Intrinsic Self-Healing Materials: The Future of Sustainable Innovation by Aarav Rishi

Abstract

Inspired by biological systems' ability to repair themselves, self-healing materials, materials that can autonomously repair damage from external stimuli without human intervention, have rapidly become a major focal point for scientists today. These have taken the world by storm due to their wide range of applications in many sectors such as medicine, infrastructure, electronic devices, and energy. A material can undergo self-healing primarily through two ways: extrinsic and intrinsic. Unlike extrinsic healing systems, intrinsic healing materials have the capability to run an infinite amount of times, as they utilize the properties of the material itself through reversible physical and chemical interactions rather than implementing external healing agents. However, a major problem continues to exist in which the mechanical strength of the material is compromised after each self-reparing cycle. In other words, the material becomes less efficient in its repairs each time damage occurs, ultimately making the material impractical. Therefore, it is necessary to look into potential solutions that may remedy this problem. This research paper focuses extensively on vitrimers, hydrogen-bonds, Diels-Alder reactions, and a joint system that utilizes both covalent and non-covalent bonds.

Keywords: Self-healing, intrinsic, dynamic, covalent, vitrimers, Diels-Alder reactions

Introduction

Over the past decade, the advent of self-healing materials has reached the forefront of science. The reason for this is, simply put, that the materials in our modern industry are no longer applicable, due to their energy-intensive extraction, processing, use, and disposal, all of which contributes to the growing problem of climate change and the release of greenhouse gases. Some of these materials include cement, steel, iron, plastics, and more. For example, cement, the primary ingredient used to build concrete, accounts for 8 % of global CO_2 emissions, while steel and iron combine to produce around 7 % of these emissions [1, 2]. While these numbers may seem insignificant alone, when combined, the problem begins to become blatantly obvious. In fact, due to the uptick in industrial processes, researchers have attributed between a quarter to a third of total global CO_2 emissions to industry [3]. Although these materials may not be unusable, their limitations have begun to overshadow some of their more advantageous characteristics. Traditional concrete is prone to developing cracks due to temperature fluctuations and mechanical loads, threatening durability and integrity [4]; steel and other metallic alloys suffer from corrosion, which could lead to chemical leaks; plastics' inability to be biodegradable, leading to large waste heaps and microplastics in the ocean. Suffice to say, self-healing materials are the future of our industrial capabilities, due to their ability to prolong product lifespans, limit human intervention, and reduce maintenance costs.

The scope of self-healing materials has constantly expanded, finding uses in several other industries beyond the realm of construction and infrastructure. Self-healing materials can be

utilized in automobiles and electric devices by acting as coatings that ensure electrical performance over long periods of time, in aerospace by autonomously repairing cracks on the hull of a spacecraft, and even in the biomedical field, in which they can release drugs in a controlled manner as part of the drug delivery system [5].

Materials can experience self-healing in one of two ways: extrinsic and intrinsic. In extrinsic healing, there are several microcapsules embedded in the materials, so that when a crack appears in the material, the capsule releases a healing agent that repairs the crack [6]. Unfortunately, extrinsic self-healing has a finite amount of cycles, as the healing agents must be constantly replenished after damage has occurred. Consequently, focus has shifted to its counterpart: intrinsic self-healing. Materials that intrinsically self-heal use the inherent properties of the material itself and involve reversible bonds, which can be broken and reformed as many times as possible [7].

Current Limitation

Unfortunately, further development in intrinsic self-healing materials cannot occur until researchers can answer a problem that has been plaguing them for the past few years: the trade-off between self-healing properties and the mechanical strength of the material. Mechanical strength can be defined as a material's ability to withstand applied forces without fracturing and includes, but is not limited to, yield strength, tensile strength, compressive strength, and flexural strength [8]. Increasing the mechanical strength of a material typically involves increasing interactions between polymers through the process of crosslinking, a process that forms strong covalent bonds between individual polymers, creating a three-dimensional network. As a result, the overall strength and rigidity of the material has been increased [9].

However, as the strength of material increases, the material loses efficiency of its self-healing properties, modeling an inverse relationship; the material doesn't retain 100 % of its efficiency after each use [7]. Due to increased rigidity and toughness, the polymeric chains no longer retain their molecular mobility that allowed them to initially heal cracks and fractures in the material; the polymers need to be mobile enough to reconnect the reversible bonds that were torn apart. Accordingly, this trade-off may result in a reduced healing rate, in which the material may take longer periods of time to achieve the same level of self-healing efficiency [10]. This is highly impractical, as the longer a crack or a fracture remains untended to, there is a greater chance of catastrophic failure that could result in the collapse of the entire material and potentially an entire structure, depending on the scope and size of the material. It is also not useful in scenarios that require self-healing to be almost instantaneous. For example, in space, if a hole was to appear in the hull of a spacecraft, the material should repair the puncture wound as fast as possible to give the astronauts more time to make a more permanent repair. If this problem of mechanical strength of a material versus the self-healing properties of a material continues to exist, decreased maintenance costs and a lack of human intervention may not be possible anymore.

Solutions

Below are four potential solutions that can solve the trade-off between mechanical strength and self-healing properties. They include vitrimers, Diels-Alder reactions, hydrogen bonds, and dual-network systems.

Vitrimers

One such solution to the persistent problem of the trade-off of durability and reprocessability properties of a material is a vitrimer. Founded recently in 2011 by Ludwik Liebner and his colleagues at CNRS, vitrimers are permanent networks of cross-linked polymeric chains with dynamic covalent bonds, which are just bonds that can break and reform, allowing for adaptiveness to external stimuli [11].

Vitrimers combine the properties of two different types of polymers: thermoplastics and thermosets. Although they are both types of polymers and rely on covalent bonds, their difference lies in the arrangement of those bonds. Thermoplastics are polymers that soften, allowing for enhanced mobility and reprocessability, due to the constant process of heating and cooling the material; thermoplastics are held together by a chain of weak, reversible, intermolecular forces, rather than strong chemical bonds [12]. Thermosets, on the other hand, are materials that undergo a one-time chemical reaction that permanently hardens the material; unlike thermoplastics, thermosets cannot be reshaped due to the irreversible bonds that contribute to the material's new strength and lack the mobility that characterizes thermoplastics. In essence, vitrimers combine the reprocessability of thermoplastics and the mechanical hardness of thermosets into a single polymeric chain [12].

A major advantage of vitrimers is their "shape memory", where they can "remember" a specific shape and regain that shape even after being exposed to external stimuli like heat, stress, and other environmental conditions. Specifically, vitrimers have a specific temperature threshold, known as the topology freezing transition (Tv) [13]. If the surrounding temperature is below the Tv, the covalent bonds act normally by remaining rigid and robust. Contrarily, if the surrounding temperature exceeds the Tv, the covalent bonds become active and undergo the breaking and reforming of bonds, allowing the polymer chains to rearrange their positions in the network [14-15]. Relating to this, the most crucial attribute of vitrimers that sets them apart from other potential self-healing materials is their dynamic covalent networks. Through the process of associative bond exchange, the overall integrity and the amount of covalent bonds remains constant at all times, even when bonds are being broken and reformed. This phenomenon is because a new bond forms simultaneously as an old bond is broken, as per the mechanisms of associative exchange, therefore allowing it to permanently retain the structure of the cross-linked network [14]. This is significant, because other self-healing polymers typically use covalent bonds *in equilibrium*, which simply means that the polymers can withstand only certain constant temperatures. If the temperature was to exceed this threshold, the polymer could lose some of its properties or network degradation would occur.

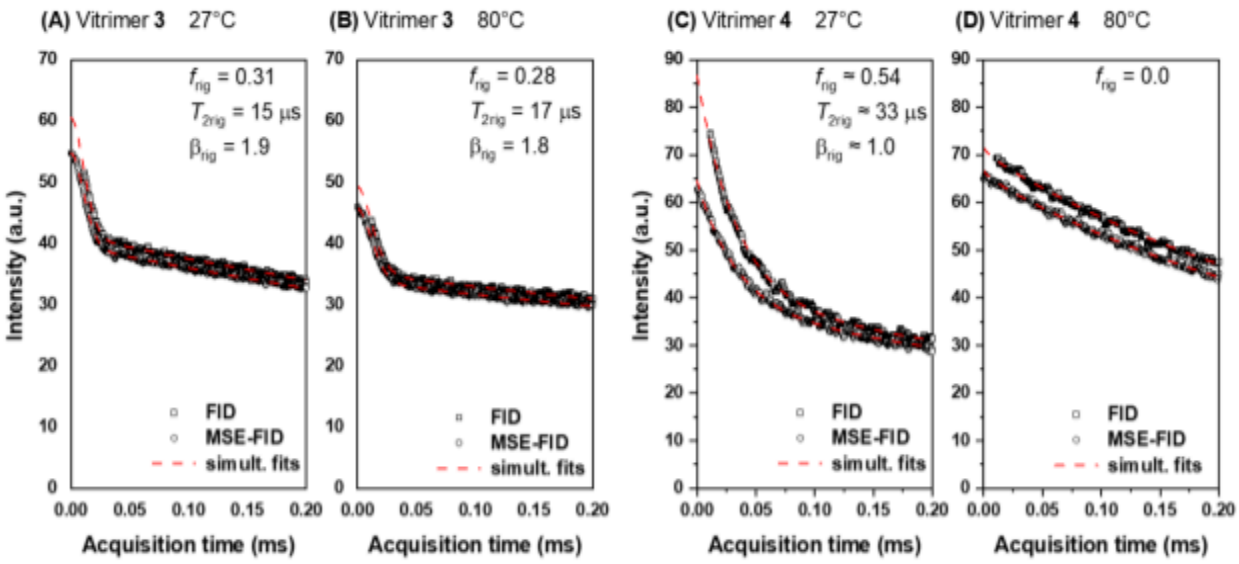

Fig 1: This figure shows contrasting images of two vitrimers. Images on left depict how the covalent bonds remain rigid and robust when temperature is below the topology freezing temperature (Tv). Images on right depict how the bonds become active and are able to break apart, as the temperature exceeds the Tv.

Although still a relatively new concept, vitrimers have already found ways to impact today's society, appearing in several industries, ranging from construction and infrastructure to energy and electronics to adhesives and coatings [16]. As of late, vitrimers have also begun to emerge as viable candidates for adjustable or reshapable prosthetic components in the biomedical field. All in all, vitrimers act as smart materials, due to their abilities to withstand all types of temperatures, allowing them to be reprocessable, while still retaining their mechanical and elastic strength.

Diels-Alder Reactions

Another potential viable approach to the trade-off between mechanical strength and self-healing cycles is the Diels-Alder reaction. Founded by Otto Diels and Kurt Alder in 1928, this reaction is a cycloaddition between a conjugated diene and a dienophile [17]. A cycloaddition is simply a chemical reaction that joins two or more molecules together to form a ring-shaped molecule. A diene is a molecule that has two double bonds between the carbon atoms, and the fact that it is conjugated means that the double bonds are separated by a single bond. A dienophile – meaning "diene-loving" – is a molecule that easily reacts with a diene, similar to a lock-and-key situation, allowing the reaction to be completed [18]. When part of polymeric chains, the reaction can create reversible crosslinks between the chains. One of the most common examples of this reaction in the context of self-healing materials is the combination of furan, acting as the diene, and maleimide, acting as the dienophile [19].

Fig 2: This figure displays the cycloaddition reaction between the diene and the dienophile ("diene-loving"), similar to a lock-and-key scenario. In this case, the butadiene acts as the diene, while ethylene plays the role of dienophile, and together, they form a 6-membered ring in cyclohexene, completing the Diels-Alder Reaction.

One advantage that sets Diels-Alder reactions apart from other self-healing materials that utilize covalent bonds is its thermal reversibility. Similar to vitrimers, temperature plays a key role in the process of the Diels-Alder reactions [20]. When exposed to extremely high temperatures, Diels-Alder materials undergo a process known as the retro-Diels-Alder reaction, and unlike vitrimers, the crosslink is temporarily broken, allowing for increased mobility of the polymeric chains, which can then reform and reconnect with each other. Upon cooling, the Diels-Alder reaction can occur again, strengthening the integrity of the loosely bound crosslinks after the retro-Diels-Alder reaction, ultimately healing the network [21]. This is because Diels-Alder self-healing polymers are designed in such a way, in which the diene and the dienophile are incredibly close to each other, allowing them to maximize the thermal energy that is being provided; this proximity is very useful, because when the retro-Diels-Alder "unzips", the partners can easily find each other when the cooling process begins. Through this way, the material can "repair" itself multiple times, without sacrificing mechanical strength.

A notable recent advancement in Diels-Alder reactions is an experiment done by a team of material scientists at Texas A&M. Led by Dr. Svetlana Sukhishvili and Dr. Edwin Thomas, the team developed a polymer – coined as the Diels-Alder Polymer (DAP) – that absorbs the kinetic energy (heat is a form of kinetic energy) of a high-speed projectile, causing the polymer to stretch and liquefy, using the properties of the Diels-Alder reactions. Due to increased elasticity of the polymer, the projectile only takes a small portion of the polymer, leaving only a tiny hole in its wake. After the projectile passes through the entirety of the polymer, the polymer quickly cools and reestablishes the bonds, returning to its original state [22]. The purpose of this experiment was to develop materials that would prevent spacecrafts from rupturing after collision with a meteoroid.

The applications of Diels-Alder reactions can vary, as they have begun to find uses in many different areas. A specific application of these reactions is in the pharmaceutical industry,

in which they are crucial elements in synthesizing drugs, such as cortisone, reserpine, morphine, and others [23]. Overall, Diels-Alder reactions, due to their unique thermal reversibility capabilities, allowing for crosslinks to break and reform based on temperature, are feasible candidates for self-healing materials that retain both their strength and recyclability.

Hydrogen Bonds

Another answer to the problem of compromising either the self-healing properties or mechanical strength of a material are hydrogen bonds. Unlike some of the other previous solutions, hydrogen bonds fall under the category of supramolecular interactions, which simply means that the bond interactions are non-covalent – they rely on electrostatic attractions rather than the sharing of electrons that characterizes covalent bonds [24]. This non-covalent attraction involves a hydrogen bond, already covalently bound to a highly electronegative atom like nitrogen, oxygen, or fluorine, and another electronegative atom with a lone pair of electrons. Although the strength of a singular hydrogen bond is weaker than that of a covalent bond, when there are many of these bonds, their strength is significantly higher yet they remain highly flexible. This strategy is known as hierarchical hydrogen bonding, in which multiple hydrogen bonds of varying strengths exist [25].

The way in which hydrogen bonds can work as self-healing materials is very unique. When subjected to external stress or damage, the idea of sacrificial bonds comes into play. Sacrificial bonds, which are typically weaker hydrogen bonds, are designed to break first, preventing crack propagation from spreading to the rest of the material [26]. Sacrificial bonds also have the capability to potentially lead to the release of “hidden lengths” of polymer chains, chains that were previously entangled or coiled and only appear *after* the sacrificial bonds have broken. The hidden lengths extend and unfold, continuing to act as absorbing energy from the stress to prevent it from damaging overall structural integrity, acting similar to a shock absorber [27]. Upon healing, the hidden lengths recoil, bringing the hydrogen bonds in close proximity with each other. Due to the reversibility of hydrogen bonds, the material is able to heal itself.

One characteristic that sets hydrogen bonds apart from other intrinsic self-healing materials is their fast healing cycles. This rapid healing response can be attributed to several factors, most notably their directionality, which means that they form in preferred geometries (arrays), typically linearly [28].

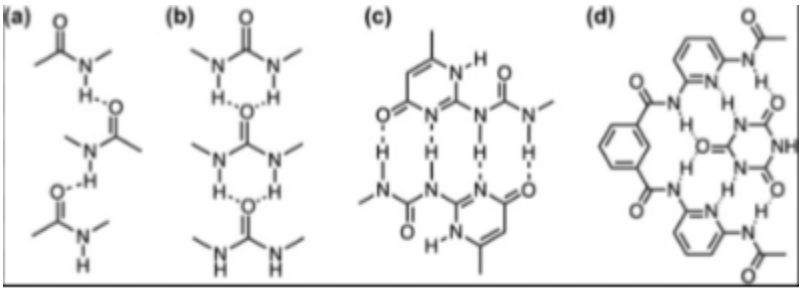

Fig 3: This image illustrates the directionality of hydrogen bonds, which typically occurs in linear geometries. When the bonds are broken apart, healing rate times for this self-healing material are significantly cut down, as the bonds can only be reorganized in a certain structure.

This is important because the more times the hydrogen bonds have to "repair" themselves, the healing process gets faster, as the bonds can only be structured in a certain order; the bonds "know" where each other are, so that they can reassemble quicker, speeding up the healing process. Furthermore, hydrogen bonds in local regions can align themselves with other bonds in those regions first, even before the backbone repair of the polymer has started [29]. This means that local repair at the site of damage can happen immediately, giving more time for the rest of the structure to regain its full mechanical robustness. Additionally, another factor that contributes to hydrogen bonds' rapid healing cycle is their ability to work at room temperature, changes in pH levels, and moisture differences.

Hydrogen bonds have the ability to work in several industries, due to their energy-efficient reprocessability and abundance in the natural world. These bonds are also very biocompatible, allowing them to be integrated seamlessly into biomaterials, minimizing their environmental impact. Recently, hydrogen bonds have become increasingly linked to wearable and stretchable electronics, due to their high flexibility and strength [30]. In short, hydrogen bonds are extremely useful, as a consequence of their rapid healing cycles, allowing them to maintain their flexibility, mechanical strength, and self-healing properties, even after damage has occurred.

Dual-Network Structures

Last but not least, dual-network structures can be a potential solution to the problem of mechanical strength versus self-healing capabilities. Often referred to as the "fourth generation" of self-healing materials, dual network structures combine properties of covalent bonds, providing strength and toughness, and non-covalent interactions, allowing for enhanced chain mobility and flexibility [30].

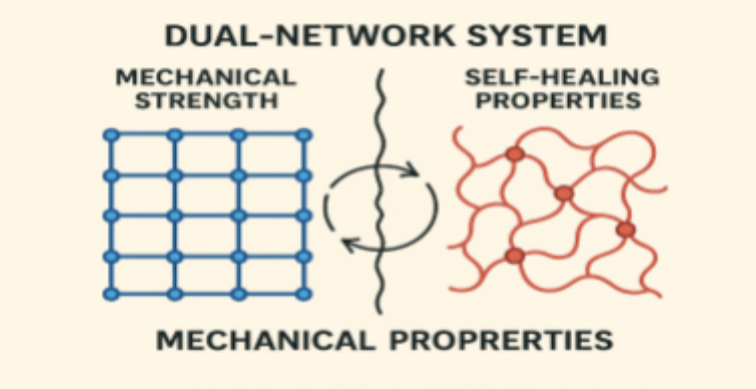

Fig 4: This figure demonstrates how dual-network (joint) systems utilize functions from both covalent and non-covalent interactions to create a synergistic effect. The irreversible covalent bonds provide mechanical strength, as seen on the left. The reversible non-covalent interactions (hydrogen bonds, metal-ligand coordinations, ionic interactions) supply self-healing properties, as shown on the right.

Unlike other covalent-based self-healing systems like vitrimers or Diels-Alder reactions, the covalent bonds in dual-network structures are *not* dynamic and rather are irreversible, meaning that the bonds never break, as they are *permanent* crosslinks [31]. As a result, the covalent bonds in joint systems act as the backbone of the polymeric chain, providing structural stability and maintaining the load-bearing ability of the material. On the other hand, the non-covalent bonds such as hydrogen bonds, metal-ligand coordination, host-guest interactions or other weak intermolecular forces are interspersed throughout the covalent network [32]; the bonds are reversible, signifying their ability to break and reform when exposed to stress. Similar to hydrogen bonds, these non-covalent interactions act as sacrificial bonds, preventing cracks from permeating to the rest of the material [33]. However, hydrogen bonds, due to their high elasticity, while able to maintain stress, often fail to retain the shape of a material under a load, a concept known as creep. Dual-networks, conversely, are able to maintain stress *and* retain the shape of a material under a load, having high creep resistance. Creep resistance is important because dimensional instability could occur in the form of sagging, bulging, or permanent deformation [34].

What sets joint systems apart from other intrinsic self-healing materials is their tunability. In the context of self-healing materials, tunability refers to the ability to adjust and modify a material's properties, by altering the composition or structure of the material to some extent [35]. By doing this, researchers can control stiffness, toughness, healing speed, conductivity, or even temperature triggers as certain scenarios may require an increase or decrease in one or more of these factors. Tunability, in essence, optimizes the performance of a material or a polymer and can be tailored to a wide variety of applications. While other intrinsic self-healing materials also have tunability, they often come with less freedom. Due to their single-network nature of the systems, it becomes inherently harder to adjust certain elements of the material without compromising some of the other properties. For example, increasing the crosslink density may increase mechanical strength, but the polymeric chains will lose their free-flowing mobility to

self-repair. On the other hand, joint systems *independently* change the elements of the two networks, so no properties are ever being sacrificed [36].

Dual-network structures are incredibly useful in real-world applications, as their potential functions span several fields. One area in which joint systems have been specifically associated with is in the energy sector, in which they could potentially store energy in more efficient manners and prevent internal short circuits. This could happen by tuning the ionic conductivity and mechanical robustness to an ideal ratio [37]. All things considered, dual-networks utilizes both covalent and non-covalent networks, creating a synergistic effect, and due to their high tunability factor, they can be used in almost any application without sacrificing either mechanical strength or self-healing properties.

Future Outlook and Conclusion

In conclusion, intrinsic self healing-materials have unlocked a new realm of endless possibilities across all sectors, due to their abilities to autonomously repair damage from external stimuli without human intervention, similar to healing systems of organisms that can regenerate. Having transitioned from extrinsic self-healing based systems that require encapsulated healing agents to intrinsic by utilizing the power of dynamic reversible bonds and supramolecular interactions, self-healing materials have seen faster healing times, enhanced durability, and more efficient healing [38]. While the trade-off between mechanical strength and self-healing capabilities has limited development and scalability until this point, new solutions like vitrimers, Diels-Alder reactions, hydrogen bonds, and dual-network systems can potentially nullify this issue. Each solution has their individual, unique characteristics that make them prime candidates for solving this problem such as shape memory, thermal reversibility, rapid healing cycles, and tunability, respectively. As a result, intrinsic self-healing materials are primed to enter a broad array of fields, ranging from biomedical to aerospace and beyond [39].

The future of intrinsic self-healing materials continues to look very promising, as reports indicate that the global market for these materials is projected to reach $14.60 billion by 2033 with a compound annual growth rate (CAGR) of 21.1% [40]. This rise in rapid demand can be attributed to the world's needs for sustainable and long-lasting materials, as they can act as a counter to the rising greenhouse gases and potentially hinder global warming's progress; not only will self-healing materials will play a pivotal role in addressing these environmental issues, as they will extend product lifespans, minimize waste, and align with circular-economy principles, but also reduce maintenance costs. While ongoing research focuses on finding materials that are more cost-efficient, it is important to recognize that intrinsic self-healing materials have become the epitome of science, due to their widespread revolutionary impact. From specialized research to being incorporated into the mainstream as standard requirements in all industries, intrinsic self-healing materials are set to become the defining blueprints of how we design, use, and maintain materials in a rapidly evolving world.

Acknowledgements

I would like to thank Sara Anjum for mentoring and guiding me through the process of writing a research paper. I would also like to thank my parents for providing me with this opportunity.

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