Techniques and various efficiency evaluation tests for self-healing cement-based matrix: State-of-the-art

Mavjot Kaur¹, Krishna Murari² and Inderpreet Kaur³

¹ Under Graduate Student, Guru Nanak Dev Engineering College, Ludhiana, India
² Post Graduate Student, Guru Nanak Dev Engineering College, Ludhiana, India
³ Assistant Professor, Guru Nanak Dev Engineering College, Ludhiana, India

E-mail: mavjotkaur@gmail.com

Abstract. As the aging leads to dilapidation of reinforced concrete structures due to formation of cracks through which chlorides and other chemical agents promote corrosion. There is need of repair or maintenance of such existing structures. Therefore, self-healing, a leading-edge method for durability enhancement, has gained a lot of importance. In this paper, the various self-healing techniques for concrete, including intrinsic healing, capsule-based healing, vascular healing and also immobilization along with healing mechanism of some materials are reviewed with their comparisons. Moreover, a comprehensive study is also done on the different tests for assessment of self-healing in concrete at macro-scale, micro-scale and nano-scale to determine and characterize healing product and to evaluate their mechanical properties.

1. Introduction

In most of the infrastructures, concrete is used at large-scale as construction material having advantage of high characteristic compressive strength, durability and cost economy. However, under the action of tensile forces it is susceptible to crack formation at micro and macro levels. Initially micro-cracks are formed, which are unnoticeable, further leads to formation macro-cracks due to environmental and mechanical actions [1]. These cracks create a pathway for the oxygen, moisture and deleterious materials which causes steel corrosion adversely affecting the durability [2]. For this purpose, the inspection and maintenance techniques have gained increasing attention. Regular inspection and maintenance are difficult rather impossible for inaccessible large scale infrastructures owing to capital investment required. Various other factors include labor and the location of the damage.

Grouting and coating materials are the temporary solutions for external cracks and has unpropitious impact on durability as 20% of the repairs fail after 5 years and 55% after 10 years [3–5]. The repair of cracks by self-healing in concrete has had great attraction as self-healing allows repairs in inaccessible regions without human intervention.

In the present paper, various techniques and evaluation tests of self-healing for cement based matrix are demonstrated.

2. Various techniques of self-healing cement based matrix

Self-healing to repair cracks in cement based matrix can be done by various techniques. Classification of self-healing for cement based matrix is given in figure 1.
2.1 Intrinsic healing

Intrinsic healing is the capability to plug micro cracks by the concrete itself [2]. It is a crack healing phenomenon, which is effective for small crack width, involving chemical reactions in the presence of water. Reduction in crack size is also studied under intrinsic healing.

Summary of the early research works: the first investigation on water retaining structures, and pipelines by the French Academy of Science was done in 1836; formation of white crystalline precipitate in the cracks due to carbonization [6]. In 1926, Glanville executed an analysis of healing phenomenon [7] and followed by Soroker and Denson in 1926 and Brandeis in 1937 [8-9]. Different aspects of intrinsic healing have been studied by Jacobson and Sellevold [10], Clear [11] and Edvardsen [12]. Cracks inside the matrix can be naturally sealed by four mechanisms including [13]: Formation of $CaO_3$ from $Ca(OH)_2$, blockage of cracks by fine particles, additional hydration of the unhydrated cementitious constituents, enlargement of the hydrated cementitious mix.

Major components of Portland concrete are C2S, C3S, C3A and C4AF. After addition of water into the cement, C2S and C3S hydrates further produce CSH gel and CH and soon after mixing, the cement particles are covered with entringite layer leaving unreacted cement. On another note, Calcium silicate reacts with Carbon dioxide to produce calcium carbonate and silica gel, blocking cracks [14]. According to Neville [15], self-healing of early concrete is caused by hydration of the unhydrated cementitious materials. Divergently, at later stage it came to conclusion that calcium carbonate paves the way for crack filling [16]. Therefore, the involvement of water is significantly related to hydration of the unhydrated components in the cementitious matrix. Recently, it was discovered that CSH gel accumulates on the crack surface whereas crystallization of portlandite occurs in the middle [17].

Li et al. used Engineered Cementitious Composite (ECC) with polyethylene fibers as reinforcement for limiting the width of crack [18]. In another study, crack width of 60μm was obtained by using Polyvinyl Alcohol (PVA) [19]. Several research studies started on the possibility of mixing Super Absorbent Polymers (SAP) in concrete mixture. SAPs are cross linked polymers which soak liquid from environment and swell. According to Minon et al. [20], SAPs are pH dependent. Kim et al. [21] used ECC in mortar mixture with PVA 2% and SAP content of 0.5% and 1% to cement weight. To develop cracks, Flexural test was conducted on pre-cracked specimens that were cured for 28days. The crack width of 15.7μm and 17.3μm were obtained for SAPs content and for the control mix 22.3μm. Azarsa et al. [22] utilized crystalline admixtures (CA) by 2% of the cement content. After 28 days of air curing, standard crack-inducing jig was utilized to induce crack. The sample was sealed and exposed to constant water head on one side. The discharge through the specimen is measured. The sample with CA obtained healing ratio of 0.996 and 0.991 in control mix.

2.2 Capsule based healing

Capsule, usually micro-sized particle, is a shell which contains healing agent in solid, liquid or gas state [5]. These capsules provide mechanical resistance and releases the healing agent only when triggered by cracks, moisture and pH changes [23]. When cracks propagate in the concrete, the content present in the dispersed capsule is pulled out by capillary action (as shown in Figure 2) [5,23]. Low viscosity is the fundamental requirement of the healing agent for efficient release[4, 24]. Diameter of
capsule, shell thickness and texture of surface are certain factors considered for encapsulation healing performance [25].

Figure 2. Capsule based Self-healing. (a). Before development of cracks (b). After development of cracks.

Figure 3. (a) Two Component micro capsules. (b) One component micro capsules.

In 1994, Dry, initiated a built-in capsule in concrete[24], utilized hollow porous polypropylene fibre capsule with methyl-methacrylate (healing agent), the improvement in flexural strength was reported [25]. Karaiskos and co-workers [26] embedded hollowed polyurethane-tabulated capsules in a grid and connected to steel bars. The preparation of capsules containing dyes was marked as the beginning of development of microencapsulation as they were included into papers replacing carbon paper[27].In 2001,White et al., introduced microcapsules utilization for polymers self-healing; used urea-formaldehydes (shell material) and dicyclopentadiene as healing agent [28]. Du et al. [3] fabricated toluene-di-isocyanate(healing material) in paraffin shell using met condensation method. In the work conducted by Wang et al.[29], melamine based microcapsules having inactive substance to protect the bacterial spores (encapsulated by poly-condensation reaction method) of size 5 μm was used. Milla et al [30] developed microcapsules with urea-formaldehyde as shell material, using water-in-oil suspension polymerization technique, carrying calcium nitrate tetrahydrate healing agent and Sorbit amono stearate surfactant was added to avoid acidification of the mixing water. Agitation rate of 800rpm and 1500rpm were utilized as it controls the average diameter of the microcapsule. The final microcapsule diameter was between 22 and 50μm.W. Du et al. prepared paraffin microcapsule containing toluene-di-isocyanate(TDI) as recovery agent and investigated microcapsule size distribution, under optimum preparation factors (one part of paraffin and two parts of TDI by mass with agitation rate 600rpm at temperature 75°C), was 30-300μm and one-tenth of their diameter was their thickness [3].

On the basis of healing agent reaction mechanism, microcapsules are classified into two categories, namely one-component and two-components (see figure 3). In the case of one-component, the healing material, which is encapsulated, is released, further reacts with air, water or curing agent scattered in the concrete mix which results in cracks filling whereas, in two-component, both the materials for healing and curing are encapsulated in separate microcapsules; after the rupture due to cracks, healing agent and curing agent reacts with each other to fill the cracks. To exemplify, Cailleux and Pollet [31] encapsulated bisphenol-F epoxy resin in gelatine based microcapsules of spherical shape and a hardener was scattered inside for the reaction to trigger. Yang et al. utilized microcapsules made of silica gel which encapsulates methyl methacrylate and triethylborane to synthesize two-component microcapsules [1,32].

2.3 Vascular healing
Vascular system is another approach to transfer healing agents into the concrete; as the name suggest, it is like a human body’s vascular network consisting of single or multiple channels(hollow tubes) through which healing agent is applied from exterior of the structure[23,33] (refer figure 4).
In 1993, Dry, first to put forward vascular healing method, installed two fibre tubes and through them methyl methacrylate (healing agent) was drawn into the concrete [33]. Other structural elements such as concrete frames were also tested by Dry et al. by embedding glass tubes to heal cracks caused by dynamic loading [34-35]. Mihashi et al. incorporated two glass tubes, attached to external reservoirs. One component of epoxy glue was stored in one reservoir and second component of epoxy glue in the other one. During crack occurrence, both the tubes broke leading to polymerization reaction. However, the regain of strength was almost same as the components did not mix effectively [36]. Joseph et al. made use of cyanoacrylate for healing by providing borosilicate glass tube as carrier in concrete [25, 37]. Sangadji and Schlangen [38–40] developed vascular network, made of porous concrete. Porous concrete cylinders were casted which were surrounded by PVA film that dissolves after casting of dense concrete around the porous matrix and creates possibility of exchange between interior and exterior layer. Installation of micro controllers is done which switches on a pump, when a crack appears, to inject healing agent into porous concrete to densify it which further promotes crack healing.

2.4. Immobilization

Immobilization involves embedment of organic compounds into inorganic compounds into inorganic matrices. for immobilization application, disparate methods, as shown in figure 5. , have been adopted; [41]. In addition to high spectral transparency and controlled porosity, immobilization also offers stability mechanically and thermally [42]. Polyurethane (PU) has biochemically inert characteristics and is strong mechanically, making it a popular medium for immobilization [43]. B. Spaericus was immobilized by utilizing PU by some authors [44]. Bhasker et al. immobilized Sposarcina ureae and Sporosarcina pasteurii using zeolite [45]. Researchers also immobilized B. Cohnii in various carrier; Sierra-Beltran et al. used Light Weight Aggregate and Zhang et al. utilized expanded clay and perlite [46-47].

3. Materials

3.1. Bacteria

The approach, biological repair technique of bacterial introduction in the concrete, dates back in 1990s in which environmentally friendly processes were suggested for crack repair [48]. Concrete is highly alkaline, the genus bacillus group of bacteria is capable of resisting to such alkaline conditions [49], [50]. A source of nutrient is provided to the bacteria which undergo metabolic process and precipitate of calcium carbonate is obtained [51-52]. In the precipitation pathway, urea -is converted into $NH_4^+$ and $HCO_3^-$ [52].
Figure 5. Different methods of Immobilization.

In the starting of microbial urease mechanism, one mole of urea is hydrolyzed intracellularly to a mole of NH$_3$ and carbamate each. Further spontaneous hydrolysis of carbamate occurs leading to formation of NH$_3$ mole and H$_2$CO$_3$ mole (Eqs. (1)-(2)) [49].

$$CO(NH_2)_2 + H_2O \rightarrow NH_3COOH + NH_3$$  \hspace{1cm} (1)

$$NH_2COOH + H_2O \rightarrow NH_3 + H_2CO_3$$  \hspace{1cm} (2)

Following that, these products further produce unimolecular bicarbonate and bimolecular ammonium and hydroxide ions each (Eqs. (3)-(4)). The rise in pH is observed which shifts bicarbonate equilibrium, and carbonate ion is formed (Eqs. (5)) [53].

$$H_2CO_3 \rightarrow HCO_3^- + H^+$$  \hspace{1cm} (3)

$$2NH_3 + 2H_2O \leftrightarrow 2NH_4^+ + 2OH^-$$  \hspace{1cm} (4)

$$HCO_3^- + H^+ + 2NH_4^+ + 2OH^- \leftrightarrow CO_3^{2-} + 2NH_4^+ + 2H_2O$$  \hspace{1cm} (5)

The formation of calcium carbonate precipitates due to availability of calcium ions after reaching a certain supersaturation level (Eq. (6)).

$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3$$  \hspace{1cm} (6)

Negative charge of the cell wall leads to attraction of calcium ions which results in crystal formation on bacterial cell [54].

Another pathway for production of CaCO$_3$ is aerobic oxidation of organic acids, which shows less environmental effect in comparison to ureolytic pathway and through this metabolic pathway carbon dioxide is produced that leads to autogenous self-healing [55–58].

3.2 Admixtures

Self-healing can be imputed by reaction of admixtures in the cementitious matrix. Addition of these minerals is done during mixing of concrete mixture. After crack formation, these minerals react when water penetrates through them which leads to formation of products that imparts in crack healing [59]. They are classified into two categories: expansive additives and crystalline additives [60]. The creation of cementitious products in cracks such as Al$_2$O$_3$–Fe$_2$O$_3$-tri, Al$_2$O$_3$–Fe$_2$O$_3$-mono and Calcium carbonate and Ca(OH)$_2$ crystals in air voids were noticed [61]. Based on the assumption that re-crystallization takes place when these leached out products comes in contact with water that is flowing through fractures, Kishi and co-workers formed a self-healing technique. Effects of several agents which includes geo-materials and chemical as well as expansive admixtures together with some of their combinations were evaluated and was concluded the proper dosage of CO$_3$-2and expansive admixtures, can enhanced self recovery of cracks[61–64]. Jaroenratanapirom et al. also examined self-healing with different additives of varying
proportions[65-66]. Researchers have utilized crystalline admixture which contains reactive silica and crystalline catalyst [22,60,67-68].

3.3 Nanomaterials
It is an irrefutable fact that nano particles are superior fillers than micro-sized materials. In recent years, nano nanomaterials had been used in concrete for various objectives, such as, to improve strength and durability, self-cleaning, self-healing and corrosion protection [69]. Nanomaterials can be converted from all materials by physical or chemical processes [70]. For the manufacture of nanomaterials, there are two ways : top-down method [71] and bottom-up method [72] (figure 6). For concrete, nanomaterials are mostly obtained from bottom-up approach [73].

Hardly any researches have been carried out for self -healing by incorporating nanomaterials in concrete. For construction usage, nanomaterials used, on the basis of accessibility, are nanosilica, nanoalumina, polycarboxylates and nanokaolin[74, 75]. In 2010, Qian et al. studied about nanoclay as water supply to promote hydration alongside the cracks in high performance concretes and showed considerable amount of improvement in recovery level by incorporation of nanoclay [76]. Examination of capability of nanosilica to react with \(Ca(OH)_2\) in concrete matrix and production of Calcium silicate hydrate gel had been done [77–79].

The Pros and Cons of different self-healing techniques are discussed in Table 1.

4. Tests to assess and characterize Self-healing efficiency
The concrete self-healing evaluation has become an attraction because of the wide range of assessment methods available. These methods are utilized to form the criteria for quality of concrete which are of macro, micro or nano levels [91]. The macro level tests consist of mainly monitoring of concrete mechanical properties and durability tests while the micro level tests consist of optical and visual analysis tests. However, at nano level, there are few researchers on mechanical properties. Table 2 shows the various tests conducted. It is evident from the literature that all the research work is evaluated by conducting macro level tests.

4.1 Macro-level tests
The paramount consideration for healing efficiency in the concrete is the varieties of materials utilized for healing and their ability to regain the mechanical properties in varying circumstances [92]. The test included for mechanical properties recovery are flexural, compression, splitting, tensile and non-destructive tests. Bending tests that are mainly conducted for the evaluation of healing capacity are three-point bending and four-point bending. In three-point bending, a notch is fabricated in the center of the deflection section while in four-point bending, fabrication of the notch in not done for a practical approach [93]. This test has been used for cementitious material containing healing agent, such as bacteria [94], light-weight aggregate [95] and expansive agents [64]. Alghamri et al. utilized light-weight aggregate with sodium silicate (healing material) and showed 80% recovery in pre-cracked strength which is quintuple of the control specimen’s recovery [95]. As defined by ASTM C39-03 [96], compression test is “applying a compressive axial load to mold cylinders or cores at a rate which is within a prescribed range until failure occurs”. Gupta et al. [94] explored biochar, obtained from wood scrap, for carrying bacteria spores in cement matrix which precipitates carbonate to regain strength and permeability. The results showed an increase in strength by 38% and 65% decrease in permeability. Ultrasonic pulse velocity, a non-destructive test, has been conducted on several healing agents on hardened concrete [64,95,97].

To increase durability, diminution of porosity is the pre-dominant aspect which eventually affects the performance of concrete in long run [52]. Water and gas permeability are the measure often used. And moisture and gas affects the durability and their transportation into the micro-cracks enlarge the cracks [98]. Kanellopoulos et al. [84] performed gas permeability test used methanol as medium on cementitious matrices containing sodium silica, colloidal silica and tetraethyl orthosilicate which were
injected manually in the cracks. The results obtained were 36% and 43% reduction in gas permeability in sodium silica and colloidal silica respectively. Various other tests are mentioned in the Table 2.

Table 1. Pros and Cons of different self-healing techniques

| TECHNIQUE       | BENEFITS                                                                 | DRAWBACKS                                                                                     | REFERENCES |
|-----------------|---------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------|------------|
| Vascular system | Healing agent discharge when required. Adjustable high quantity of healing material. | Casting difficulties. Strength parameters of cement matrix affected in presence of too many tubes. | [29]       |
| Encapsulation   | Healing agent discharge when required Medium quantity of healing agent     | Casting difficulties. Strength parameters of cement matrix affected in presence of too many hollow fibers. Difficulty in release of healing agent may be possible. | [80-84]    |
| Microencapsulation | Healing agent discharged when required response to multiple cracked areas at same time | Complexity in preparation of capsules and casting Restricted amount of healing agent.            | [63,85]    |
| Nanomaterials   | High recovery in mechanical characteristics Outstanding healing efficiency Cost-effective and energy saving Applied internally and externally | High safety requirements.                                                                 | [86-87]    |
| Bacteria        | Natural and pollution less and biologically effective way                 | Bacteria protection measures should be taken Need to meet several prerequisites.               | [29,49,88] |
| Admixtures      | Excellent efficiency in healing Healing products are more compatible with cement matrix | Under ill-treatment, undesirable expansion Uncertainty in generation of healing products.       | [89,90]    |

4.2 Micro-level tests
The micro-level tests conducted to make the results more reliable. The purpose of these tests is the visualization of crack healing process in concrete and the morphology, identification and characterization of the accumulated matter post-healing process. The commonly used methods are scanning Electron Microscopy (SEM), X-ray diffraction (XRD) and Energy Dispersive Spectroscopy (EDS).

Some researchers used SEM to discover the mechanism involved in self-healing [99], while others conducted studies to observe to structure, texture and size of the material[100]. For the determination of the composition of healing product, XRD test is conducted. Using this technique, Wang and coworkers discovered that calcite was the major healing compound produced[100]. Similarly, Kalhori and Bagherpour also used XRD to recognize the healing products induced by bacteria, Bacillus subtilis[101]. It is interesting to know that, Wang and coworkers conducted X-ray tomography on
products formed by using hydrogel immobilized bacteria and the results illustrated the minerals precipitated in the whole concrete specimen along with the cracks, thus, resulting in overall upsurge in the compressive strength [102]. Similarly, other tests have also been used to enhance the results as given in the Table 2.

Table 2. Tests for Evaluation of concrete self-healing

| TESTS                | EFFICIENT HEALING MEASUREMENT | AUTHORS                  |
|----------------------|-------------------------------|--------------------------|
| **Macro Level**      |                               |                          |
| Bending test         | Measure strength              | [64,84,95,104-105]       |
| Compression test     | Measure strength              | [44,47,88,94,106-107]    |
| Tensile test         | Measure strength              | [102,108-109]            |
| Splitting test       | Measure strength              | [110-111]                |
| Ultrasonic Pulse Velocity | Measure tightness            | [64,95,97,112]          |
| Water permeability   | Damage, healing efficiency    | [29,113-115]             |
| Gas permeability     | Damage healing efficiency     | [84,119-120]             |
| Corrosion Test       | Corrosion resistance          | [117-118]                |
| **Micro level**      |                               |                          |
| Optical microscopy   | Visualization of healed crack | [84,119-120]            |
| Scanning Electron microscopy | Visualization of crystal deposition | [99-100,114,121]        |
| X-ray diffraction    | Identification of crystal matter | [84, 88,119]        |
| Fourier transform infrared spectroscopy | Identification of crystal matter | [84, 95, 105]        |
| Thermo-gravimetric analysis | identification of crystal matter | [64, 108, 122]        |
| X-ray tomography     | Visualization of healing      | [102, 104, 123]          |
| Neutron tomography   | Visualization of healing      | [124-125]                |
| **Nano level**       |                               |                          |
| Nano-indentation     | Determination of Mechanical properties | [103]                 |

4.3 Nano-level tests
These tests are conducted to further enhance the results by making them more reliable. Recently, Xu and Yao employed Nano indentation test, that is utilized for measurement of mechanical properties of small volumes (healing products), for evaluation of healing efficiency of bacteria-based concrete and discovered that the average Nano-mechanical values were 20% more of the transition zone in comparison to outer precipitates, that binds the deposited layer and matrix [103].

5. Conclusion
It is apparent that the mechanism of self-healing not only involves physical but also chemical process. In this review, several techniques of self-healing developed, including intrinsic, capsule-based, vascular and immobilization, as well as different healing materials have been discussed. Table 1 shows their reasons to support and oppose. Maximum research work has been reported using bacteria as it is proved to be comparatively more efficient in crack healing and the least by Nano-materials which has been recently discovered for self-healing in concrete.
For the examination of efficiency of several self-healing approaches, a series of tests are conducted (Table 2); out of which macro-scale tests were adopted by most of the authors which showed contrasting results for the same healing agents, making them inconclusive. Determination and visualization of the healing product, by using micro-scale tests, increased reliability by measuring the reduction size of crack. Researchers opting for Nano-scale tests were a few. However, none of the authors reported the macro, micro as well as Nano-scale tests in one experiment. Hence, making it obvious that standardization of the methods to analyze self-healing efficiency is required.

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