrete, the completed pull out of the fibers can be considered as the main reason for the concrete failure. And the pull-out effect of the fiber can be determined by the bonding condition between the fiber and matrix. It can be expected that a longer fiber generally leads to an increment in the bonding area between fiber and the matrix, which results in an improvement in the ductility of the concrete [65]. Therefore, tailoring the fiber length can potentially affect the mechanical performance of the concrete. In addition, the researcher noticed that the increase in the fiber length could potentially lead to fiber entanglement and poor distribution, which may lead to a more porous structure [66]. It was reported that concrete material with longer fiber exhibited a lower compressive strength compared with those with shorter fiber [67]. Similar experimental result was also found in Noushini et al.'s study, where the increase of the length of the PVA fiber enhanced the strain capacity of the sample while the improvement in splitting tensile strength of the concrete was not observed [68]. Based on previous work, it can be concluded that the length of the fiber can effectively influence the mechanical performance of the designed concrete. However, a balance exists between the positive and negative effects of using longer or shorter fibers. Apart from the fiber length, previous studies showed that the aspect ratio of the fiber is another primary factor that can affect the mechanical properties of the concrete. It was reported that the increase in the aspect ratio could lead to higher compressive strength and toughness [69, 70]. In Gao et al.'s study, it was proposed that a larger aspect ratio of the fiber could result in a better control of the crack propagation. Therefore, the increment of the compressive and flexural strength can be expected when fiber with a high aspect ratio is used [71].

According to the literature review, it can be seen that the parameters of the fiber may affect the properties of the concrete material in a different way. Therefore, during the mixture design of the fiber-reinforced cementitious material, the selection of the fiber needs to depend on the specific requirement or application.

2.2.2 Engineered cementitious composite (ECC)

Among different types of fiber-reinforced concrete, engineered cementitious composite (ECC) has exhibited outstanding crack width control ability (less than 100μm wide), it was firstly introduced by Li and has been developed for over twenty years [72, 73]. ECC is a class of high-performance fiber reinforced cementitious composite that possesses ultra-high ductility with pseudo-strain hardening behavior, i.e. the ability to sustain higher tensile loading and deformation after the generation of the first crack. The tensile strain capacity of the ECC can be several hundred times higher than the conventional concrete [33].

Conventionally, the design of the high-performance fiber-reinforced concrete is focused on developing a tightly packed cementitious matrix combine with high strength fiber to increase the tensile or compressive strength [74]. For ECC, the optimization of the mixture was based on adjusting the relationship between fiber, cementitious matrix and the interface [75]. Moderate volume fraction of the short polyvinyl alcohol fiber (2%) with a tensile strength of 1600-2500MPa was used as reinforcement in ECC. During the loading process, the bridging fiber will be deboned and pulled out from the cementitious matrix, during which, the fiber/matrix interface plays an important role. If the fibers do not debone or slip due to an extremely high bonding strength between the fiber/matrix interface, the fiber would tend to break instead of controlling the crack. On the contrary, if the interface is too weak to control the slippage process, the crack width could propagate aggressively [76]. To solve this problem, Li et al. tailored the fiber/matrix interface by applying oil coating on the surface of the fiber. The interface bonding strength can be controlled by the dosage of the coating oil [75]. The toughness of the cementitious matrix ECC is tailored to be weaker than the PVA fiber, which indicates that the cementitious matrix will yield and generate new cracks before the bridging fiber reaches its ultimate tensile strength. And the fibers across the newly formed cracks will simultaneously participate in transferring the tensile loading [77]. The repeat of the above-mentioned process can eventually result in the formation of multiple microcracks.

It was reported that the tensile strain capacity of the ECC is generally higher than 2% with a crack width smaller than 100 μm [78]. Various studies have been done to further improve the tensile performance of the ECC. Yang et al. investigated the influence of high-volume fly ash usage on the mechanical properties of ECC. The result indicated that increasing the amount of fly ash can potentially improve the tensile strain capacity. The author concluded that the use of high-volume fly ash can increase the fiber/matrix interface frictional bond which restrains the fiber from pulling out of the matrix. Therefore, a larger number of cracks with smaller crack width can form during the loading process, which leads to a higher strain capacity. However, the study also reported that the compressive strength of the ECC was reduced especially at the early age due to the low reactivity of the fly ash [79]. Similar conclusion was also found in Şahmaran's research [80]. In order to solve the issue of low mechanical strength caused by fly ash, granulated blast furnace slag was incorporated to increase the early age reactivity of the ECC. In ternary cement–slag–fly ash system, slag can be considered as a beneficial ingredient for the early age mechanical performance of the concrete while the fly ash will mostly contribute to the long-term development of the concrete strength [81, 82].

Apart from the mechanical properties, the durability of the ECC under aggressive environments was also evaluated by various researchers. Şahmaran et al. evaluated the alkaline resistance of the pre-loaded ECC specimens. According to the result of the accelerated mortar bar test, ECC did not show any expansion after 30 days of exposure. It was concluded that due to the use of fly ash, a finer pore structure could be expected in the cementitious matrix, which prevents the alkali from infiltrating into the sample [83]. Similar conclusion was also found in Li et al.'s study, where the ECC samples were exposed to the chloride environment [45]. In Şahmaran's another study, the durability of the ECC was evaluated under freezing and thawing cycles. The test result showed that, in terms of mass loss, pulse velocity change and flexural parameters, ECC exhibited a better freeze-thaw resistance compared with conventional concrete. Most of the ductility and mechanical strength of the ECC sample can be maintained after 300 freezing and thawing cycles [84].

2.3 Internal curing for concrete material

To design a high-performance concrete, people generally increase the content of the cementitious material to produce a denser and stronger cementitious matrix. However, even though the low water to cement ratio benefits the strength of the concrete, it also increases the risk of the early age shrinkage cracks formation [85]. During the hydration process, the loss of water and the decrease of relative humidity leads to the self-desiccation that generates internal stress. Under the constraint condition, the internal stress can cause the formation of the cracks, which may lead to the early age failure of the concrete. For concrete with relatively high water to cement ratio, the external curing method can be used to supply moisture and mitigate the shrinkage. However, when it comes to the concrete with low water to cement ratio, a low permeability caused by the dense matrix does not allow the transfer of external water from the surface to the interior of the material [86, 87]. Therefore, external curing has limited efficiency on the shrinkage reduction for the concrete with high binder content [88].

In order to mitigate the potential damage caused by shrinkage in concrete with low water to cement ratio, the internal curing method has been developed. Pre-wetted internal curing agent with high water absorption capacity are used to serve as water reservoirs to supply extra water from the inside of the concrete material [89]. It has been proved that the pre-wetted internal curing agent can releases the water once held in its structure during the hydration, and prevent the autogenous shrinkage of high-performance concrete [6].

2.3.1 Internal curing aggregates

In the application of the internal curing method, the chosen of the internal curing agent is of vital importance. The internal curing agent can not only affect the shrinkage reduction efficiency, but also the mechanical properties of the concrete. Pre-wetted porous aggregates were initially used to partially replace the sand in high performance concrete to serve as the internal curing agent. The porosity of the internal curing aggregates is generally responsible for the absorption capacity. High absorption capacity can potentially increase the effectiveness of the internal curing by carrying a larger amount of extra water. In addition, the size distribution of the pores inside the internal curing aggregate also influences the internal curing efficiency [89, 90]. The previous study indicated that the pores in internal curing aggregate need to be larger than those in the cementitious matrix, so that the water carried by the aggregate will be preferentially released and consumed by the hydration process [91].

Various types of internal curing aggregates have been studied and used in high-performance concrete materials [92-94]. Among them, expanded lightweight aggregate is one of the most frequently used internal curing agents. It has been proved that complete elimination of the shrinkage may be expected when 25% of the normal-weight aggregate was replaced by lightweight aggregate [6]. The large pore with a diameter of greater than 1μm in lightweight aggregate can be saturated within a short time and exhibit low water retention ability [89]. During the early age hydration process, those large pores are capable of releasing most of its absorbed water at high relative humidity. Thus, it is an effective internal curing agent to reduce early age shrinkage by providing water to replenish the empty pores caused by self-desiccation [95]. On the other hand, clinoptilolite zeolite, a porous aluminosilicate mineral, was reported to have nm-sized pores that are capable of storing water down to low relative humidity levels [96-99]. The previous study indicated that the incorporation of pre-wetted zeolite could result in a shrinkage reduction of over 30% [100]. In Ghourchian et al.'s study, the internal curing efficiency of the zeolite is compared with expanded lightweight aggregate. It is reported that the 24 hours absorption capacity of the zeolite is around 41.8% higher than the lightweight aggregate. The size distribution of the pores was evaluated by the mercury intrusion porosimetry test. The result showed that lightweight aggregate possessed a coarse pore structure, while a large volume fraction of the nano-pore existed in the zeolite. For the absorption/desorption behavior, the lightweight aggregate was capable of releasing more than 95% of its absorbed water at a relative humidity of 90%, while zeolite retained around 90% of the water under the same environmental condition. Therefore, it is more likely for the zeolite to store part of its absorbed water, instead of releasing it for early age hydration.

Consequently, due to the high water retention capacity of the zeolite, less effective in the reduction of shrinkage could be expected when the zeolite is used as an internal curing agent [97].

For the mechanical properties of the concrete incorporated with internal curing aggregate, a balance exists between the side effect of the porous structure of the internal curing aggregate and the positive effect of the internal curing. The high porosity of the internal curing aggregate may negatively affect the strength of the concrete while a higher degree of hydration caused by the extra water carried by the curing agent has a positive effect on the mechanical properties of the concrete [5]. In previous studies, both decrease and increase in strength caused by the internal curing have been observed. It was reported that the use of the internal curing method resulted in a decrease of the early age compressive and tensile strength of the concrete, mostly due to the porous structure and larger particle size of the lightweight aggregate [101, 102]. On the contrary, an increase in the long-term compressive strength was observed in Bentz's study. It was concluded that in the designed high-performance blended cement mortar, the improvement in the hydration overcame the negative influence of the porous structure of the internal curing agent, which resulted in an increase of the concrete strength [103]. Similar result was also found in other studies [104, 105].

2.3.2 Superabsorbent polymer (SAP)

After the invention of the internal curing method, some concerns have been reported about the use of porous aggregates, requiring the pre-soaking prior to mixing, which could be difficulty to handle in the field applications due to the inaccurate control of the absorption capacity of the curing aggregate. Also, the incorporation of the porous aggregate will lead to the decrement of the mechanical properties due to the incorporation of porous component [106]. Therefore, superabsorbent polymers (SAP), which can be added in the cement mixtures in the form of dry powders, was proposed as a new type of internal curing agent to prevent the shrinkage of concrete and has been widely studied [107-109]. SAPs are a group of cross-linked polymers with the ultra-high water absorption capacity of over hundreds of times of its own weight [110]. SAPs are able to absorb water and expand to form an insoluble gel that acts as a barrier to the flow of the water. Afterward, SAPs can release the absorbed water within the concrete and prevent the shrinkage when the surrounding humidity drops. The mechanism of the water absorption of SAP is mainly the osmotic pressure arising from the difference in the concentration of ions between the gel and the surrounding solution [111]. In the application of internal curing, crosslinked poly-acrylic/acid-acrylamide molecule is the most commonly used type of SAP [112]. In Jensen et al.'s study, a high-performance cementitious composite blended with silica fume was designed with a water to cement ratio of 0.3 [113]. In this reference sample without the addition of SAP, shrinkage deformation was observed up to 3700μm/m at the age of 21 days. However, when SAP was added at a rate of 0.3 and 0.6wt% of binder, it was observed that the internal relative humidity of the concrete was effectively increased during the early age of the hydration process. The test result indicated that a 0.6wt% of SAP addition could fully eliminate the shrinkage [113]. Similar result was also found in other studies which proved that SAP can be used as an effective internal curing agent [107, 114].

In Hasholt et al.'s paper, it was reported that the overestimate of SAP water absorption capacity could result in an incorrect mixture design, where the extra water added for internal curing is too large. As a consequence, the increase of the actual water to cement ratio may result in a decrease of the concrete strength [115]. In Lee et al.'s study, it was observed that the concentration of the cations and the alkalinity of the environment can intensively influence the swelling behavior of the SAP. The study found out that the calcium ions are capable of binding with carboxylate groups in the acrylate chains of the SAP which further decreases the absorption capacity of the SAP [111]. Similarly, Zhu et al. reported that a stiff shell structure was formed when the SAP sample was immersed in the Al^{3+} solution. The presence of the barrier layer directly reduces the water absorption capacity of the SAP by hindering the water transportation between the SAP and the solution [112]. Therefore, it can be concluded that the behavior of the SAP can be affected by environmental conditions. It is necessary to understand the robustness of the SAP to optimize the mixture design when SAP is used as an internal curing agent.

2.4 Summary

According to the literature review, autonomous healing of the concrete is based on the artificially incorporated healing agents, while the autogenous healing is an inherent property of the concrete materials. Compared with autonomous healing, autogenous healing is more favorable for the in-field application since it will not increase the difficulty of the construction. However, it is also worth noted that autonomous healing allows the complete healing of cracks larger than several hundreds of micrometers, while the autogenous healing exhibited a less healing capacity of less than 100 μm. Therefore, the study about improving the efficiency of the autogenous healing using internal curing agent is necessary.

Previous researches indicate that the healing of crack with smaller width requires less amount of solid product which is more likely to be completed healed. Therefore, the efficiency of the autogenous healing performance could be improved by incorporating fibers to control the crack width propagation. On the other hand, according to the mechanisms of the autogenous healing, it can be noticed that water is a critical factor for the autogenous healing of the concrete materials. A sufficient self-healing phenomenon can only be observed when the external water is available.

However, external water supply is not continuous and unstable. Thus, the development of a method that can avoid the limitation of the external water supply will be beneficial to the autogenous healing efficiency. Internal curing is a well-developed method that supplies additional water for cementitious material to eliminate early age shrinkage. Internal curing agents with high water absorption capacity can be used as internal water reservoirs for cement hydration. Thus, with proper design, the internal curing agent has the potential to supply water for autogenous healing. There are various types of internal curing agents that have been studied. Among them, porous aggregate and superabsorbent polymer (SAP) have been widely used. However, as the direct experimental evidence is lacking, it is still unclear if the healing performance can be enhanced by the internal curing method. In addition, limited studies have been found on the robustness of the SAP in the fresh concrete mixture, which may potentially negatively affect the mechanical properties of the concrete materials.

3. EXPERIMENTAL PROCEDURES

3.1 Material and sample preparation

To study the influence of fiber parameter on the mechanical properties of the concrete, PVA fiber (Polyvinyl Alcohol) and two different types of macro-fiber approved by Indiana Department of Transportation (INDOT), Strux 90/40 fiber and Masterfiber Mac Matrix fiber were used to reinforce the designed cementitious composite. The material properties of the fibers are shown in Table 1.

To verify the hypothesis that the internal curing agent can improve the autogenous healing performance, two types of internal curing aggregates, clinoptilolite zeolite (KMI Zeolite Inc., USA) and expanded shale lightweight aggregate (US Aggregates, USA), were used to act as internal water reservoirs to supply water. Internal curing aggregates are designed to partially replace natural sand (US Aggregate, USA) with two replacement ratios of 15% and 30%. The water absorption capacities of the natural sand, zeolite and lightweight aggregate were tested according to the ASTM C128 [116], and the results are 1.6%, 18.00% and 13.64%, respectively. The particle size distribution of the natural sand and internal curing aggregates are shown in Fig. 1. The other ingredient that used in the mixture included Type I Ordinary Portland cement (OPC, Buzzi Unicem, USA), Grade 100 blast furnace slag (Skyway cement company LLC, USA), Class C fly ash (fly ash direct, ltd.- Joppa power plant, USA), natural sand and tap water. The mix composition is listed in Table 2. The use of supplementary cementitious material intends to potentially increase the amount of un-hydrated cementitious material during the self-healing period. In order to achieve high mechanical performance, relatively low water to binder ratio of 0.35 was used. 2% volumetric fraction of fiber was incorporated to control the crack width and enhance the ductility of the designed material.

Table 1 Material properties of the fibers

Fiber name	Specific gravity	Length (in)	Tensile strength (MPa)	Aspect ratio
Masterfiber Mac Matrix	0.91	2.1	585	70
Strux 90/40	0.92	1.55	620	90
PVA	1.3	0.3	1600	205

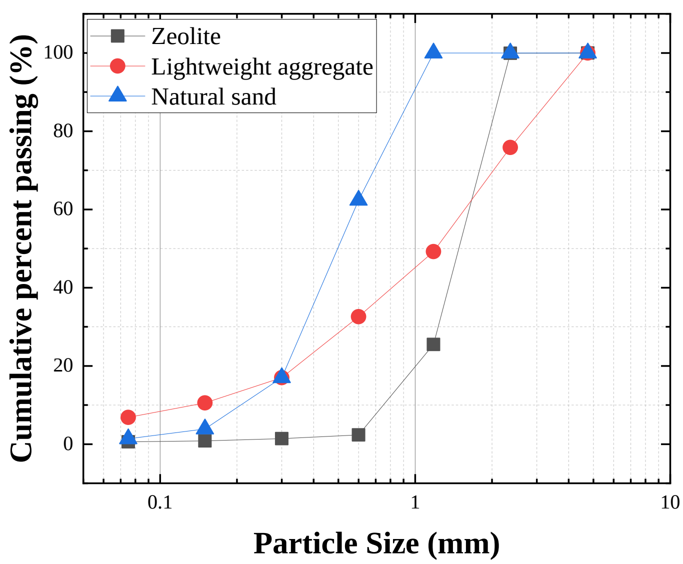

Fig. 1 The particle size distribution of natural sand and internal curing aggregates

As shown in Table 2, mixture R_(name of fiber) represents the reference mixture with different types of fiber. Mixtures with initials “Z” means incorporated with zeolite as curing aggregate, while “L” indicates lightweight aggregate. Internal curing aggregates were pre-wetted by tap water for 48 hours before mixing. The extra free water carried by the aggregate was calculated and the amount of mixing water was adjusted. The cementitious materials, sand and internal curing aggregates were firstly dry mixed for 3 mins at low speed to reach a uniform state. After that, water was added and mix for 3-5 mins until the mixture reached good fluidity and uniformity. Finally, the fibers were slowly added into the mixture at low mixing speed within 1 min, followed by a medium speed mixing for 3 mins to achieve good fiber dispersion. The fresh cementitious materials were then cast into 50 mm cubic molds for the compressive test, 240 mm × 60mm × 15mm plate for four-point bending test, and dog-bone specimens (Fig. 2) for uniaxial tensile test and self-healing evaluation, respectively. The samples were de-molded after 24 hours and cured in a 100% relative humidity chamber for 28 days.

Table 2 Mixture design (by weight of cement)

Mix	OPC-I	Blast furnace slag	Fly ash	w/b*	Fiber (volume %)	Zeolite	Lightweight aggregate	Sand	Replaced (%)+
R_PVA	1	0.15	0.15	0.35	2% PVA	0	0	0.80	0
R_Strux	1	0.15	0.15	0.35	2% Strux 90/40	0	0	0.80	0
R_MasterFiber	1	0.15	0.15	0.35	2% MasterFiber	0	0	0.80	0
Z_15	1	0.15	0.15	0.35	2% PVA	0.12	0	0.68	15%
Z_30	1	0.15	0.15	0.35	2% PVA	0.24	0	0.56	30%
L_15	1	0.15	0.15	0.35	2% PVA	0	0.12	0.68	15%
L_30	1	0.15	0.15	0.35	2% PVA	0	0.24	0.56	30%

*: water-to-binder ratio

+: Internal curing aggregate replaced sand rate (weight %)

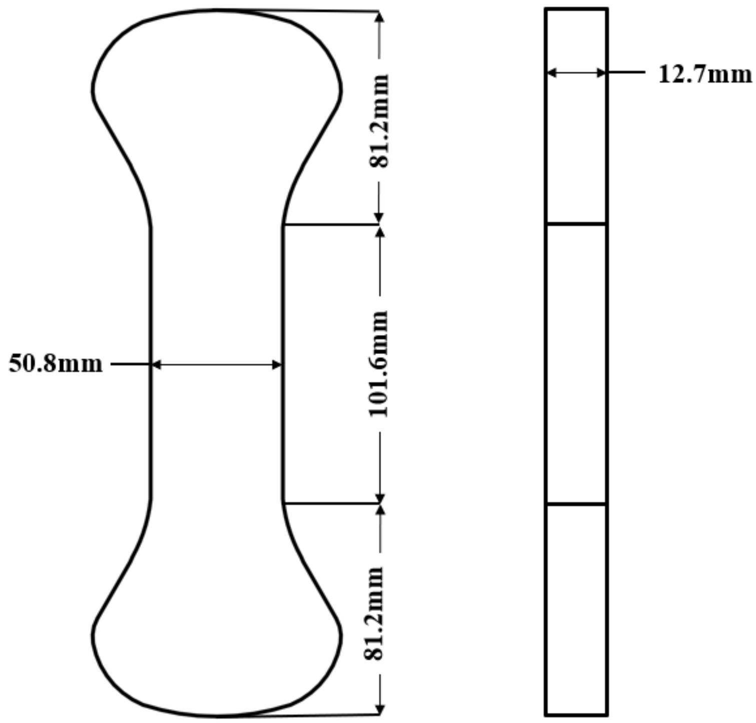

Fig. 2 Dog-bone specimen for uniaxial tensile loading

3.2 Mechanical tests

The Universal Tensile Machine (MTS Insight 10) was used to conduct the mechanical tests. The 28th day compressive and flexural strengths for each mixture were determined by five cubic and plates samples according to ASTM C109 and ASTM D6272, respectively. For the uniaxial tensile test, a customized fixture was used, as shown in Fig. 3. The loading rate was controlled at 0.5mm/min to simulate the quasi-static loading condition. During the flexural test, a linear variable displacement transducer (LVDT) was used to monitor the midspan deflection of the sample, as shown in Fig. 4. The result of the mechanical tests was averaged from the five replicates of each mixture.

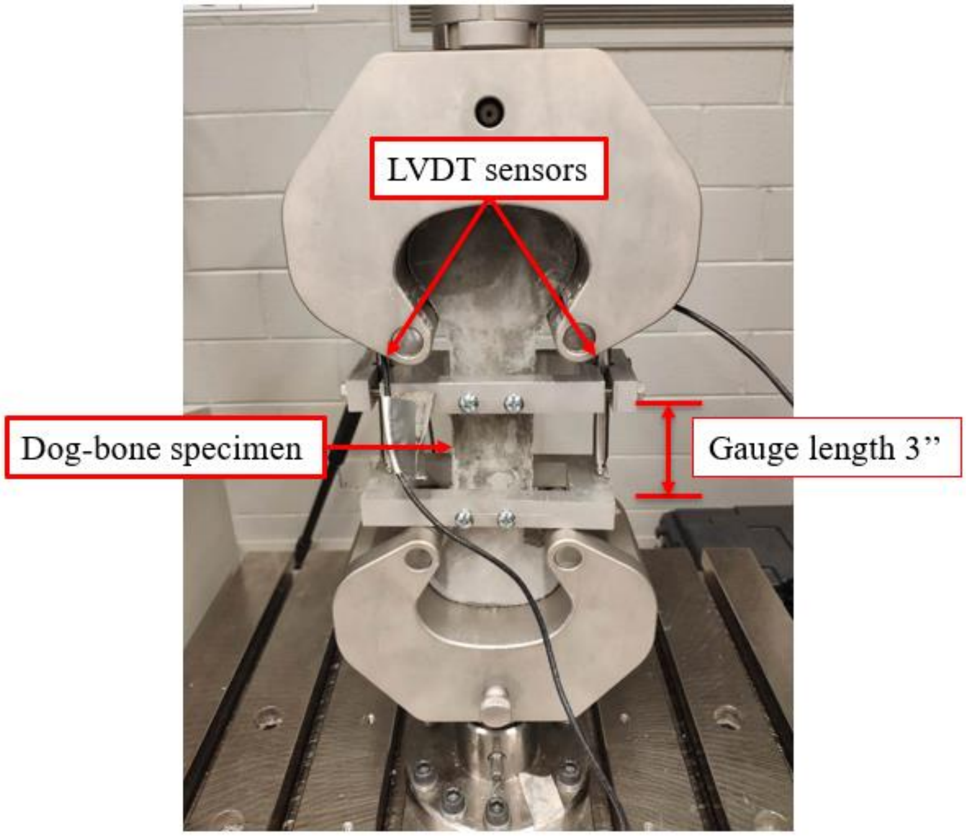

Fig. 3 Dog-bone specimens and setup for uniaxial tensile test

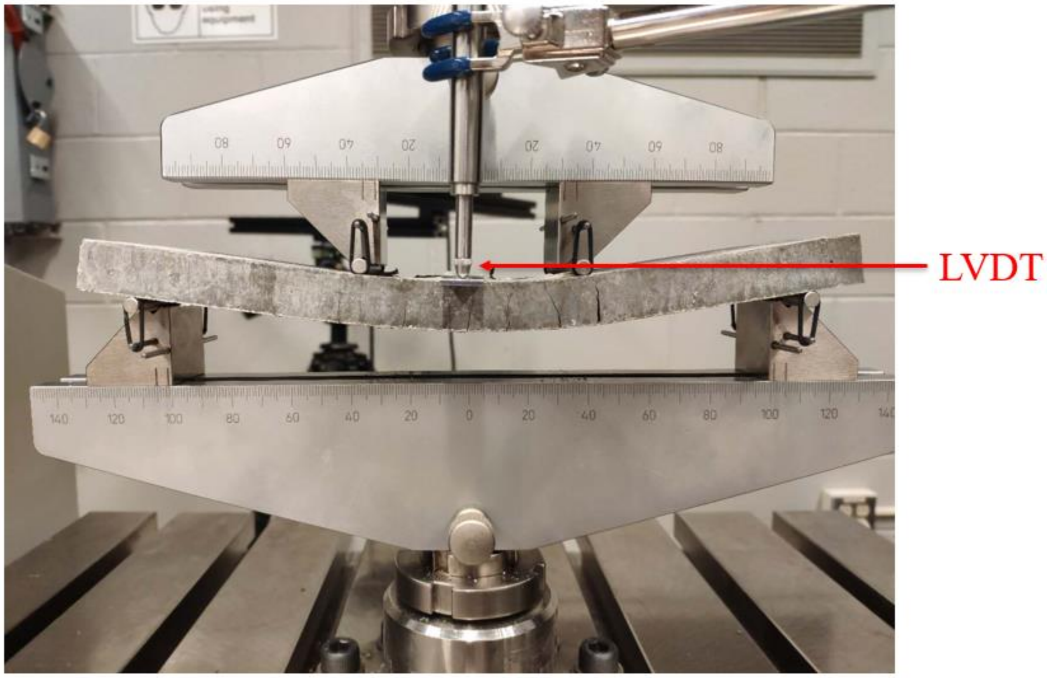

Fig. 4 Experimental setup for flexural test

3.3 Sample pre-cracking and wet/dry cycles

To evaluate the autogenous healing performance of the sample, designated level of damage was induced to the sample. A tensile strain of 0.5% was chosen as the pre-loading level to induce cracking on the specimens. During the loading process, two linear variable displacement transducers (LVDTs) were mounted on the specimen to monitor the deformation (as shown in Fig. 3). After the pre-loading, samples were exposed to wet/dry cycles to simulate the natural environment of raining and sunny, which has a discontinuous water supply. Each wet/dry cycle includes two days curing at 100% relative humidity, and another two days at 50% relative humidity. The total healing period was 28 days (7 cycles). The experimental procedure is shown in Fig. 5.

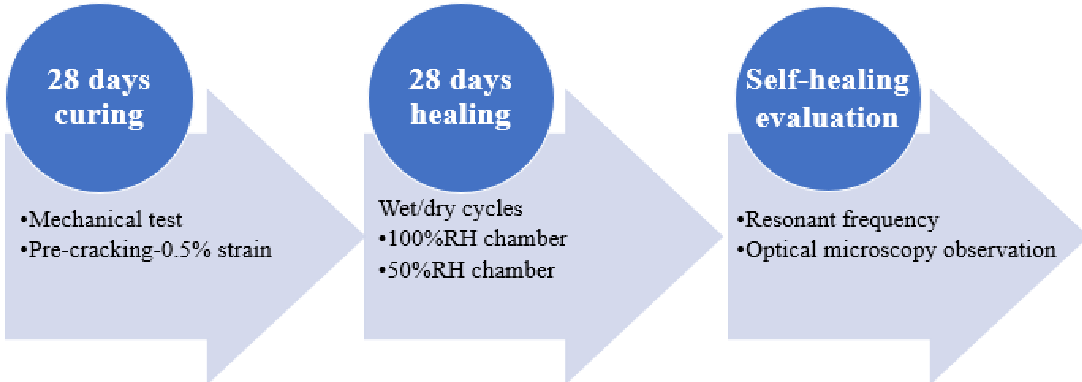

Fig. 5 Experimental procedure for autogenous healing evaluation

3.4 Autogenous healing evaluation

Resonant frequency test was conducted to evaluate the autogenous healing performance of the designed concrete mixture. The measurement of resonant frequency is based on ASTM C215 [117]. It has been widely accepted as a promising and simple method to evaluate the self-healing performance of the cementitious composite [32, 49, 118]. Li et al. [119] correlated the relationship between changes in resonant frequency signal and applied strain level of the cementitious composite. It proved that resonant frequency measurement can quantify the degree of damage, and therefore the extent of self-healing as well. The resonant frequency of the sample was measured before and after pre-loading, and after the healing period, denoted as N_{virgin}, $N_{pre\text{-}damaged}$, and N_{healed}, respectively. The experimental setup consists of a piezoelectric oscilloscope (GrindoSonic Mk5, Penn Tool Co), vibration detector, wooden gavel, and sponge support. In previous work, the recovery ratio, calculated by Equation (5), was proven to be effective as an index to represent the self-healing performance of cementitious materials [4]. The resonant frequency reduced due to the introduction of cracks and recovered when the cracks healed. Thus, the recovery ratio is capable of indicating the degree of autogenous healing.

$$\text{Recovery ratio (\%)} = \frac{\mathrm{NDT_{Healed}} - \mathrm{NDT_{Pre-damaged}}}{\mathrm{NDT_{Virgin}}} \times 100\% \quad (5)$$

The closing of the cracks was examined through an optical microscope. By using image analysis software (Image J), the sealing of the crack mouth can be observed and quantified. For each crack on the specimen, test points were taken at an equal distance of 200μm. The optical microscopic image of cracks was compared before and after 7 wet/dry cycles curing. Changes in the crack width for all the test points were analyzed before and after the healing period. Crack width reduction ratio (Equation 8) was used to represent the healing extent of the crack mouth. The result is shown in **Error! Reference source not found.**.

$$\text{Reduction ratio (\%)} = \frac{\text{Original crack width} - \text{Healed crack width}}{\text{Original crack width}} \times 100\% \quad (8)$$

3.5 Evaluation of the water retention behavior

Water desorption test was conducted to compare the water desorption profile of zeolite and LWA. Sufficient amounts of zeolite and LWA were firstly pre-soaked in de-ionized (DI) water separately for 48 hours, and later, the weight of saturated surface dry (SSD) condition was obtained by following the ASTM C128 [116]. Then, the internal curing aggregates were placed in the chamber with 50% relative humidity and temperature of 23.5℃. The weight of the aggregates was periodically recorded until 24 hours, afterward, the samples were put into a chamber with 10%

relative humidity. The changes in the weight were monitored for another 24 hours. The normalized moisture content defined as $\frac{Mass\ Water}{48h\ Water\ absorption}$, is used to quantify the extent of the water desorption. A normalized weight of 1.0 represents the saturated internal curing aggregate.

To evaluate the influence of internal curing and porous aggregates on the absorption capacity of bulk hardened samples, the self-healed samples were submerged into water. The surface dry (SD) weight of the samples was measured after 24 and 48 hours of immersion to evaluate the weight gain.

3.6 SEM observation for internal curing aggregates

The pore structure of internal curing aggregates was investigated by Scanning electron microscope (SEM). The surface of internal curing aggregates was coated with gold to enhance the surface conductivity before SEM analysis. Then the surface was observed by using a scanning electron microscope (FEI Nova NanoSEM 450) to characterize the microstructure of internal curing aggregates.

3.7 Evaluation of the robustness of SAP

In order to prevent the overestimation of the absorption capacity of the SAP in concrete mixture, the robustness of the SAP was evaluated. The SAP used in this study is a commercially available SAP Floset 27. Various dosages of the superplasticizer, hydration accelerator and fly ash were designed, and the absorption behavior of the SAP was tested by the filtration test and void size analysis.

3.7.1 Filtration test

A filtration test was performed to evaluate the influence of superplasticizer and hydration accelerator on the water absorption behavior of SAP. The concentration of the SP and accelerator were ranged from 0 to 2.0wt% and 0 to 1.0wt%, respectively. A constant mass of the SAP of 0.2g was prepared for each test. The test set up is shown in Fig. 6. The SAP was filtrated by a funnel after 15mins soaking in the 100mL DI water with different concentrations of the superplasticizer or hydration accelerator. A pump was used to provide a constant pumping pressure to accelerate the filtration process. The absorption capacity of the SAP was calculated by Equation 6:

$$\text{Absorption capacity } (g/g) = \frac{m_{liquid}}{m_{SAP}} \quad (6)$$

Where Δm_{liquid} is the mass of the liquid that is absorbed by the SAP and the m_{sap} is the original mass of the SAP that adds into the solution.

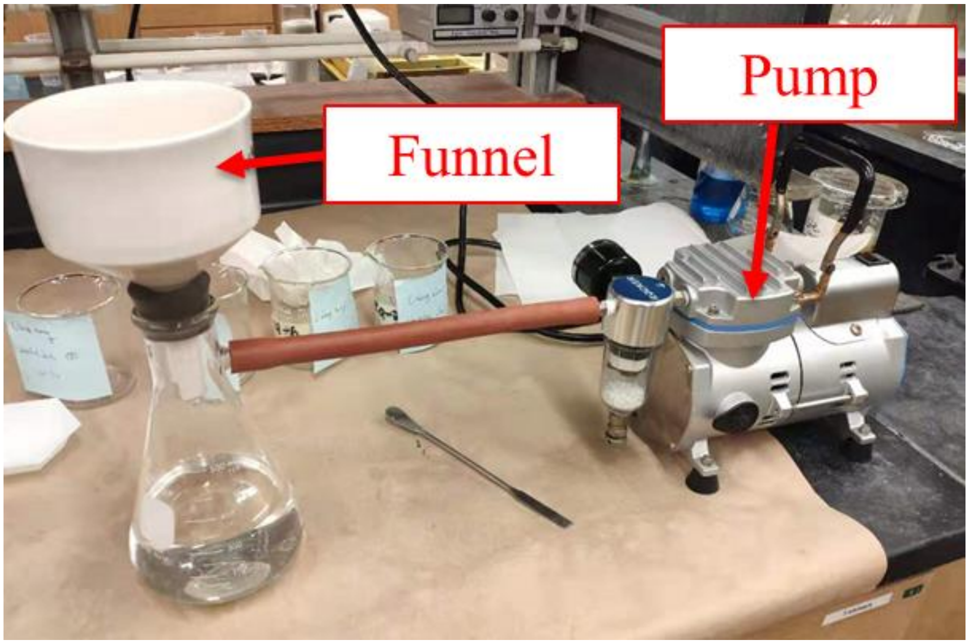

Fig. 6 Experimental setup for filtration test

3.7.2 Void size analysis

Void size analysis was conducted to analyze the size of the pores that leave by SAP after it releases the absorbed water. The void size left by the SAP can be used as an index to represent the absorption behavior of the SAP during the mixing and curing process. Different dosages of superplasticizer, hydration accelerator or fly ash were designed. Samples with four fly ash to cement contents (FA/CEM) range from 0 to 1.2 were prepared. Based on the technical data sheet provided by the manufacturers, the designed dosages for superplasticizer and accelerator was ranged from 0 to 2wt% and 0 to 1.79wt%, respectively. All the sample was incorporate with 0.25w% of the SAP and the water to cement ratio was 0.4. The samples were de-molded after 24 hours and cured in the laboratory condition for 7 days.

After the curing, the samples were cut into 25.4 mm cubic by a diamond saw, and calcinated under 600℃ for 1 hour to decompose the SAP particles. Then, acetone was used to clean the surface of the sample. To enhance the contract between the void and the matrix under the microscope, the surface of the sample was colored by black marker, and then pre-treated by the barium sulfate powder. To evaluate the average void size on the surface of the sample, the microscopic images were analyzed by the software Image J.

3.8 Water flow test for self-sealing evaluation

A water flow test was designed to evaluate the self-sealing performance of the SAP in concrete materials. Cement paste with 1 wt% of the SAP was cast into 76.2 mm×152.4 mm cylinder and cured in the laboratory environment for 7 days. A reference sample without the addition of SAP was also prepared. The water to cement ratio of the mixtures was 0.4. After the 7 days curing, splitting tensile loading was conducted to pre-crack the sample. The crack width was controlled by two steel rings as 0.16mm. The experimental setup includes a water tank, PVC tube, rubber tube, balance and computer, as shown in Fig. 7. The water tank can continuously supply water, while a rubber tube connected to the PVC tube controls the water level to make sure the constant of the water pressure to the testing sample. Balance and the computer are used to monitor and record the weight of water passing through the sample with time.

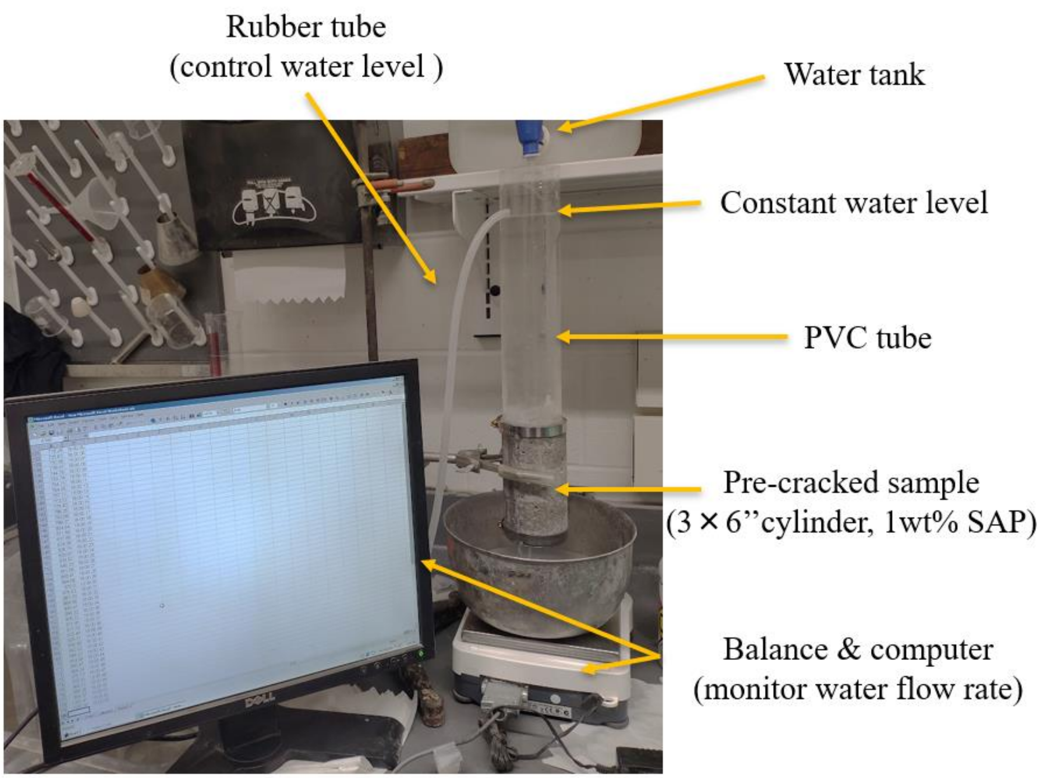

Fig. 7 Experimental setup for water flow test

3.9 Flow chart

In this section, a flow chart for the design of the experimental work is presented. As can be seen in the Fig. 8, three types of fibers were firstly evaluated by mechanical tests. The crack width control abilities of these fibers in the cementitious composite were analyzed and compared based on the result of the flexural test. Secondly, two types of porous internal curing aggregates were separately incorporated into the mixture. The mechanical properties of the sample were evaluated in order to examine the functionality of the concrete material after the incorporation of porous particles. In the meanwhile, part of the samples was pre-cracked, and the self-healing performance of the cementitious composite was investigated by the resonant frequency measurement and optical microscopic observation. To understand the role of internal curing aggregates in the self-healing process, the bulk absorption capacity of the bulk sample was tested. And the pore structure of the internal curing aggregates was investigated by the SEM analysis and water desorption test. Thirdly, the robustness of the SAP was evaluated by the filtration test and void size analysis in order to understand the stability of the absorption capacity of the SAP under various conditions. The water flow test was then conducted to investigate the self-sealing effect of SAPs.

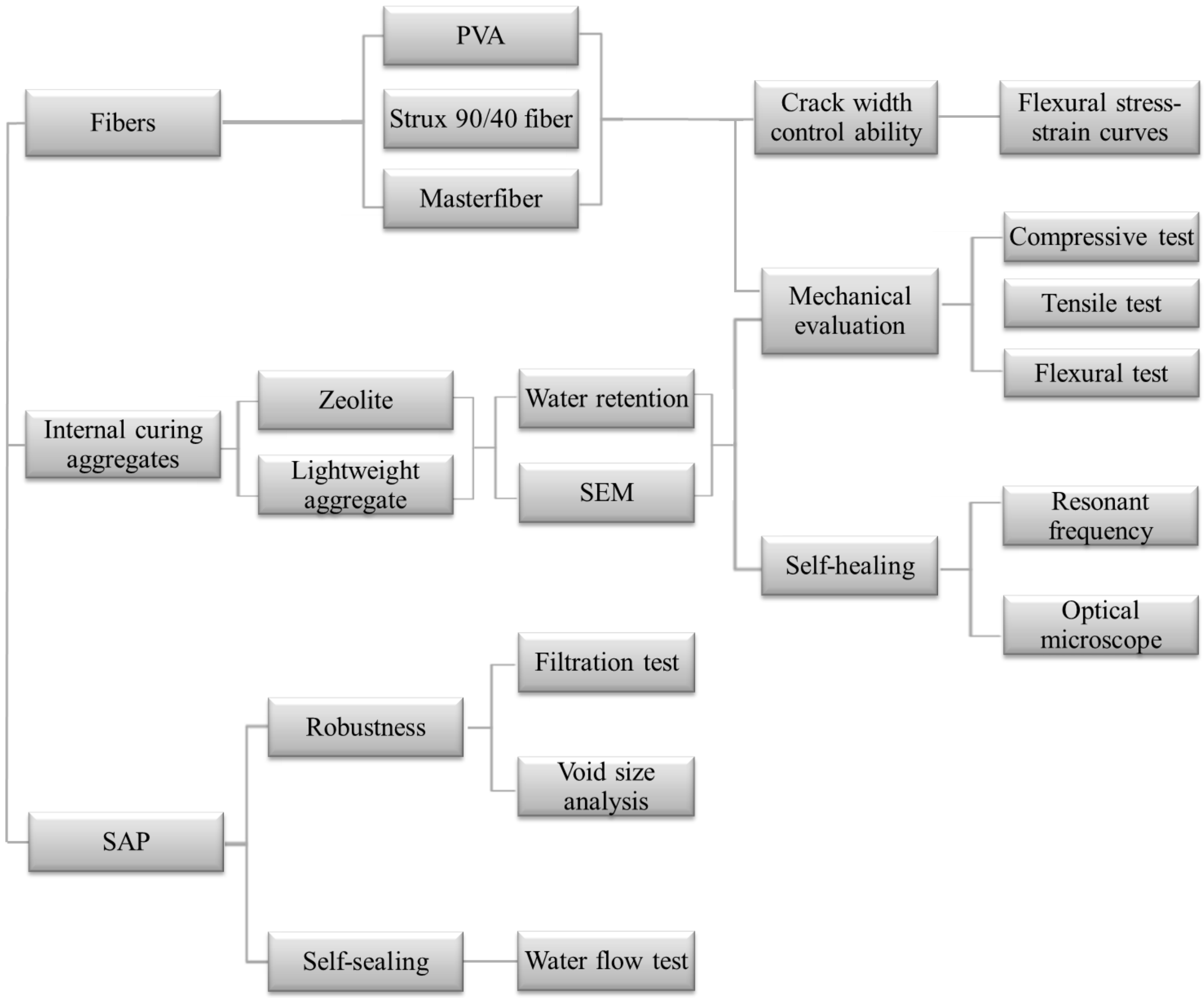

Fig. 8 Flow chart for the experimental design

4. RESULT AND DISCUSSION

4.1 Fiber evaluation and crack width control ability

4.1.1 Mechanical performance

The mechanical properties of the designed cementitious composite with different types of fiber are shown in Table 3. It can be observed that samples with PVA fiber and Masterfiber exhibited higher compressive strength of 60.73MPa and 55.4MPa, respectively, while the sample with Strux 90/40 fiber showed a compressive strength of 39.8MPa. As for the uniaxial tensile test, the incorporation of PVA fiber led to the highest uniaxial tensile strength of 3.94MPa, while the sample with Strux 90/40 fiber and Masterfiber show a tensile strength of 2.39MPa and 2.95MPa, respectively. For the flexural test, samples with PVA fiber and Strux 90/40 fiber exhibited a similar strength level of 9.03MPa and 8.60MPa, respectively. The use of Masterfiber resulted in a flexural strength of 6.8MPa, which is 25.1% lower than the sample with PVA fiber.

It can be observed from the result that, overall, the sample with PVA fiber exhibited better mechanical performance, compared with the other two types of fiber. There are three underlying reasons for this phenomenon. Firstly, PVA fiber has a higher tensile strength. Upon the formation of the crack during the loading process, PVA fiber can transfer a higher level of stress across the cracks. Secondly, PVA fiber has a short length of 0.3 inches, compared with Strux 90/40 fiber and Masterfiber of 1.55 and 2.1 inches, respectively. When longer fiber is used, the fiber entanglement is more likely to happen during the mixing process. Thus, the poor distribution of the fiber could lead to a worse mechanical performance of the concrete material. Thirdly, the aspect ratio of the fibers can affect the mechanical strength of the concrete. Various studies have proved that a higher aspect ratio of the fiber potentially leads to a higher strength of the concrete material [69, 70, 120]. It was reported that a high aspect ratio could delay the debonding of the fiber from the cementitious matrix during the loading process [121]. Since the aspect ratios of the PVA is much higher than the other two types of fiber that used in this study, better mechanical performance can, therefore, be expected for the sample with PVA fiber.

Table 3 Result of the mechanical tests

Mechanical Properties / **Fiber used**	Compressive strength (MPa)	Uniaxial tensile strength (MPa)	Flexural strength (MPa)
PVA	60.73 (1.43)	3.94 (0.32)	9.03 (0.52)
Strux 90/40	39.80 (0.8)	2.39 (0.58)	8.60 (1.15)
MasterFiber Mac Matrix	55.4 (1.0)	2.95 (0.33)	6.8 (1.04)

* Items in parentheses (): standard deviation.

4.1.2 Flexural stress-strain curves

During the flexural test, the midspan deflection of the sample was monitored by a linear variable displacement transducer (LVDT). According to ASTM-D6272, the flexural strain was calculated by equation (7):

$$\text{Strain} = \frac{4.70 * D * d}{L^2} \qquad (7)$$

where D is the maximum midspan deflection (in), d is the depth of the beam (in) and L is the support span of the sample (in).

The flexural stress-strain curves of the tested material are shown in Fig. 9. It can be observed that the sample incorporated with PVA fiber showed a stronger flexural strength, while the use of Strux 90/40 fiber and Masterfiber led to a higher flexural strain capacity. Unlike the brittle behavior of the conventional concrete, mixtures tested in this study exhibited pseudo-strain-hardening behavior during the loading process. As can be seen in the stress-strain curve, every sudden drop of the flexural stress represents the formation of a new crack [122, 123]. In Fig. 9, after the formation of a crack, the stress dropped and then continued to increase, rather than drastic decrease and leads to the failure. The reason for this phenomenon is that the fibers across the crack successfully acted as bridges to transfer the stress, which prevented the uncontrollable crack propagation.

As for the crack width control ability, it can be observed from Fig. 9 that for the sample with Strux 90/40 fiber, totally 5 cracks were formed during the flexural loading. In addition, the formation of the cracks mostly limited within the early loading process when the flexural strain is below 0.00015, which indicates that the crack width propagated without effective limitation throughout the loading process A wider crack opening can thus be expected. A similar phenomenon was also observed from the sample with the Masterfiber. On the other hand, for the sample with PVA fiber, a formation of more than 10 cracks can be observed from the flexural stress-strain curve. The formation of cracks in the sample with PVA fiber was continuous and evenly distributed throughout the loading process. In spite of a lower flexural strain capacity, a better fiber bridging and crack width control performance can be expected.

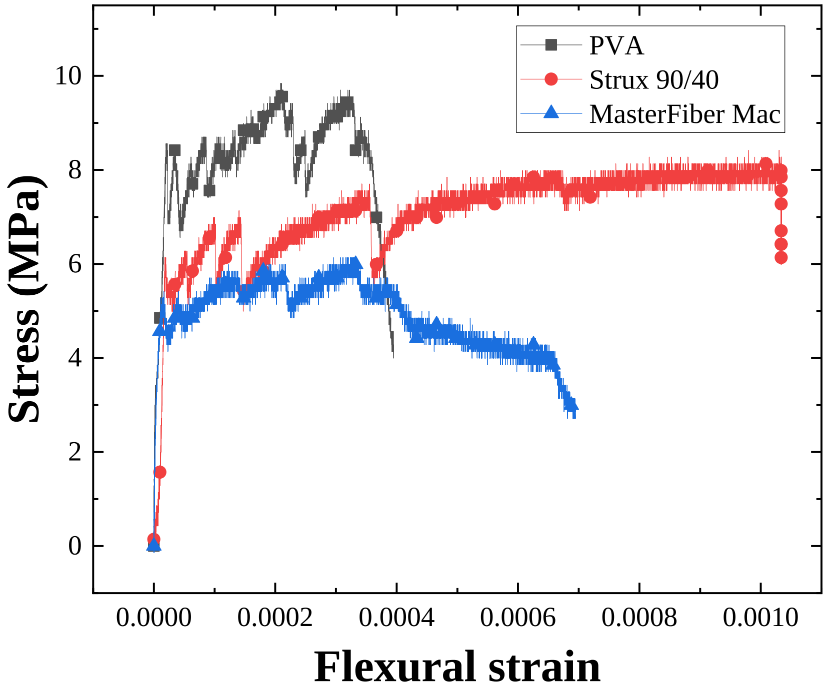

Fig. 9 Flexural stress-strain curves of the tested materials

4.1.3 Summary

In this section, three types of fiber were used in the designed cementitious composite. Mechanical properties were tested, and the crack width control abilities of the fiber were evaluated by the flexural stress-strain curves. The result shows that the sample with PVA fiber exhibited higher mechanical strength and stronger crack width control ability, while the incorporation of Strux 90/40 fiber could lead to the highest flexural strain capacity. In this thesis, the goal is to design a fiber-reinforced cementitious composite with autogenous healing ability. It is favorable to choose a fiber with stronger crack width control ability into the mixture, which could potentially increase the efficiency of the self-healing. Therefore, PVA fiber is more suitable for this specific study and will be used in the following study about the evaluation of the autogenous healing performance.

4.2 Influence of internal curing aggregate and autogenous healing evaluation

(this part of study will be published in a journal)

4.2.1 Mechanical performance

After 28 days of moisture curing, compressive and uniaxial tensile test were conducted for the sample. Fig. 10, Fig. 11 and Fig. 12 present the compressive, flexural and uniaxial tensile test results, respectively. The mixture R_PVA represent the reference sample without the incorporation of the internal curing aggregate. It can be observed that the incorporation of internal curing aggregates slightly increased the compressive and flexural performance while the tensile strength remained largely the same. During the curing period, the extra water released by internal curing aggregates increased the hydration degree of cementitious material to enhance the interface transition zones in cementitious composite [104, 124, 125], which led to a higher mechanical strength. Moreover, by comparing the two types of internal curing aggregates, the sample with zeolite exhibited a more pronounced improvement in compressive and flexural strength. The phenomena may be attributed to the higher water absorption capacity of zeolite. A considerable amount of water was supplied by zeolite that leading to a denser cementitious matrix, which is favorable for mechanical behavior. When it comes to the uniaxial tensile test, the incorporation of zeolite or lightweight aggregate was expected to be detrimental to tensile performance because of the larger particle sizes of internal curing aggregates. The porous aggregates can act as flaws in the cement matrix when uniaxial tensile loading was applied. The higher hydration degree due to the internal curing compensated the negative effect of adding large internal curing aggregates.

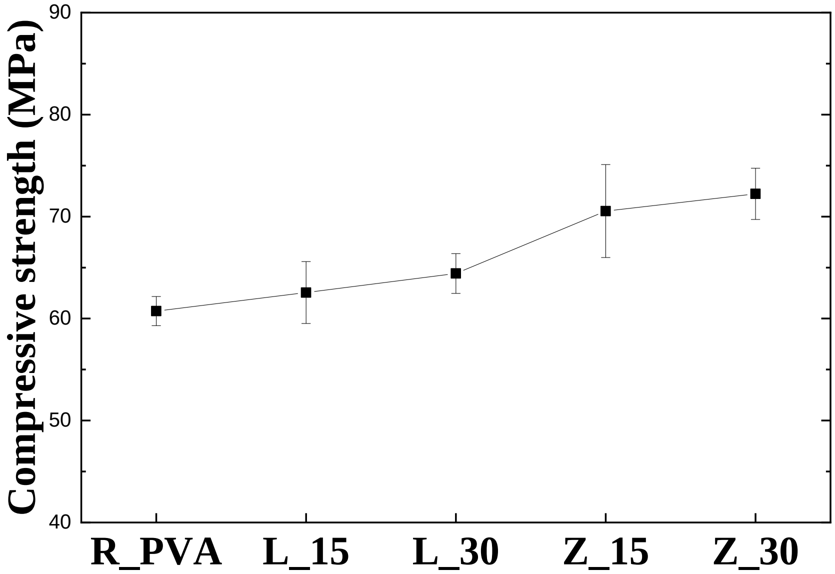

Fig. 10 Result of 28 days compressive test

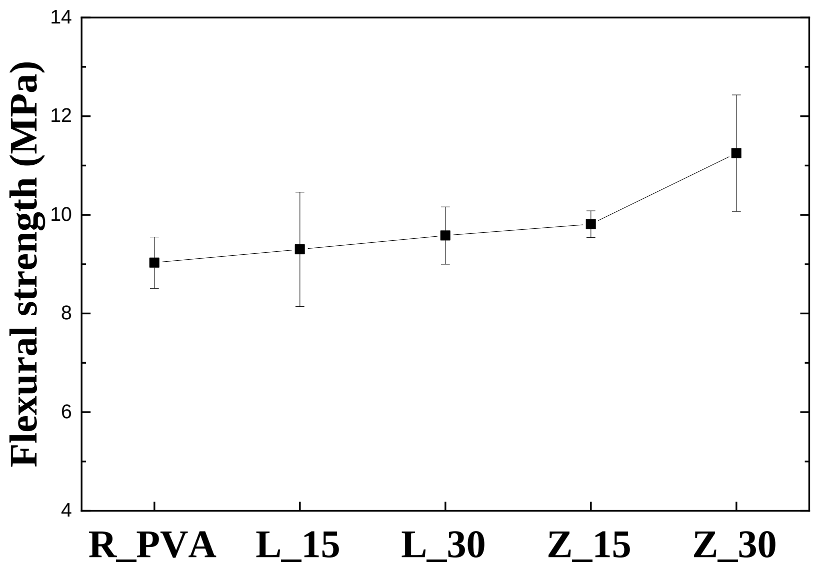

Fig. 11 Result of 28 days flexural strength

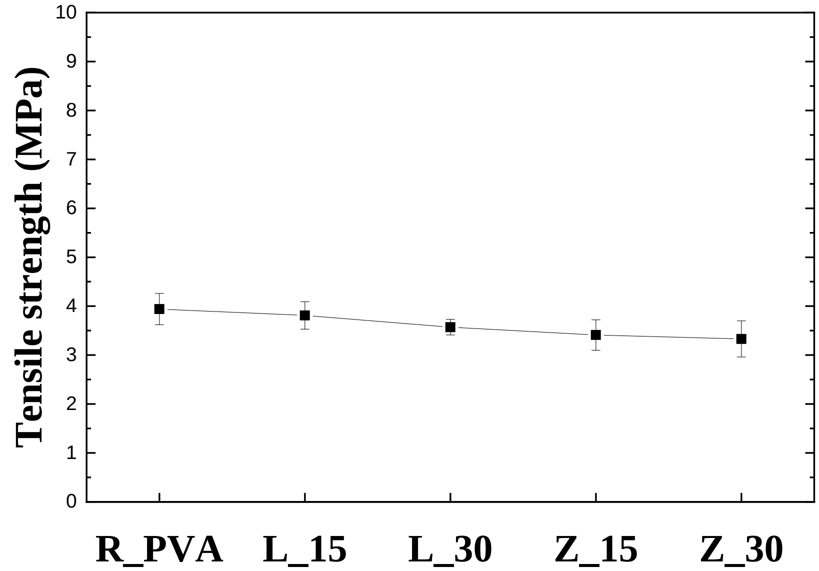

Fig. 12 Result of 28 days uniaxial tensile test

4.2.2 Autogenous healing evaluation

As shown in Fig. 13, after 28 days of healing, all the samples showed certain levels of healing. Among this, samples with internal curing aggregates exhibited a more significant self-healing ability compared with the reference group. Specifically, while the R_PVA had 12.6% of the recovery ratio, the sample L_15, L_30, Z_15, and Z_30 showed a higher recovery ratio of 18.3%, 23.8%, 25.3% and 21.3% respectively. The recovery ratio increased with the increase in the replacement level of lightweight aggregates. The effect of zeolite was somewhat more complicated. It had the highest recovery ratio at the replacement level of 15%, but there was a decrease in recovery ratio at the replacement of 30%, which will be discussed later.

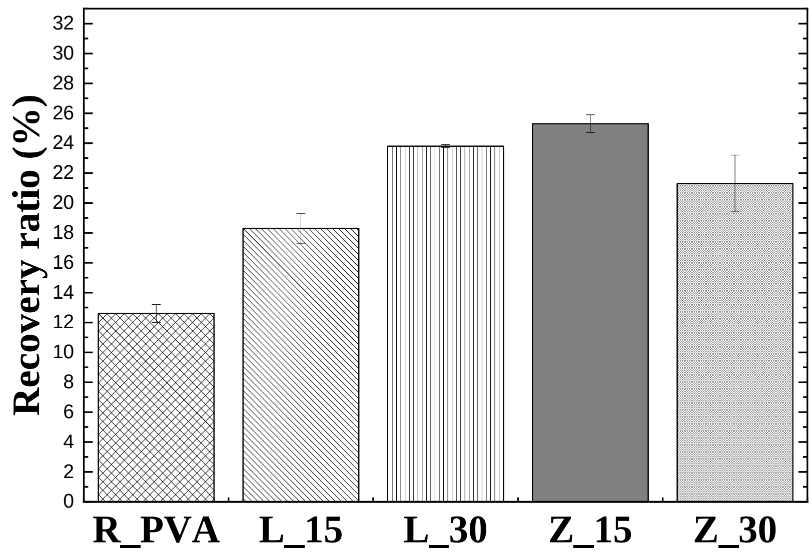

Fig. 13 Recovery ratio of designed materials based on resonant frequency measurement

According to the result of the morphology observation, as shown in Table 4, all the mixtures showed complete sealing for crack widths below 20 μm due to the self-healing behavior. For R_PVA, the reduction ratio dropped to around 40% at the crack width of 20~40 μm and remained at a low level of around 20% above 40 μm. With the incorporation of pre-wetted internal curing aggregates, it effectively increased the crack width reduction ratio for all crack width ranges. Z_15 achieved the best healing performance among all groups. The reduction ratio was almost two times higher than the other groups at 20~30 μm. Even above 50 μm, it still retained around 50% of the crack width reduction ratio.

Table 4 Crack width reduction ratio of different mixtures

Crack width range(μm)	R_PVA	L_15	L_30	Z_15	Z_30
0~10	100	100	100	100	100
10~20	100	100	100	100	95.56
20~30	43.52	44.24	44.73	88.61	48.17
30~40	42.79	40.96	55.73	59.69	50.3
40~50	25.27	27.70	31.67	46.28	33.96
>50	20.85	34.09	23.64	49.09	31.28

4.2.3 Water retention characterization

To measure the absorption capacity of bulk hardened samples, after the evaluation of self-healing, the samples were submerged into water. The surface dry (SD) weight of the samples was measured after 24 and 48 hours of immersion to evaluate the weight gain. As shown in Fig. 14, the sample with internal curing aggregates exhibited a higher absorption capacity compared with the reference mixture. It can be seen that the bulk sample absorption capacity increased when the replacement ratio of LWA increases from 15% to 30%. However, as the zeolite's content increased, the absorption capacity decreased otherwise. The possible explanation is presented in section 4.2.5.

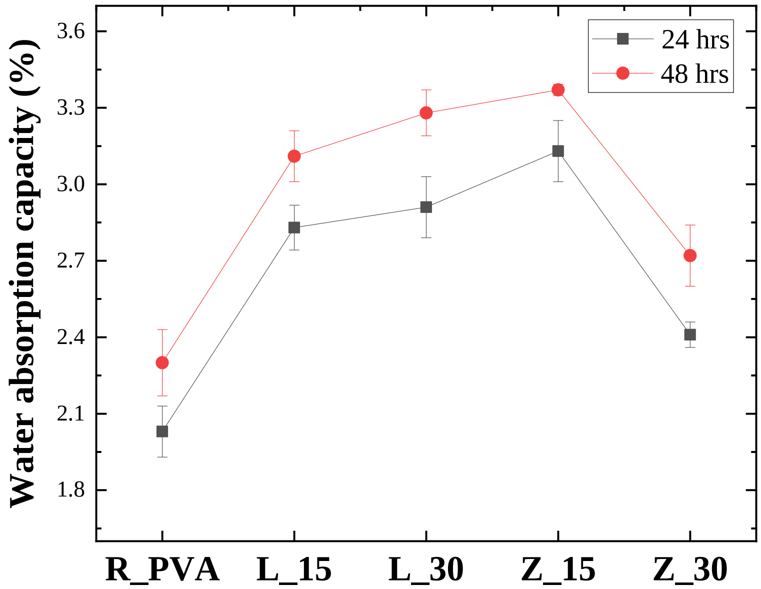

Fig. 14 Bulk sample water absorption capacity

The result of the water desorption kinetic test for internal curing aggregates is shown in Fig. 15. It can be seen that zeolite and LWA reached equilibrium after 8 hours at a relative humidity of 50%. Furthermore, 80% and 90% of their absorbed water was released, respectively. It is expected since LWA and zeolite have been used to supply water for the reduction of the early age shrinkage, a high release rate of water is required under such level of relative humidity [126]. Afterward, when samples were moved to the chamber with 10% relative humidity, zeolite released another 10% of its absorbed water while LWA stayed the same. The result implies that zeolite had a higher water retention capacity compared with LWA. It is likely that zeolite stored part of its absorbed water under the ambient environment and released it under extremely low humidity.

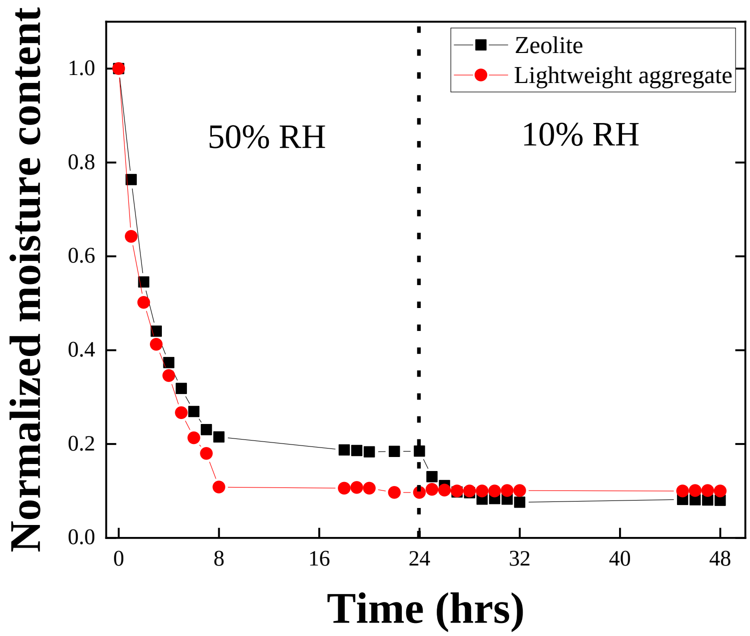

Fig. 15 Water desorption behavior of internal curing aggregates

4.2.4 SEM of internal curing aggregates

The SEM images of LWA and zeolite were shown in Fig. 16. The highly porous structure can be seen for both zeolite and LWA, which suggests the high water absorption capacity. In general, the water absorption process in aggregate is caused by capillary effect [127]. During the hydration process, the larger pores tend to lose the water first [90, 128]. A finer pore structure can lead to a stronger capillary force that tends to retain the water. By comparing SEM images of LWA and zeolite, the size of the pores in zeolite is clearly smaller than LWA. Thus, a slower water release rate can be expected for zeolite, which verifies the water desorption results, as shown in Fig. 15. In contrast, the open void and the connection between pores can be observed in LWA. Therefore, a high water transport efficiency with rapid water absorption/desorption rate in LWA can be expected [129] and was observed in Fig. 15.

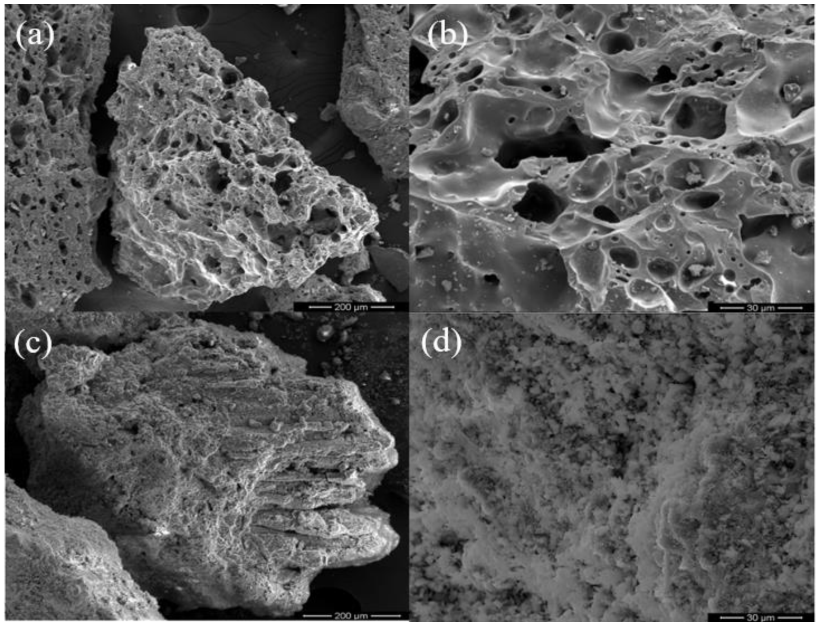

Fig. 16 SEM images of internal curing aggregates.
(a)(b): Lightweight aggregate; (c)(d): Zeolite.

4.2.5 Mechanisms of the improvement in healing performance

In this section, the mechanism of the improved self-healing of internal curing aggregates is discussed with the help of the water absorption capacity of bulk samples, water desorption results, and SEM.

There are three primary mechanisms for autogenous self-healing. Firstly, the further hydration of un-hydrated cementitious materials [32]. The reactions between water and cement grains produce amorphous calcium silicate hydrate (C-S-H) gel and calcium hydroxide ($Ca(OH)_2$) on crack mouth that can seal the crack [130]. Besides, un-hydrated supplementary cementitious materials in the concrete system can hydrate and transform $Ca(OH)_2$ into more stable C-S-H gel when water is available. Secondly, another important reason for autogenous healing is the formation of calcite from the carbonation process. When the external water is available, concrete specimens will release Ca^{2+} ions into the water [131]. In the meanwhile, CO_2 in the air dissolves into the water; then the CO_3^{2+} ions can combine with Ca^{2+} and lead to the formation of $CaCO_3$ precipitates [30]. The other mechanism for autogenous healing is the leaching and recrystallization of portlandite. Calcium hydroxide can transport with water to the surface of the crack mouth. Then, when the environmental humidity is low, the loss of water will cause the recrystallization of the calcium hydroxide [132].

The formation of healing products is capable of sealing the crack. In each mechanism mentioned above, it could be noticed that water is an indispensable factor that could directly affect the intensity of self-healing. Thus, the availability of water is critical for the improvement in autogenous healing performance.

We observed that water absorption capacity correlates very well with the self-healing ability of studied systems. A higher capacity of water storage capacity coincides with better self-healing performance. The addition of porous zeolite and LWA tends to increase the bulk absorption capacity of the sample (Fig. 14), which makes it more likely to absorb and store part of the external water when it is under wet curing condition [133, 134]. Consequently, this part of the stored water can be slowly released for continuous secondary hydration and precipitation of the calcium carbonate when the external water is no longer available. Besides, if we take a closer look at the effect of zeolite when the content of zeolite increased from 15% to 30%, the water absorption capacity decreased, and its decreasing water absorption compromised the healing performance. Considering the water absorption capacities of zeolite and LWA are 18.00% and 13.64%, respectively, mixture Z_30 is expected to have the highest water absorption capacity. However, the test result shows that this is not the case. In fact, mixture Z_30 exhibited the lowest absorption capacity among all the samples with internal curing aggregates, even though the mechanical tests have already proven that Z_30 did have the most robust matrix due to a larger amount of internal curing water carried by 30 wt% of zeolite. The reason may be the existence of the balance between the incorporation of porous aggregate and internal curing effect. When the internal curing effect is under a certain threshold, the incorporation of porous curing aggregate will increase bulk absorption capacity. In contrast, when the amount of internal curing water beyond that threshold, the increment in compactness of the matrix due to the internal curing effect starts to block the external water from absorbing by the bulk sample, so-called water blocking effect, which leads to a lower absorption capacity. This phenomenon was also observed in X. Liu et al.'s study [135].

Based on the result of the internal curing aggregates desorption test, as shown in Fig. 15, zeolite has a higher water retention capacity, compared with LWA. LWA tends to release all its absorbed water in the ambient environment while zeolite can store part of it. Also, the SEM images show that zeolite has finer pores compared with LWA (Fig. 16), leading to a slower water release rate. These features of zeolite are favorable for the improvement in self-healing since it is vitally essential for the internal curing aggregate to has the ability to continuously supply water when the external moisture is limited. It can be noticed that, although Z_30 had lower the bulk sample absorption capacity than the mixtures with LWA (Fig. 14), it still led to the comparable self-healing performance compared with LWA (Fig. 13 And Table 4) owning to the its high water retention capacity and slower water release rate (Fig. 15).

4.3 Evaluation of the SAP robustness and self-sealing effect

4.3.1 Robustness evaluation

The result of the robustness evaluation is shown in Fig. 17~Fig. 19. For the filtration test, as can be seen in Fig. 17(a) and Fig. 18(a), the absorption capacity of the SAP decreased from

287.5g/g to 102.6g/g as the dosage of the superplasticizer increased from 0 to 2 wt%. On the other hand, when 1 wt% of the accelerator was used, a much more aggressive inhibition can be observed, where the absorption capacity of the SAP was decreased down to 8.4g/g. Therefore, it can be concluded that the presence of the superplasticizer or hydration accelerator can effectively inhibit the swelling of the SAP particles. The result was further verified by the void size analysis, as shown in Fig. 17(b) and Fig. 18(b). During the mixing process, the dry SAP particles swelled by absorbing part of the mixing water. Afterward, the absorbed water was released for cement hydration as the relative humidity decrease, which caused the shrinkage of the SAP particles and generated empty voids inside the cementitious matrix. Therefore, the average void size can be used to analyze the absorption behavior of the SAP in fresh concrete. The incorporation of the superplasticizer or accelerator led to a smaller average void size, which indicates that the swelling of the SAP was inhibited by the additives that incorporated. On the contrary, as shown in the Fig. 19, the addition of the fly ash led to an increase in the average void size. This could be due to the low solubility of the fly ash compared with cement. Since the presence of cations such as Ca^{2+} and Al^{3+} was reported to have a inhibiting effect on the swelling of the SAP [111, 112], the low solubility of the fly ash reduced the cation concentration of the pore solution, which promote the absorption capacity of the SAP.

It is worth noted that even though the result of the filtration test shows that the superplasticizer and accelerator can aggressively reduce the absorption capacity of the SAP, only minor influence was observed from the void size analysis. Especially for the hydration accelerator, only 3.2% reduction was observed when the dosage of the accelerator was 1.79wt%. And the average void size was reduced for 10.0% when 2wt% of the superplasticizer was used. Similarly, when the fly ash to cement ratio was 1.2, only 8.1% increment on the average size was found.

Therefore, it can be concluded that the SAP is relatively robust under various environmental condition. However, during the mixture design with SAP, the use of superplasticizer, hydration accelerator and fly ash should be considered as a factor that may influence the efficiency of the internal curing.

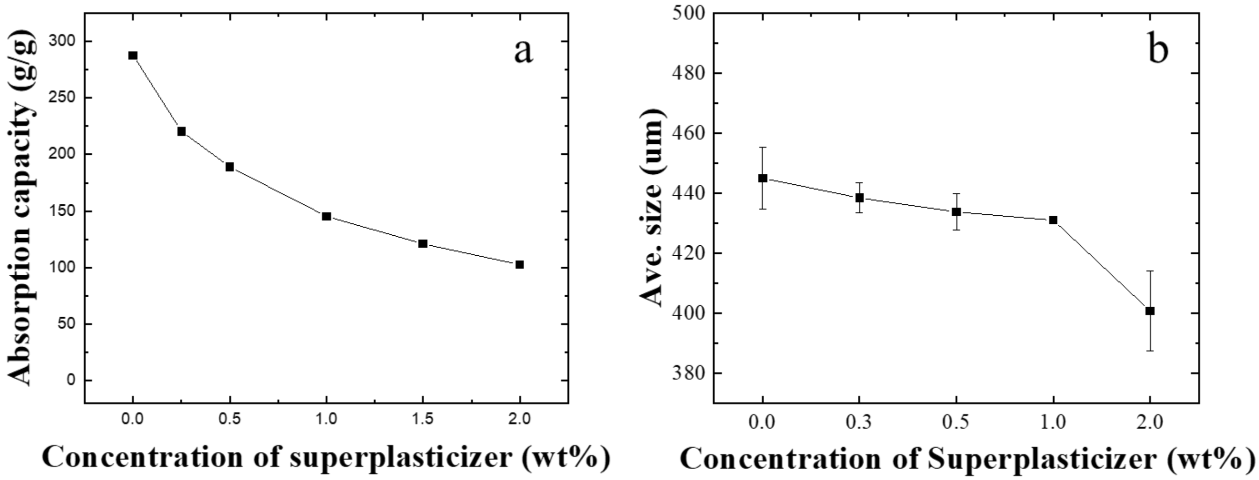

Fig. 17 Evaluation of the SAP robustness under various superplasticizer dosage
a) Filtration test; b) void size analysis

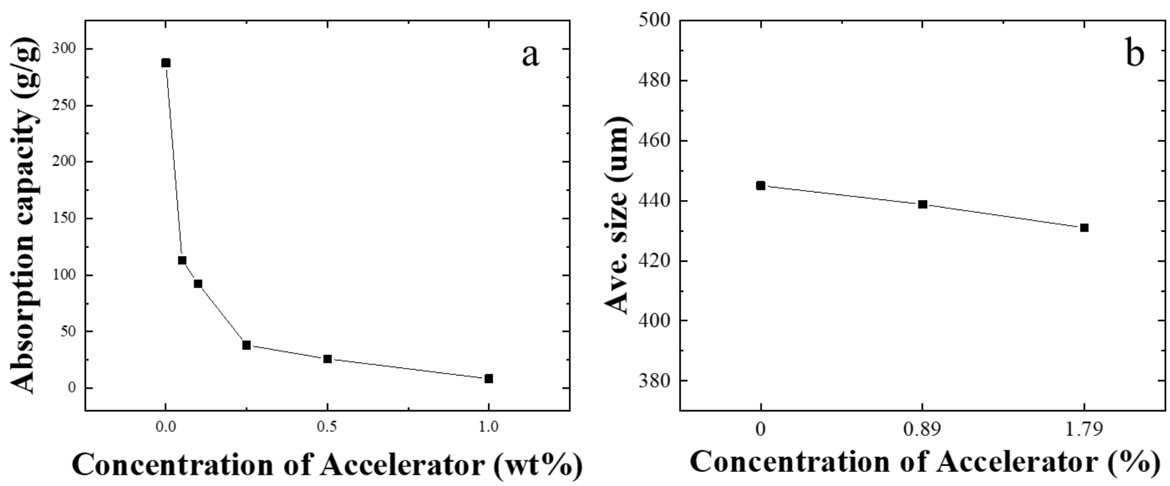

Fig. 18 Evaluation of the SAP robustness under various hydration accelerator dosage

a) Filtration test; b) void size analysis

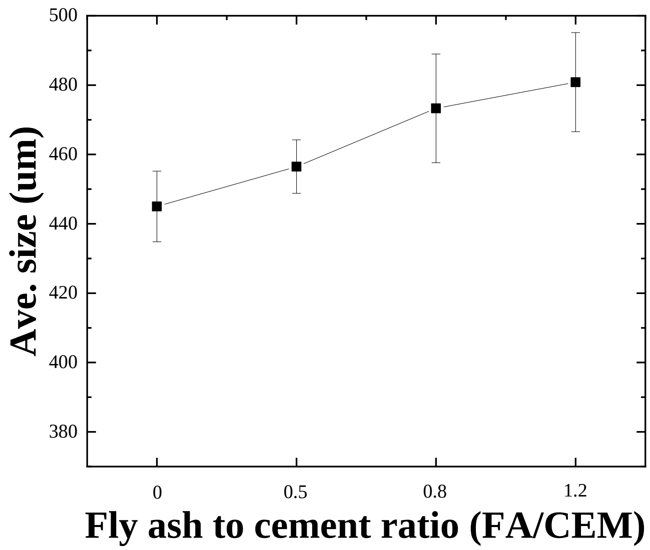

Fig. 19 Evaluation of the SAP robustness under various fly ash content

4.3.2 Self-sealing evaluation

If the cracked concrete exposed to the outdoor environment such as precipitation or groundwater, SAP inside the concrete would form a soft gel that fills in the formed cracks, preventing the further water penetration, which is called self-sealing. The self-sealing occurs rather rapidly (usually within a few minutes in the presence of external water). Afterward, the water retained in SAP triggers the hydration and carbonation at the surface of cracks, leading to a long-term self-healing. Thus, as a precursor of self-healing, the self-sealing ability of SAP will be studied here.

According to the data recorded by the computer, the water flow rate can be calculated by the weight of water passing through the sample per unit time, as shown in Fig. 20. The cumulative passing water with time was also acquired, as shown in Fig. 21.

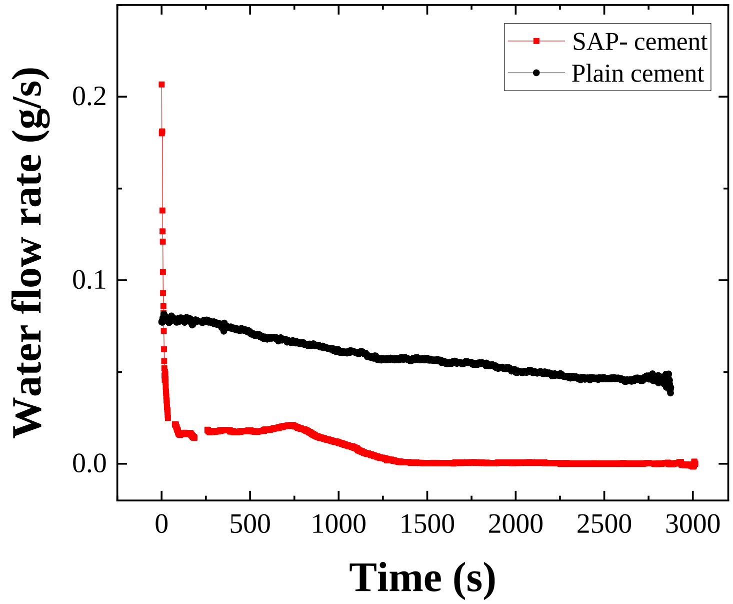

Fig. 20 Water flow rate of the testing samples

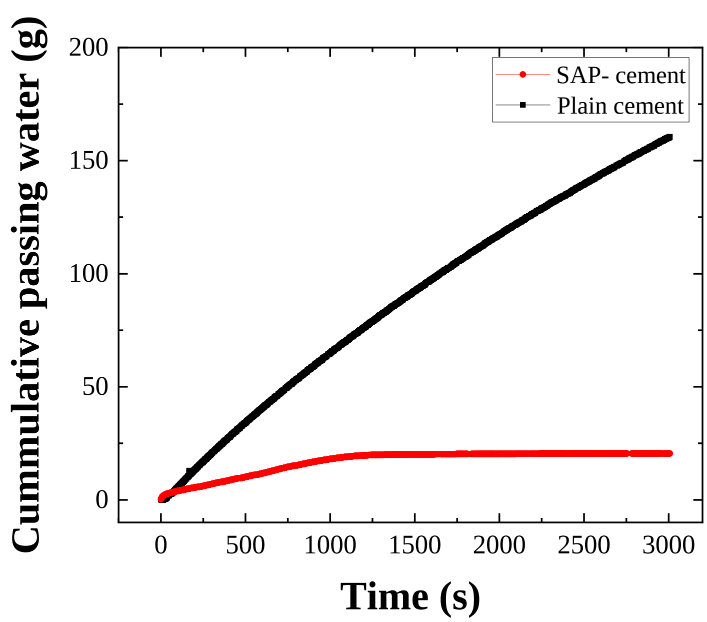

Fig. 21 Weight of the cumulative passing water

It can be observed from the result that the incorporation of SAP effectively controlled the seeping of the water, compared with the plain cement sample. In Fig. 20, it can be noticed that compared with the reference mixture, the sample with SAP has a higher water flow rate at the beginning. It could be due to the large pores left by drying SAPs that favor the passing of the water. However, it can be noticed that within 60 seconds, the water flow rate decreased from over 0.2g/s to 0.02g/s, which is already lower than the plain cement sample. In this process, there are two mechanisms for the reduction of the water flow rate. Firstly, once the SAPs embedded inside the cement paste contact with water, it will start to absorb water and prevent part of the water from passing through the sample. Secondly, after the SAP absorbs water, the swelling effect of the SAP can seal the crack and block the further seeping of the water. In Fig. 21, it can be observed that the passing of water was effectively controlled for the sample with SAP. On the contrary, the water passing through the sample without SAP increased almost linearly with time. The total weight of the passing water was over 150g after 3000s, which is 7 times higher than the sample with SAP. Therefore, it can be concluded that the self-sealing effect of the SAP is capable of controlling the seepage of the external water.

5. CONCLUSION

This thesis aimed to design, evaluate and optimize a cementitious composite with self-healing ability. To control the crack propagation and enhance the tensile performance of the designed material, three types of fibers were separately incorporated into the cementitious composite. The experimental result showed that the sample with PVA fiber exhibited higher mechanical strength and stronger crack width control ability. To further enhance the autogenous healing efficiency of the designed material, this study proposes a hypothesis that the internal curing agent can be used to supply water for the autogenous healing of the concrete. In order to verify this hypothesis, two types of internal curing aggregates, zeolite and lightweight aggregate, were experimentally investigated in this study. The result showed an improvement in compressive and flexural performance when internal curing aggregates were used. It was probably due to the extra water provided by the pre-wetted curing aggregates for the hydration of cementitious materials that enhanced the cement matrix. Among all the studied mixtures, Z_30 exhibited 18.9% and 24.6% improvement in compressive and flexural strength, compared with reference samples. However, there was no significant change in tensile strength due to the larger particle sizes of internal curing aggregates acted as flaws under uniaxial tensile loading. Based on the result of the resonant frequency measurement and microscopic observation, the incorporation of internal curing aggregates effectively improved the autogenous healing of designed cementitious composite. The self-healing recovery ratio increased from 12.6% to over 18 % while the crack width reduction ratio increased for all crack width ranges. The porous curing aggregates increased the absorption capacity of the specimen. They can absorb and store part of the water during wet curing, and continuously release water under the dry curing condition. Since water facilitates the healing of concrete, a better healing performance was obtained. The result of this study verified the hypothesis that by using internal curing aggregate, the autogenous healing performance of the designed material can be improved.

To further understand the mechanism of improvement in self-healing, the water absorption capacity of the bulk sample and the pore structure of the internal curing aggregates were evaluated. Base on the result of the tests, the water-blocking effect could be observed when the replacement level of zeolite reached 30%, which led to the decrement of the bulk sample water absorption capacity. The water-blocking may be due to the dense matrix with lower porosity produced by the strong internal curing effect, preventing the external water from absorbing by the sample during the wet curing. Consequently, a limited amount of water was available for the autogenous healing, which resulted in a decrement of the healing efficiency. The pore structure of the internal curing aggregate was evaluated by the SEM image and the water desorption test. According to the microstructure characterization, zeolite has a finer pore size compared with LWA. And the desorption test indicated that zeolite is capable of storing part of its absorbed water under the ambient environment and released it under extremely low humidity. Therefore, during the healing period, zeolite is able to slowly but continuously supply water to cementitious materials when the external water supply is unavailable. As a consequence, a better autogenous self-healing performance can be expected.

The evaluation of the SAP indicated that the absorption behavior of the SAP can be influenced by the superplasticizer, hydration accelerator and fly ash. However, only a minor influence of less than 10% was observed. Therefore, it can be concluded that SAP is relatively robust under the designed environmental conditions. For the self-sealing effect of the SAP, a dramatic reduction in the water flow rate from 0.2g/s to 0.02g/s was observed within 60 seconds of the test when the SAP is incorporated. In addition, the 5 hours cumulative passing water of the reference sample was 7 times higher than that with the incorporation of SAP. The experimental results indicated the effectiveness of using SAP as a self-sealing agent in concrete materials.

The overall results obtained through this study suggest the feasibility of using of internal curing method to improve the healing performance of the fiber-reinforced cementitious composite. The internal curing aggregates can act as water reservoirs to improve the autogenous healing efficiency, and the robust SAP can act as self-sealing agent to regain the water tightness of the cracked sample.

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