Review Article

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Dentistry Section

Prospects and Future Scope of Self-healing Composites

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ABSTRACT

A biomaterial is a component that has been developed to interact with biological systems for therapeutic or diagnostic purposes in medicine. Tissue engineering, bioprinting, and regenerative medicine are just a few of the increasingly complex fields where biomaterials are being used. These applications frequently require difficult or even paradoxical combinations of biomaterial qualities that cannot be satisfied by traditional biomaterials. Many novel proposals have been introduced over the past 10 years to make biomaterials self-healing, opening up fresh possibilities for enhancing the functionality of conventional biomaterials. Thus, the self-healing composite that will be discussed in the present article is one such biomaterial that comes into play. Self-healing composites are composites that automatically heal in the area where damage has occurred. This material draws inspiration from the way our body heals itself through regenerative processes, and these self-healing composites utilise various additional healing methods as well. This paper outlines the various aspects of self-healing composites and their types, with a focus on capsule-based and vascular self-healing systems. As a complement to previous reviews, this paper provides insights into the diverse self-repairing concepts proposed so far, as well as compares the study of healing mechanisms and manufacturing approaches for the assembly of capsule and vascular networks. The current concept of self-healing polymers provides advanced avenues for secure, longer-lasting, and more durable products and parts across a wide range of industries, such as veneers, electronics, transport, and energy.

Keywords: Biomaterial, Capsule-based, Healing mechanism, Vascular healing system

INTRODUCTION

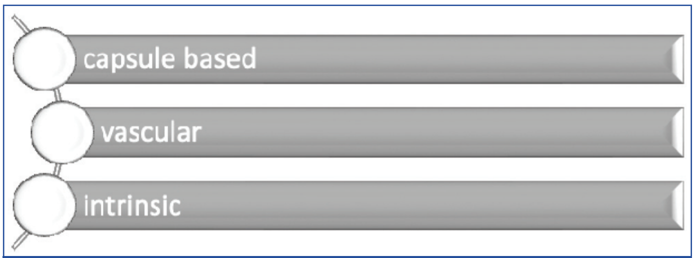
Composite resins are an established class of restorative materials that have advanced with the development of finer particle sizes, superior bonding techniques, improved curing techniques, and sealing techniques [1]. They fail as a result of a long-term deterioration process caused by continuous loading of the structures, resulting in the formation of microcracks. Bridging these cracks is impossible, especially when the structures are in distant regions [2]. Hence, self-healing composites were developed; they have the capability of recovering on their own and flexibly reacting to changing environments. This extraordinary material behaviour is influenced by biological systems that are capable of self-healing [3].

In the human body, any trauma or injury to the tissue triggers processes that allow regeneration in a step-by-step manner, beginning with an inflammatory response and progressing to matrix remodelling through cell proliferation. However, in this material, the injury acts as a triggering factor, setting off a chain of events that transports material to the injured site and causes it to polymerise to fill the crack and achieve strenuous resistance. This material then undergoes chemical repair, depending on the type of healing mechanism [4]. It is believed that self-healing composites will lead to an increase in safety and reliability, bring down the overheads of sustaining synthetic composites, and enhance material vitality. For more than a decade, this field has seen rapid growth and several noteworthy accomplishments [5].

Approaches to Self-Healing

Three different forms of self-healing composites are currently available: i) capsule-based; ii) vascular; and iii) intrinsic self-healing [Table/Fig-1].

In the capsule-based self-healing material, tiny capsules holding a liquid (healing agent) capable of filling and terminating cracks are implemented beneath the material surface. Cracks in the material cause some capsules to burst, leading to the discharge and interaction of the healing agent in the place where the damage has occurred [6].



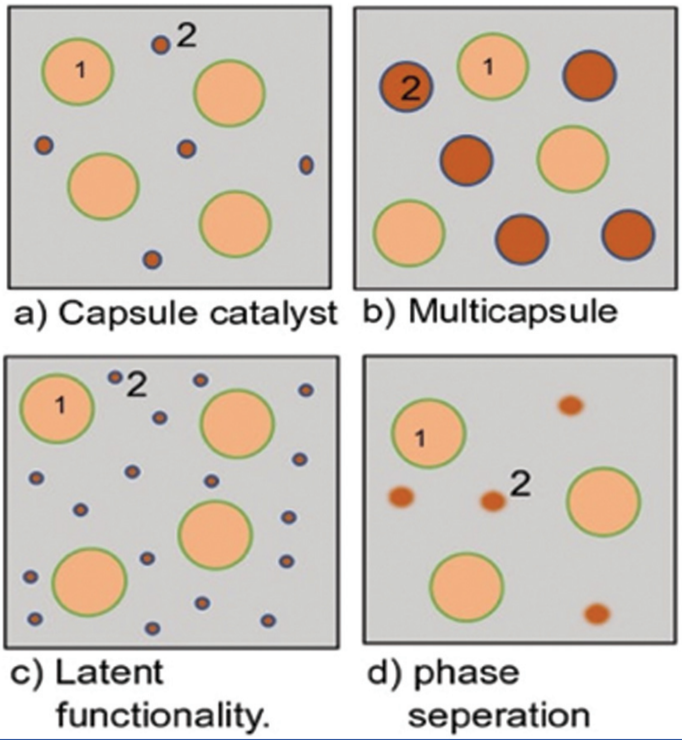
[Table/Fig-1]: Classification of self-healing composites.

Vascular self-healing material conceals the healing agent in a capillary network or vacant channels and utilises these networks to fill the gaps after the crack occurs. In comparison, intrinsic self-healing materials have a latent self-healing capability that is activated by an external stimulus rather than a healing agent. Healing occurs through the intrinsic reversibility of physical or chemical bonding, chain flexibility and entrapment of fibres, polymerisations that can be reversed, and liquefaction of the thermoplastic aspect [7].

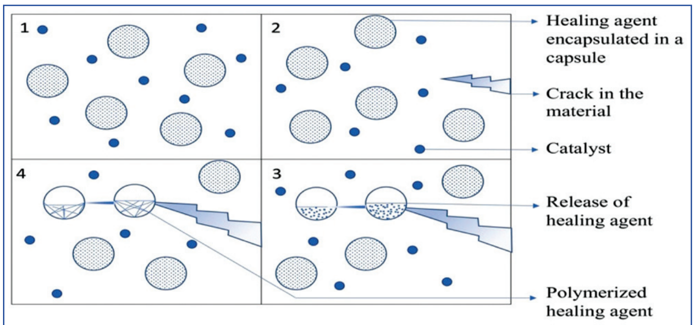
I. Capsule-based self-healing structures: Similar to how humans repair injuries through the regenerative process of cells, encapsulation techniques have been used to create tiny capsules capable of establishing connections when cracks appear [7].

a. Mechanisms of healing: There are four forms to conceal the healing agent shown in [Table/Fig-2].

In the initial basic form, the healing agent, which is a liquid, is enclosed in the capsule while the catalyst is disseminated in the matrix containing polymer [Table/Fig-2a]. When cracks appear as a result of damage in the Poly Urea-formaldehyde (PUF) shield, the healing agent Dicyclopentadiene (DCPD) collects at the area of damage. In the ubiquity of Grubb's catalyst, DCPD undergoes Ring-opening Metathesis Polymerisation (ROMP). This technique converts a combination of cyclic olefins to a polymeric material by expanding and rejoining the stretched rings in monomers to create long chains [Table/Fig-3] [7]. As a consequence, about 75% of toughness can be recovered at room temperature in about 48 hours. Kessler MR et al., placed these capsules containing DCPD into a fibre-reinforced composite to increase the material's life [8,9].



[Table/Fig-2]: Four forms of concealing the healing agent where: 1) healing agent; and 2) catalyst.



[Table/Fig-3]: Showing mechanism of healing of capsule-based healing material enclosed within the capsule containing the healing agent (DCPD) and Grubb's catalyst. Damage in the material leads to the polyurea-formaldehyde shell breaking, causing the release of a healing agent that fills the crack and polymerises by its interaction with the Grubb's catalyst.

In the second form, when the healing agent and polymeriser are both enclosed, the capsules can be increased to incorporate multiple capsules required to concentrate the active elements of the healing agent [Table/Fig-2b]. Keller MW et al., used two different forms of capsules, each filled with one portion of the two-part Sylgard 180 poly-dimethylsiloxane, to demonstrate multiple capsule self-healing in an elastomeric matrix (Poly-dimethylsiloxane-PDMS) [10,11]. In this technique, the repair mechanism includes vinyl-terminated PDMS resin hydrosilation, which is the addition of silicon catalysed by platinum. Combining PDMS resin capsules and DMDNT capsules that act as a catalyst in an epoxy coating leads to the extension of PDMS multi-capsule healing to inhibit corrosion of the underlying substrate [12,13].

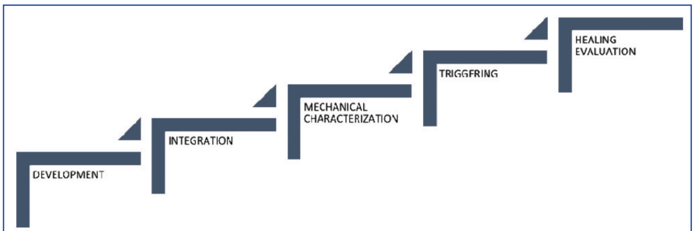
The healing agent is encapsulated or dispersed as particles in the third form (latent functionality), and the polymeriser is either residual reactive functionality in the matrix or an external stimulus [Table/Fig-2c]. In this self-healing method, the healing agent is supplied, and the remaining amine functionality in an epoxy matrix is used to initiate the polymerisation process. By adding thermally polymerisable, meltable epoxy spheres to epoxy composite materials, Zako M and Takano N created a unique system with latent functionality [14].

In phase-separated systems, which are the fourth form, other components may be enclosed, and atleast one healing component or polymeriser is phase-separated inside the matrix [Table/Fig-2d].

A work by Cho SH et al., using phase-separated Hydroxyl End-functionalised Polydimethylsiloxane (HOPDMS) and Polydiethoxysiloxane (PDES) in an epoxy vinyl ester matrix can be used to understand the aforementioned concept [15]. The tin catalysts Dibutyltin Dilaurate/Dimethydineodecanoate Tin (DBTL/DMDNT), initially confined for protection until matrix damage forced their release, interacted with the phase-separated HOPDMS/PDES in these systems.

b. Fabrication process: For the construction of capsule-based self-healing composites, two major steps include healing agent encapsulation and embedding capsules with matrix material. In-situ polymerisation in an oil-in-water emulsion is now the most common and successful technique. This technique involves polymerising shell monomers onto the exterior of core materials [16]. The study by Sottos, Brown, Kessler, and White in 2003 employed PUF as the shell material to be utilised in the encapsulation of DCPD healing agents and provided a very thorough explanation of this in-situ polymerisation process in an oil-in-water emulsion [17]. Other than PUF, Melamine Urea Formaldehyde (MUF), and for two-part epoxy, PUF, Poly Melamine Formaldehyde (PMF), and poly methyl methacrylate were used as shell material by different researchers worldwide. The diameter of the capsules formed when there is in-situ polymerisation in an oil-in-water emulsion depends on agitation and has a Gaussian distribution. Hence, to achieve smaller capsule sizes, numerous methods were adopted, one of which involved the addition of nanoparticles in the shell, known as the physiochemical method, thus reducing the size as well as the roughness of the capsules. Additionally, other techniques used by Zhang H and Yang J devised etched glass bubbles as a healing agent jar as it is fragile and easy to rupture [18]. Moreover, more than one type of material may be used to create the shell. Jin H et al., created a two-layer capsule shell to enhance the thermal stability of the capsule without compromising its splitting activity [19].

c. Capsule-based self-healing material design: The design cycle consists of five steps: development/sequestration, integration, mechanical characterisation, triggering, and healing evaluation [Table/Fig-4].



[Table/Fig-4]: Showing steps in the formation of capsule-based healing material.

i) **Sequestration:** The main goal is to develop a method to sequester the healing agent and polymeriser within the capsule, which is achieved through encapsulation or phase separation. Another factor is the material to sequester ideally; it should be unreactive, low viscosity, and non volatile [20].

ii) **Integration:** After sequestration, the next step is integration, which involves considering the forces acting on the capsules during various mixing and processing procedures. These forces can vary depending on the type of capsule. PU, MF, and UF (polyurethane, melamine-formaldehyde, and urea-formaldehyde) capsules have demonstrated their ability to withstand processing conditions in typical thermoset matrix and composite construction processes [3].

iii) **Mechanical characterisation:** Following the integration process, the material needs to be characterised based on its mechanical properties, triggering mechanism, and healing performance.

The mechanical properties include strength, elastic moduli, and fracture toughness. These properties can be influenced by factors such as the bond strength between the capsule and the matrix, the stiffness of the capsule, and the volume fraction of capsules [1].

iv) **Triggering mechanism:** The triggering mechanism can be verified by observing the capsule splitting and the discharge of the healing material into the fracture site. Techniques such as optical microscopy, Infrared Spectroscopy (IR), Scanning Electron Microscopy (SEM), and Energy-dispersive X-ray Spectroscopy (EDS) can be used to confirm the material discharge at the fracture site [2].

v) **Healing evaluation:** The healing of the composite depends on factors such as the extent of damage, the rate of damage, the healing temperature, and the bond strength between the matrix material and the healed material. The amount of healing can be assessed using techniques such as optical microscopy [5].

II. Vascular self-healing materials: Vascular self-healing materials also contain a healing agent, but in the form of capillaries or channels that can connect and fill the damaged area, leading to healing.

The overall design is similar to capsule-based healing, but the fabrication and incorporation of the matrix material differ. Dry and McMillan developed hollow glass tube containers filled with the healing agent, which solidifies to fill the cracked surface. These hollow fibres are one-dimensional vessels and can be fixed using glass fibre-reinforced composites [20]. In 2007, Toohey KS et al., introduced the concept of three-dimensional vascular self-healing materials using the ROMP reaction as the healing mechanism. The healing efficiency was found to be lower compared to capsule-based self-healing composites [21].

Two strategies were utilised in the organisation of these structures. First, hollow channels of glass fibres were created and filled with sufficient healing agents. The hollow glass fibres are inert to commonly used healing agents such as epoxy resin systems and cyanoacrylates. The second strategy is to create channels in the host material itself to hold the healing agent, as briefly discussed in the following paragraph. The performance of these agents improves with increased connectivity, making refilling easier [22,23].

- **Fabrication process:** Although capsule-based and vascular self-healing composites have similar processes, research in this area has been progressing slowly due to poor fabrication methods. In vascular self-healing composites, the vessel diameters should be small enough to provide sufficient strength, coverage, and connectivity, making fabrication techniques challenging. In fact, the study of vascular self-healing materials has focused more on construction techniques compared to capsule-based self-healing materials, where most attempts have been made to develop innovative mechanisms to improve healing efficiency. Popular fabrication techniques can be grouped and examined in this section [3,24].

One approach involves inserting tubes containing the healing agent, which are made of hollow fibres and keep the healing agent separated from the surrounding matrix [3]. Any damage to the material causes these fires to break, releasing the healing agent, which interacts with the matrix and leads to healing. However, the drawback of this design is that refilling and creating an interconnected network can be challenging [25].

Another approach is the use of scaffolds, which are 3D structures made of materials that are easy to dissolve or remove. These structures are incorporated into the host material after the curing process. Once the host material is cured, the scaffold is removed, leaving behind a hollow structure that can hold the healing agent [5]. This approach can be used to create 1D hollow structures and form 3D structures using advanced methods such as 3D printing of sacrificial scaffolds, melt spinning, electrospinning, etc., [26].

Electrostatic discharge has been considered the fastest method for creating microvascular networks that closely mimic natural designs [3]. In this technique, an electron beam is used to rapidly produce a discharge, similar to lightning, which creates a tree-like structure. The structure created using any of the above methods is then incorporated with the healing agent, which is released in the damaged area. Recently, lasers have also been used to create vascular networks [9].

III. Intrinsic self-healing material: Intrinsic systems operate on reversible reactions and are simpler compared to capsule-based and vascular self-healing materials. The material itself possesses self-healing properties, eliminating the need for incorporating additional hardeners and avoiding integration and compatibility issues. Intrinsic self-healing can be achieved through mechanisms such as hydrogen bonding, ionomeric coupling, meltable thermoplastic phases, or molecular diffusion [27].

There are three major approaches to illustrate intrinsic self-healing materials:

1. **Reversible bonding:** This approach utilises chemical reactions that can participate in self-healing.
2. **Chain re-entanglement:** The mobile part of the crack surface entangles the chains, enabling healing.
3. **Non covalent healing:** This approach relies on hydrogen bonding, which leads to cross-linking in polymers [7].

The design cycle for intrinsic self-healing materials includes:

1. Material development
2. Mechanical characterisation
3. Triggering
4. Healing performance evaluation.

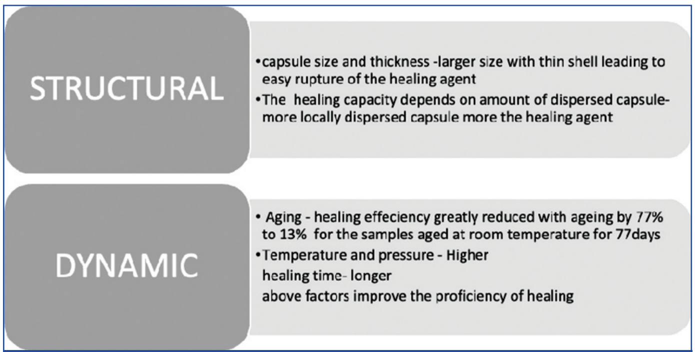
Intrinsic self-healing materials can be understood based on the various reactions that take place for self-healing and can be classified as follows:

1. **Intrinsic self-healing materials based on reversible reactions:** These materials include components that can reversibly change from the monomeric state to the cross-linked polymeric state with the input of external energy [3]. In this case, the damaged area is subjected to heat or photo illumination to increase mobility and enable repair through bond formation. The Diels-Alder (DA) and retro-Diels-Alder (rDA) reactions are popular reaction schemes for remendable self-healing materials [5].
2. **Self-healing from dispersed thermoplastic polymers:** Self-healing in thermoset materials can be achieved by incorporating a meltable thermoplastic additive. This additive melts to fill the crack and physically interlock with the surrounding matrix material [28].
3. **Ionomeric self-healing materials:** Ionomeric copolymers are materials with an ionic segment that forms clusters, acting as reversible cross-links. These clusters can be activated by environmental factors such as temperature or ultraviolet light. The reversible creation of these clusters allows for multiple local healing processes [6,29].
4. **Supramolecular self-healing materials:** Supramolecular self-healing materials involve engineering polymers to create strong end-group and/or side-group associations through complementary, reversible hydrogen bonds. Cordier P et al., demonstrated the self-healing capability of these elastomeric polymers, and Montarnal D et al., developed a simpler synthesis for the precursor molecules of such systems [30,31]. The components of these rubbery self-healing materials can be brought back into proximity to allow for the reformation of hydrogen bonds after damage.

Healing Performance Analysis

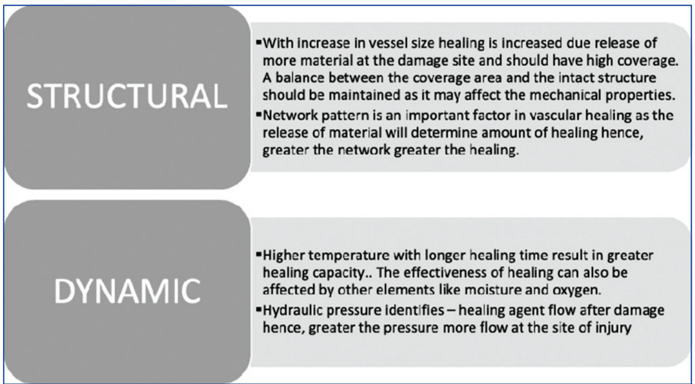
Capsule-based self-healing composite: In a single healing cycle, small and medium fractures can be adequately healed. The Ring-Opening Metathesis Polymerisation (ROMP) with Grubb's catalyst achieves a 75% healing rate within 48 hours, according to the fundamental mechanism [32].

The concentration of the catalyst is an important factor in healing. Total healing is achieved when the catalyst-to-DCPD (dicyclopentadiene) ratio is greater than 10:1, but this significantly increases the cost. An alternative catalyst, WCl6, when combined with DCPD, improves healing [7]. Healing performance is not only dependent on the mechanisms but also on healing time and temperature, which play crucial roles. Factors for healing performance analysis are divided into structural and dynamic factors for both capsule-based and vascular self-healing composites [Table/Fig-5] [3,5].



[Table/Fig-5]: Healing performance of capsule-based self-healing composite containing structural and dynamic factors [3].

Vascular self-healing composite: The healing efficiency of vascular self-healing composites is lower compared to capsule-based composites and is less than 80% under various conditions [Table/Fig-6] [2,3].



[Table/Fig-6]: Healing performance of vascular self-healing composite containing structural and dynamic factors [3].

Intrinsic self-healing composite: Little is known about intrinsic self-healing composites, and more research is required in this area to compare them with capsule-based and vascular self-healing systems.

Despite claims that scientists have significantly improved the healing ability of self-healing composites, performance uncertainties have hindered their real-world applications. Only a few self-healing composites function well under optimal conditions and minimal damage [19]. For self-healing materials to be practically useful, they must achieve "sustainable healing," meaning that healing occurs effectively regardless of the surrounding environment or the extent of damage [33].

Future Prospects

Continued progress in this area will result in the development of new healing chemicals with better durability, higher reactivity, and faster reaction rates [3]. While significant advancements have been made in recent years, there are still many technical challenges that require intensive research efforts to address. There is an urgent need for environmental testing of healing systems [6]. Self-healing designs can also incorporate targeted and localised delivery of healing constituents to improve efficiency while minimising costs and negative impacts on matrix materials. However, there is a scarcity of engineering models that can predict the lifespan performance of self-healing polymers [34].

In addition to restoring mechanical properties, self-healing techniques can also lead to improvements in the properties and performance of other materials. This is particularly advantageous in the field of microelectronics, where current solutions often involve chip replacements [12]. Restoring features like conductivity is especially beneficial, and organometallic polymers with semiconductor-level conductivity and self-healing properties have been developed. Vascular or capsule-based approaches with conductive materials have also shown potential for restoring conductivity [35].

Biological systems provide a roadmap for potential research pathways. Many important biological materials, such as bone, have the ability to regenerate and remodel. In the future, it is possible that self-healing materials could be more controlled and regulated, allowing for regeneration [36].

CONCLUSION(S)

Researchers nowadays are working on enhancing material qualities and restoring material properties after damage. Self-healing composite materials for aircraft and space operations have great potential for reconstructing microcracks and damages that occur during operation in space. However, there are significant limitations in understanding the healing process and its stability. Identifying internal fissures and promoting effective repair are two major challenges in the self-healing process. The research in this field encourages researchers to explore application-based self-healing materials, with a focus on self-healing hydrogels for various structural applications. Aerospace applications often require the use of carbon fibre composites. The progress in studying self-healing composites is reviewed to encourage researchers to explore sustained self-healing at low temperatures in composites. Stress-strain curves are used to study the effectiveness of self-healing thermo-reversible elastomers. Successful repair of cracks in carbon nanotube composite materials has been achieved.

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