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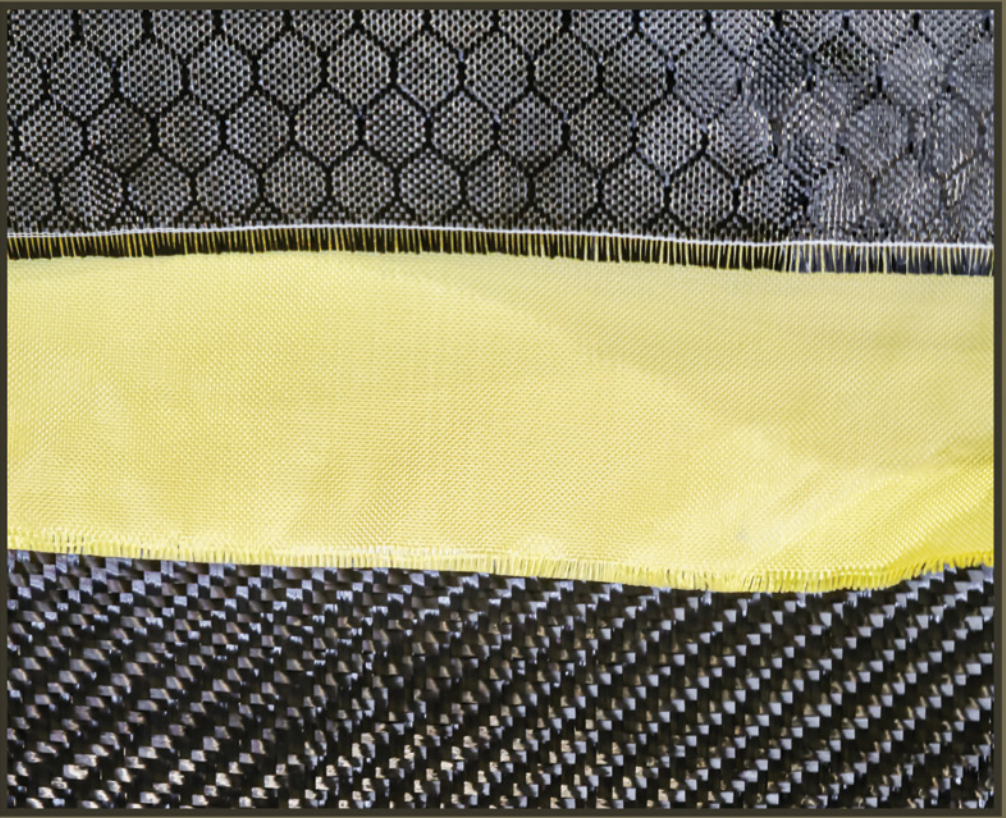

SMART BIOCOMPOSITE MATERIALS

FABRICATION, APPLICATIONS, AND SUSTAINABILITY

Edited by
MD. REZAUR RAHMAN
MUHAMMAD KHUSAIRY BIN BAKRI

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Smart Biocomposite Materials

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Fabrication, Applications, and Sustainability

Edited by

Md. Rezaur Rahman

Muhammad Khusairy Bin Bakri

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Dedication

This work is dedicated to my amazing wife and daughters, Shirin Akther, Fahriah Rahman, and Faizah Rahman, who are very special to me and made it possible for me to complete this work.

—Associate Professor Ts. Dr. Md. Rezaur Rahman

First, I would like to thank the Almighty God for the guidance, strength, power of mind, protection, and for giving us a healthy life. All of these we offer to you. Every difficult task needs self-effort as well as the guidance of elders, particularly those who are near to our hearts. I offer my humble dedications to my beautiful and loving father, mother, wife, and brothers, whose devotion, love, support, and nightly prayers have enabled me to work toward this significant achievement, along with all the dedicated, well-liked, and well-respected teachers and supervisors.

—Ts. Dr. Hj. Muhammad Khusairy Bin Bakri

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Preface

Materials science has now reached a critical phase of development, where combining sustainability, intelligence, and functionality is no longer visionary but the absolute requirement of the time. While the world is facing increasingly aggravated environmental concerns and its industries face growing demands for responsive and high-performance materials, the combination of biobased components with intelligent attributes is an opportunity for innovation, at the same time as it is eco-friendly and technologically advanced.

Intelligent biocomposite materials are one class of engineered systems combining renewable natural fibers with biodegradable or recyclable matrices and functional fillers or additives with responsive behaviors to external stimuli. These materials successfully address the imperative of reducing dependency on fossil-derived resources and possess enhanced features like self-healing, shape memory, and active sensing or actuation functions as well. The last 10 years have seen tremendous development in this field, driven by interdisciplinarity among materials engineering, biotechnology, polymer science, nanotechnology, and applied mechanics.

At the core of this discussion is a deep understanding of the interactions between different components, natural fibers, polymer matrices, and added functionalities. Advances in fabrication technologies, ranging from conventional compounding methods to advanced additive manufacturing methods, have enabled the large-scale production of biocomposites with tailored mechanical, thermal, and functional properties. The combination of responsive functionalities, such as sensors, stimuli-responsive fillers, or drug delivery systems, greatly expands their utility in high-end markets, such as biomedical devices, aerospace components, structural health monitoring, and soft robotics.

An important aspect of research in this area is mirroring the built-in self-sufficiency of nature with the development of materials with self-healing tendencies, the ability to change their shape in the presence of outside stimuli, and the ability to perform regulated-release functions. These attributes improve the potential of biocomposites and open up new prospects for research and application.

Sustainability remains the underlying tenet of this field, including not just the origin of materials but also life cycle assessment, recyclability, and environmental impact considerations. As the world economy increasingly moves toward more circular and biobased models of development, novel biocomposites are expected to play a key role in enabling this transformational process.

This book aims to act as a comprehensive scholarly reference for researchers, engineers, and postgraduate instructors to explore the scientific basis, processing methodologies, functional characteristics, and potential uses of intelligent

biocomposite materials. This book integrates fundamental knowledge and state-of-the-art advancements to prompt further research and enable the development of advanced materials with features like intelligence, long-term performance, and sustainability.

Associate Professor Ts. Dr. Md. Rezaur Rahman
Ts. Dr. Hj. Muhammad Khusairy Bin Capt. Hj. Bakri

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Integration of intelligent sensors and actuators in materials

6

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6.1 Introduction

Certain materials utilized for sensors and/or actuators include highly intelligent microstructures that enable their performance to be altered in response to environmental fluctuations and/or over time. For instance, self-filtering glass and InP semiconductors have self-decay characteristics when exposed to radiation. Nevertheless, it is sometimes the case that a solitary substance lacks the intricate capabilities necessary for many applications. Typically, the expected performance in applications is achieved by combining two or more types of materials. Traditional materials can be augmented with sensors or sensitive parts only for the purpose of bearing loads, to address the mechanical issues such as weight, strength, and fatigue. Nevertheless, the emergence of material science and processing technology has led to the intriguing possibility of creating sensor/actuator systems that combine different functionalities of the compositions. The materials and structures that are produced as a result are referred to as smart materials or intelligent materials. Intelligent materials and structures are composed of sensors and actuators that are connected by a controller, allowing them to have built-in sensing and actuation capabilities. These materials have the ability to detect external stimuli, analyse and process the gathered information, and subsequently carry out actions autonomously. These entities possess the abilities to detect, provide input, identify and gather data, react, analyse their own condition, fix themselves, and adjust to new circumstances. Intelligent materials typically consist of four components: matrix materials, sensor materials, actuating materials, and information processors.

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6.2 What are intelligent materials and how are they made?

The matrix materials serve as load-bearing components, allowing for the selection of lightweight materials. Polymer is typically chosen as the matrix material due to its lightweight nature, excellent corrosion resistance, and particularly its nonlinear viscoelastic properties (Adamu et al., 2019; Hari et al., 2021; Jayamani et al., 2016, 2015; Kiew et al., 2013). In addition, metals and alloys, such as nonferrous light alloys, can also be chosen. Sensing materials are responsible for detecting and measuring changes in the environment, such as pressure, stress, temperature, electromagnetic fields, and pH. Widely utilized sensing materials encompass shape-memory materials, piezoelectric materials, optical fibers, magnetostrictive materials, electrochromic materials, electrorheological materials, magnetorheological materials, liquid crystals, and others. Actuating materials are utilized for reaction and control purposes, owing to their notable reactive properties (such as significant strains and high stresses under specific conditions) in response to environmental stimuli. Actuating materials commonly employed include shape-memory materials, piezoelectric materials, electrorheological fluids, and magnetostrictive materials. Certain types of materials can serve as both actuating and sensing elements simultaneously in composites, providing a practical and efficient method for designing intelligent materials.

Intelligent materials and structures are developed to detect and monitor the conditions of materials in use, such as damage, loads, shape, temperature, and pressure. They are also designed to have the ability to change their deformation, shape, stiffness, inner stresses, temperature, color, and other properties through adaptive actuation. In most situations, controllers have been positioned outside the material, although there have been instances when they have been integrated into the composite itself. The primary matrix material used in these composites is predominantly polymer resin; however, metals or concrete matrices have also been taken into account. Intelligent materials can exhibit varying degrees of coupling between the matrix material and its active parts, ranging from classical mechanical coupling between two structures (as seen in many mechanical systems) to molecular-level design. Smart composites serve as a practical intermediate stage of connection, enabling us to easily create and advance intelligent materials. This leads to a diverse range of intelligent materials with different characteristics.

To create an intelligent material, it is necessary to adhere to rigorous standards, scientific procedures, and precise processing while composing or assembling its components, rather than just mixing or putting many materials together. The integration of software functions with composites is the most appealing technique. The composites that are created possess comparable characteristics to the information processing units found in animals, specifically neurons. These composites combine various software functionalities in diverse structural hierarchies, with a thickness range spanning from a few nanometers to over 10 nm. By adopting this approach, it is possible to include intelligent attributes into the macro or microstructures of the composites. Various technological challenges must be addressed during the design

and production of intelligent composites. An important factor to take into account is the compatibility between the sensing/actuation components and the materials they are integrated. The stiffness ratio among these various materials is an important characteristic. If the actuation or sensing materials are too pliable, it will pose challenges in effectively transmitting the actuation to the host material or the sensing material for the purpose of actuation or sensing designs, respectively. An optimal design guideline is that the stiffness ratio should be within the range of 0.5–2.

The quality of the interface between different materials is crucial, as it must be capable of effectively transferring substantial shear force. Inadequate interface stress transmission can also occur due to chemical incompatibility or the elevated temperature produced by heating the actuation element. It is necessary to take into account the dimensions, configuration, and number of the sensing/actuation components to assess the impact of notches when these components are included in the composite material (Bakri et al., 2018; Fatema et al., 2024; James et al., 2024; Jayamaui et al., 2020). Depending on the specific use case, employing several tiny pieces that are widely distributed may be more advantageous than relying on a single large part positioned at a single location. The position and quantity of sensing/actuation components are highly contingent upon the geometry and function of the component and structure being examined. The optimal design of the intelligent composite material might depend heavily on the overall design of the component.

Precision is crucial in the production of composite materials for sensors and actuators, since it is imperative that the sensors, actuators, and wire remain unaffected by mechanical, thermal, or chemical reactions during the manufacturing process. This pertains to the attributes of the entities and their placement inside the composites. Manual laying and bonding can provide sufficient care, but it is the costliest method. Currently, preliminary research is being conducted to include active fibers, such as those composed of shape-memory alloys (SMA) or piezoelectric materials, utilizing automated processes. Automated fiber and tape laying can be considered an option; however, these technologies must still be modified to suit the unique characteristics of these novel composite materials. In addition, there are several concerns that require attention while developing and producing intelligent composites. Examples of topics covered include the link between components, mathematical models, simulations based on biological principles, computation techniques for intelligent systems, the mechanics of systems at both macro and meso scales, the interaction between many parts, the features of phase transitions, and the control of hysteresis. These difficulties necessitate further extensive, methodical, and thorough investigation.

6.3 Humidity sensor and smart actuator

In recent times, a wide range of miniature soft robots have been created to imitate organic organisms. These robots rely on a soft actuator, which is a crucial component that enables them to perform many types of movements due to its flexible and adaptable construction. To ensure the effectiveness of soft actuators, such as hydrogel or film actuators, it is necessary to build them using dependable heterostructures (Wei

et al., 2021). Nevertheless, the presence of a heterogeneous structure is not an essential characteristic of the actuator, as many environmental stimuli, such as light and humidity, possess inherent "potential directionality." More precisely, the interaction between these ambient inputs and the uniform structure might lead to the formation of directionality and heterogeneity (Du et al., 2022). When water vapor comes into contact with a composite film made of cellulose nanofiber (NF)/graphene oxide/carbon nanotube (CNT) with a uniform structure, the film quickly bends because the water is rapidly absorbed on one side of the film, causing that side to expand in volume. This leads to a disparity in volume between the side where water is absorbed and the opposing side, resulting in heterogeneity in bending behavior. Therefore the presence of diverse characteristics is a result of the influence of ambient influences on the uniform structure. Actuators with homogeneous structures are significantly more straightforward to manufacture compared to intricate designs with heterogeneous components. Homogeneous film actuators have gained significant attention in research due to their straightforward fabrication process and rapid environmental response speed.

Due to their environment-friendly character and the widespread occurrence of water in the natural environment, the development and production of uniform film actuators that are responsive to humidity are often seen (Li, 2021). Various hygroscopic materials, such as sodium alginate (SA) (Xiang et al., 2023), chitosan (Huang et al., 2021), and cellulose nanocrystals (CNC) (Ge et al., 2022), are combined to create laminar-flow membranes with high purity. These membranes provide consistent actuation qualities that prevent needless interlayer stripping. However, their limited range of response restricts their potential applications. Soft actuators specifically engineered for versatile environmental adaptability possess a clear superiority over single-response equivalents in intricate real-life environments. Simultaneous environmental stimuli, such as temperature, humidity, light, and electrical impulses, are frequently encountered in real-life situations. Actuators designed to handle a range of environmental inputs not only expand the potential applications but also enable cooperative interaction among different stimuli. This collaboration allows actuators to adapt to complicated surroundings and perform tasks with enhanced intricacy and accuracy. The double-sided azobenzene antiopal actuator utilizes the combined effect of the Janus structure and the modification of plasticity generated by the reaction of polymer segments to solvent, heat, and light. The complex design of this actuator enables it to effortlessly transform into a ring form and perform quick rolling movements. These actions are made possible by the combined effect of three external stimuli (Liu et al., 2021). The ability of soft actuators to react to various stimuli is a crucial factor in determining their potential for practical use.

Creating film materials using a combination of two-dimensional (2D) nanomaterials and matrix networks has been shown to be an effective approach for enabling films to react to various stimuli (Namakka, Rahman, Bin Mohamad Said, et al., 2024; Namakka, Rahman, Mohamad Bin Said, et al., 2024; Namakka et al., 2023). In addition, 2D nanomaterials can enhance the mechanical and actuation characteristics of the actuator and aid in achieving asymmetric hydration by augmenting the

material's resistance to the diffusion of water molecules from one side to the other (Li et al., 2021). Recently, there has been a growing interest in utilizing 2D nanomaterials, such as graphene, MXene, and MoS_2, to create high-performance actuators. These nanomaterials have unique structures and have shown promising results in terms of driving performance (Namakka et al., 2023). This highlights the significant potential of new 2D materials in the development of advanced actuators. The bilayer soft actuator, which combines polydimethylsiloxane (PDMS) ionic gels with graphene oxide and reduced graphene oxide (RGO) films, has a remarkable ability to respond in various environments. By using the hygroscopic properties of graphene oxide, the actuator demonstrates a high level of responsiveness to changes in humidity. Moreover, it exploits the photothermal conversion characteristics of graphene oxide and the incongruent expansion between the two layers, allowing for a light-responsive behavior. Moreover, the electrothermal conversion capacity of RGO improves the actuator sensitivity to electrical stimuli. As a result, this bilayer soft actuator demonstrates exceptional performance in various environmental conditions, such as humidity, light, and voltage (Chang & Lin, 2024). Black phosphorus is a recently discovered 2D nanomaterial characterized by its distinct layered structure, which make it very promising for applications in biomedicine (Hou et al., 2020) and energy storage (Wang et al., 2023). Black phosphorus demonstrates thermal expansion (Sansone et al., 2018), rendering it a suitable choice for constructing actuators. An innovative photo-actuator was created by combining a heterogeneously structured combination of black phosphorus and CNTs. This photo-actuator exhibited exceptional performance in terms of light-driven functionality and self-oscillation. Black phosphorus has a strong photothermal effect and thermal expansion, making it a highly suitable material for generating rapid photo-response reversible drive and exceptional bending deformation (Hu et al., 2021). Polydopamine (PDA), which takes its inspiration from mussels in nature, is a very adaptable coating that has exceptional adherence on nearly all surfaces and possesses a strong near-infrared (NIR) photothermal effect (Lan et al., 2020). Coating a 2D nanomaterial with PDA often improves its photothermal conversion capability, enhances water dispersibility, and safeguards the interior structure from oxidation (Xu et al., 2020). A liquid crystal film actuator was created by applying a selective coating of PDA over a liquid crystal matrix doped with azobenzene. This actuator exhibits dual responses to both NIR and ultraviolet (UV) light. The use of the PDA coating not only greatly increased the photothermal effect but also effectively prevented UV light from causing photo-heating. As a result, the liquid crystal film became programmable (Shen et al., 2021). Magnetic-responsive nanomaterials have garnered significant interest due to their rapid reaction rate, heightened sensitivity, excellent reversibility (Namakka, Rahman, Mohamad Bin Said, et al., 2024), and applicability for noncontact manipulation (Yadid et al., 2023). By manipulating the physical and chemical characteristics of magnetic nanoparticles, we can accurately forecast their path when subjected to a magnetic field. This makes them highly versatile for many uses, including medication administration and magnetocaloric physiotherapy (Ling et al., 2021).

When the black phosphorus (BP)@PDA@Fe_3O_4 (BPPF)@2,2,6,6-tetramethylpiperidinooxy-oxidized bacterial cellulose (TOBC) film is subjected to a high-humidity

environment, it rapidly takes in water, causing the side exposed to the high humidity to expand due to the large number of hydrophilic carboxyl groups and hydroxyl groups present in TOBC. Furthermore, the rapid formation of hydrogen bonds occurs between BPPF nanosheets, hydrophilic groups such as –COOH and –OH groups on the TOBC chain, and the water molecules that have been absorbed. The opposite side does not undergo the high-humidity conditions necessary for the absorption of water molecules, so it does not undergo any swelling. The disparity in volume between the two sides induces tension in the initially uniform film, causing it to bend towards the side with lower humidity.

The film's reaction to NIR light is strongly correlated with its sensitivity to humidity. Due to the efficient NIR photothermal conversion shown by BPPF nanosheets, when one side of the film is exposed to NIR light, the temperature on that side increases rapidly. This causes the moisture on the side to evaporate quickly, creating a moisture gradient inside the film. As a result, the films bend. Given that the NIR light response of the BPPF@TOBC film is derived from the nanosheet photothermal conversion capacity, our initial investigation focused on assessing the photothermal conversion capacities of several nanosheets. After analyzing the mechanical properties, it was determined that the optimal quantity of TOBC is 40 mg.

The film actuator demonstrated a robust mechanical strength of 51.88 MPa and a high level of toughness at 3.93 MJ/m^3. Additionally, it showcased a fascinating humidity-sensing mechanism. The film's responsiveness to humidity is attributed to the high water-absorption capacity of TOBC and the ability of BPPF to restrict the spread of water. When there is a difference in humidity levels, water molecules can readily be absorbed into the film, resulting in uneven expansion on both sides of the film. The film's reaction to NIR light is attributed to its effective NIR photothermal conversion facilitated by BPPF. The rapid increase in temperature leads the water molecules in the film to undergo evaporation, leading to the formation of an asymmetric structure. The film's magnetic reaction is attributed to the strong magnetism of BPPF. Hence, by intentionally altering 2D nanomaterials and incorporating them into a compatible framework, actuators may be endowed with desired characteristics. The BPPF@TOBC film possesses favorable environmental reaction and mechanical qualities, allowing it to serve as a pliable gripper, circuit switch, and controlled mover for various jobs in diverse situations. Due to its properties, this material has great potential for usage in advanced domains with a wide variety of applications, such as those that need sophisticated environmental response and intelligent management.

Actuators transform various external stimuli, such as electric, electrochemical, optical, thermal, and other forms of stimulation, into deformation or motion. Actuators may be seen as an energy conversion mechanism that transforms external energy into mechanical energy. Graphene possesses superior mechanical, electrical, optical, and thermal characteristics in comparison to conventional actuating materials, making it a highly suitable choice as an actuation material (Huang et al., 2012). Graphene-based actuators may be manipulated by an external input to experience a reversible alteration in their form, volume, or other mechanical characteristics. Graphene exhibits strain variations in its planar structure when charge is injected,

mostly due to the presence of the electrostatic double layer and quantum-mechanical effects. Meanwhile, the surface tension and edge stress contribute to its high flexibility and susceptibility to bending.

Electrically stimulated actuation is a widely used method where the ability to regulate the injection of charge is transformed into mechanical motion (Jung et al., 2011). Applying an electrical field to graphene sheet can result in two primary outcomes (Yu et al., 2017). Initially, the injection or extraction of charge from graphene sheets causes the film to expand or contract. Furthermore, a portion of the electric power is transformed into heat energy as a result of the resistance shown by the graphene layer. Chang et al. (2019) created a flexible bimorph actuator using a process that involved applying a layer of graphene by drop-coating and then attaching polypropylene (PP) to the graphene film (Chang et al., 2019). The actuator displayed a flexural deformation when subjected to electrical stimuli, which may be attributed to the electrothermal characteristics of the RGO, the thermal expansion of the PP film, and the bimorph structure. Furthermore, this actuator is capable of demonstrating optically stimulated movement. When exposed to the stimuli, this actuator produced an angle shift exceeding 100 degrees. It had a reaction time of around 8 seconds and required a driving voltage of no more than 7 V. In addition, a biomimetic device consisting of four actuator components was created to mimic an artificial flower. This device demonstrated electrically controlled blooming. Yang et al. (2017) incorporated RGO nanosheets into a hydrogel made of poly(2-acrylamido-2-methylpropanesulfonic acid-*co*-acrylamide) (poly(AMPSco-AAm)) to enhance its actuation capabilities. The addition of RGO nanosheets created physical cross-links between the poly(AMPS-coAAm) chain, leading to a uniform distribution inside the hydrogels. This allowed for efficient dissipation of strain and transmission of load between the polymer networks and the RGO nanosheets. The exceptional electrical conductivity of RGO nanosheets enhances the movement of ions inside the hydrogels, resulting in increased electro-responsive features such as deswelling/swelling and bending/unbending capabilities. Simultaneously, the inclusion of RGO nanosheets resulted in an enhancement in both tensile strength and compressive strength. This may be attributed to the composite structure formed by the polymer matrix and RGO nanosheets, which facilitates the dissipation of energy. The suggested nanocomposite hydrogels have the ability to be produced into soft robotics, such as cantilevers and grippers, due to their electro-induced bending capabilities.

Electrochemically stimulated actuation refers to the activation of actuator materials by the introduction of ions in an electrochemical environment. This method eliminates the requirement for temperature differences to activate the actuators. Typically, these actuators are made up of a bilayer structure, where two materials on opposite sides of the actuators respond differently to the identical electrochemical circumstances, causing the actuation to occur (Liang et al., 2011). Hyeon et al. (2019) created coiled graphene/CNT yarns using the biscrolling technique to enhance capacitance. These yarns are capable of generating higher tensile actuation by using a greater number of ions at the same voltage compared to pristine CNT coils (Hyeon et al., 2019). Graphene, when used as a guest material in CNT yarn, enhances the

capacitance of the entire artificial muscle system. When a three-electrode system is used at a voltage of −3 V, the graphene-based yarn can achieve a tensile actuation of 19%. This is about 2.5 times more than the coiled bare CNT muscle, which has a tensile actuation of 8%. Additionally, the graphene-based yarn has a work capacity of 2.6 J/g. The use of biscrolled graphene significantly improved the performance of producing yarn structures, resulting in enhanced actuation capabilities. Yang et al. (2019) incorporated PDA functionalized graphene nanosheets (GNS) into the carboxylated nitrile butadiene rubber (XNBR) matrix to create high-quality dielectric composites. This was achieved through the formation of hydrogen bonds between the –COOH groups of XNBR and the phenolic hydroxyl groups of GNS–PDA during the mixing process with latex (Yang et al., 2019). The XNBR composite filled with GNS–PDA exhibited greater dispersion of the filler, improved dielectric constant and dielectric strength, and reduced conductivity in comparison to the XNBR composite filled with pure GNS. The GNS–PDA/XNBR composite exhibited a simulated strain of 2.4% when subjected to an electric field of 18 kV/mm. This simulated strain was much greater than the 1.3% strain observed in pure XNBR under the same electric field.

In addition to electrically and electrochemically driven actuations, there have been studies documenting the use of various stimuli to generate graphene-based actuators. Graphene is a hydrophobic substance. Graphene oxide, being derivative of graphene, possesses a multitude of oxygen-related functional groups that facilitate rapid adsorption and desorption of water molecules. A recent development involved the creation of humidity actuators using graphene materials that exhibit hydrophilic and hydrophobic properties. Park et al. (2010) documented the initial instance of a macroscopic actuator made from graphene, achieved using a straightforward process of sequentially filtering an aqueous solution of multiwalled CNTs (MWCNT) and GO platelets. The bilayer paper composed of GO and MWCNT has the ability to undergo curling in response to changes in humidity or temperature. The paper was rolled up with the side containing MWCNT facing outward, under low relative humidity conditions of 12%. As the relative humidity increased, the bilayer paper slowly unfolded, reaching a nearly flat state at a relative humidity of 55%–60%. When the relative humidity surpassed 60%, the bilayer paper began to curl, with the GO side facing outward in the opposite direction. Utilizing two distinct building blocks to create asymmetric materials has shown to be an effective method for producing mechanical actuation. In addition, Xu and Hsieh (2019) created a uniform and consistent mixture of graphene and cellulose nanofibrils, resulting in nanopaper. The efficient method of exfoliating graphene-based nanopaper using amphiphilic cellulose nanofibrils as multifunctional agents for exfoliation, dispersion, and structure formation in aqueous solution resulted in a much quicker response time of only one second (Rahman et al., 2013; Rahman, Hamdan, Hasan, et al., 2015). The combination of cellulose nanofibrils paper, which has a high capacity to absorb moisture, and graphene paper, which is not affected by moisture, has created opportunities for a wide range of applications in moisture or vapor actuators. However, the development of functional cellular 3D macrostructures that are effective has proven to be difficult. Xiang et al. (2020) created a flexible actuator

that responds to moisture by uniformly distributing 2D graphene oxide (GO) sheets into one-dimensional polyvinyl alcohol-*co*-ethylene (PVA-co-PE) NFs and silver nanowires (AgNW). The GO sheets serve as a continuous moisture-sensitive component dispersed within a three-dimensional (3D) network (Xiang et al., 2020). The moisture sensitivity of the AgNW/NF/GO composite membrane was enhanced by the hygroscopic features of GO. Additionally, the inclusion of AgNW allowed the membrane to exhibit significant bending deformation on a wide scale, rapid reaction to moisture, and stable performance throughout cycling. Experimental evidence demonstrated that the bending deformation of the membrane-based actuator, when exposed to moisture, was primarily influenced by the absorption of water between the layers. This water absorption directly led to an expansion of the volume of the film, while the absorption of water on the surface had a detrimental impact on the bending angle of the membrane.

6.4 Intelligent polymers

During the 1990s, some organic materials were created by replicating the sensing, processing, and executive capabilities of biological tissue, resulting in the emergence of intelligent polymers that appear to possess human emotions and perceptions. The system consists of a 3D polymer network and solvent, capable of undergoing volume phase change, provided specific criteria are met. The network exhibits insolubility and retains its original form by virtue of its cross-linked topology. Hydrophilic groups present in the network structure can be expanded by a solvent and achieve a state of equilibrium volume. The swelling phenomenon is primarily driven by the contact between macromolecule chains and solvent molecules, the interaction among macromolecular chains within the network, and the osmotic pressure resulting from varying ionic concentrations between the gel and the external medium. Hence, these particular polymers have the ability to detect minute variations in the surrounding environment, such as temperature, pH level, or electric field. External stimuli that elicit the reaction of intelligent polymers can be either physical (such as temperature, electric field, magnetic field, and light stimuli) or chemical (such as pH response, molecule recognition, and oscillation reaction). Intelligent polymers have the potential to be utilized in several domains, including optical sensors, thermal sensors, pH sensors, ion-selective sensors, artificial muscles, micromachinery actuators, displays, optical communications, medicine, catalyst carriers, biotechnology, and intelligent fabrics, based on their response characteristics. Through the integration with other materials, it has promising prospects in the realms of functional carriers, environmentally conscious surfaces, and intelligent textiles.

The proliferation of artificial intelligence and the intelligence community have led to a significant focus on wearable electronics. These devices have great promise in several domains such as healthcare monitoring, human–machine interfaces, and artificial intelligence technology (Liu et al., 2022). Due to its significant advantages, including high flexibility, outstanding integrability into smart textiles, and comfortable attachment ability with the human body, conductive fibers with high resilience

are sought to be produced and employed in intelligent wearable devices (Qu et al., 2022). A crucial aspect of producing stretchable conductive fibers is the assembly of conductive materials onto the surface of the polymeric substrate or the encapsulation of conductive materials inside a protective layer to create a conductive core. The manufacturing methods include direct coating, wet spinning, printing, and electrostatic spinning. Coating polymers, including polyaniline (PANI), polypyrrole (PPy), and poly(3,4-ethylenedioxythiophene):poly(4-styrenesulfonate) (PEDOT:PSS), offer significant advantages in the production of stretchable conductive fibers compared to metal nanomaterials and liquid metals. These advantages stem from their lightweight nature, intrinsic high conductivity. and biocompatibility (Wu, 2023).

Chen et al. (2024) produced a composite fiber (PU@PEDOT:PSS fibers) consisting of a mechanically stretchable and electrically conductive core made of H_2SO_4-doped PEDOT:PSS hydrogel and a robust sheath layer made of PU (Chen et al., 2024). The size and performance of the fiber were modified by controlling the fluid composition and processing parameters during MST. The generated PU@PEDOT:PSS fibers were integrated into a textile material suitable for use as a 2D triboelectronic nanogenerator (TENG) or a wearable electric heating fabric. Furthermore, the PU@PEDOT:PSS fibers showed significant conductivity fluctuations during extension, making it a highly efficient fibrous strain sensor with both exceptional sensitivity and a broad detecting range.

The PU@PEDOT:PSS fiber exhibits remarkable stretchability due to the integration of an elastic PU shell and a deformable PEDOT:PSS hydrogel core. This characteristic makes it very well-suited for various advanced smart wearable applications, including conductive fabric, strain sensor, triboelectric nanogenerator, and electrothermal heating textile. Upon extension, the H_2SO_4-doped PEDOT:PSS hydrogel within the core of the PU@PEDOT:PSS fiber fractures into several pieces, resulting in a notable alteration in its conductivity. Hence, the microfiber has the capability to function as a strain sensor for detecting the extent of extension, so demonstrating significant potential for usage in intelligent wearable devices. The detecting capabilities of the PU@PEDOT:PSS fiber-based strain sensor are significantly affected by the composition of the conductive core. This composition determines the density of the sensing network and the extent to which it breaks up during extension-induced deformation. The sensing capabilities of the PU@PEDOT:PSS fiber-based strain sensor are influenced by the PU content in the middle fluid. This effect is due to the diverse initial conductivities of the microfibers that are spun by the middle fluid with varying PUI contents.

Hyaluronic acid is a principal element of the extracellular matrix, and its derived hydrogels exhibit biocompatibility, biodegradability, and adjustable mechanical properties, which can diminish the rejection of biological tissues and improve their stability within the organism. The hydrogel demonstrates exceptional penetration capability, effectively resolving the challenges of sluggish absorption and low efficacy typically linked to traditional drug delivery techniques. Consequently, it promotes sustained drug release and effective delivery of tissue nutrients (Chang et al., 2015). PDA, an inorganic nanomaterial exhibiting NIR photoresponsivity, holds significant potential for application in the biomedical sector (Du et al., 2021).

Thermotherapy has been utilized since antiquity to facilitate the healing of somatic ailments and the restoration of bone defects through thermal simulation (Tong et al., 2019). NIR (808 nm)-guided photothermal therapy (PTT) has garnered significant interest in antimicrobial therapy and bone regeneration research as an innovative medical intervention (Tan et al., 2021). In this process, the localized thermal effect produced by PTT can significantly encourage bone mesenchymal stem cells to undergo osteogenic differentiation, thereby facilitating bone tissue repair and regeneration (Wu, Sun, et al., 2023).

To investigate the photothermal conversion capacity of the materials, hydrogels with varying particle concentrations were subjected to 808 nm laser (1 W/cm^2) irradiation to measure the temperature variation over 10 minutes. The temperature of the hydrogels with 0.5–2 mg/mL of modified PDA (PZP) increased to 25.5°C–60.3°C, respectively (Wei et al., 2024). The studies demonstrated that PZP nanoparticles displayed concentration-dependent thermal variations under NIR (1 W/cm^2) irradiation. In comparison, the blank hydrogel group experienced a temperature increase of only 3.3°C, whereas the hydrogels with varying concentrations of PZP particles exhibited a consistent and moderate rise in temperature, a finding corroborated by thermal imaging results. Furthermore, utilizing the negative natural logarithm of the cooling time ln θ, they get the values of ts (ts=414.9121), which facilitate the computation of the photothermal conversion efficiency (η), yielding an approximate value of 44.6%. η is a crucial aspect of PTT, which directly influences the intensity of the irradiated light throughout the treatment (Xi et al., 2020). The exceptional photothermal conversion efficiency, coupled with its requisite low intensity, surely inflicts minimal or no harm to adjacent healthy tissue cells. Subsequently, photostability was assessed, and three cycles of pulse irradiation (totaling 30 minutes) did not appreciably alter the gel state of the hydrogel, showing notable thermal stability. To compare the photothermal performance of hydrogels with liquids, they investigated the photothermal conversion efficiency of PZP solutions. The photothermal conversion efficiency of the PZP solution is 43.47%. The superior thermal stability and enhanced photothermal conversion efficiency of hydrogels modified PDA (PZPG) demonstrate that a single minimally invasive method (hydrogel implantation) can deliver sufficient PTT in in vivo studies. In conclusion, the PDA-coated nanoparticle hydrogels exhibit significant NIR light absorption, elevated photothermal conversion efficiency, and exceptional photothermal stability, making them suitable for effective photothermal conversion and storage, and positioning them as a promising photothermal material.

Hyperthermia therapy has been used for healing somatic diseases and restoring bone defects since ancient times (Leon, 1993), with thermal osteogenesis being more desirable due to its on-demand performance independent of the physiological environment. However, in vivo application of heat-stimulated osteogenesis is limited, and strategies such as direct heating, microwave heating, and photothermic have been proposed (Leon, 1993; Weber et al., 2006; Serrat et al., 2015). NIR light is used in biomedicine to trigger biological events noninvasively due to its good transmittance through biological tissues and high spatial and temporal precision. Various nano agents have been proposed to target tumors for NIR-mediated photothermal tumor

ablation due to their enhanced permeability and retention effects and poor thermal tolerance (Yin et al., 2014). This therapeutic strategy can be realized if bone substitutes at defect sites photothermally respond to external NIR light, accelerating bone healing. Tong et al. (2019) studied a degradable biocomposite made of black phosphorus nanosheets (BPs) and poly(lactic-*co*-glycolic acid) (PLGA) is designed for photothermally remote control of bone regeneration (Tong et al., 2019). PLGA is a degradable biopolymer approved by the FDA, commonly used in tissue engineering and producing innoxious end-products like H_2O and CO_2. BPs have been employed as photothermal agents due to their excellent NIR response and ability to flaw polymeric shells, promoting bone regeneration via bioactive strontium component release (Wang et al., 2018). By incorporating BPs into the host PLGA polymer, the obtained BPs@PLGA can facilitate photothermal osteogenesis directly after inheriting the NIR-mediated characteristics and excellent biodegradability.

The black phosphorus nanosheets and poly(lactic-*co*-glycolic acid) (BPs@PLGA-60) group exhibits superior in vitro performance, attributable to the influence of physiological stress. To investigate the fundamental process of osteogenesis following photothermal stimulation, human bone mesenchymal stem cells (hBMSCs) derived from several experimental groups are analyzed for the expression of heat shock protein (HSPs) (Wang et al., 2018). Gene expression of HSP 47 and HSP 70 are two representative HSPs in mammalian cells, whose function in facilitating osteogenesis has been well established (Dafforn et al., 2001). HSP 70 is closely linked to cellular thermos-tolerance, while HSP 47 is specifically associated with heat shock and is essential for the biosynthesis and molecular maturation of osteogenesis-related type I collagen by regulating collagen-cross-linking to produce a distinctive extracellular matrix. The real-time PCR assay indicates that the gene expression levels of HSP 47 and HSP 70A-C (three distinct genes of the HSP 70 protein associated with various stressors) (Lepock, 2003) in hBMSCs cultured on BPs@PLGA groups remain constant. Concerning the protein expressions of HSP 47 and HSP 70, there is no significant difference between the PLGA groups and BPs@PLGA-0; however, the levels of HSP 47 and HSP 70 in BPs@PLGA-60 are 66.2% and 50.1% more than those seen in BPs@PLGA-0. In summary, the BPs@PLGA sample can enhance the production of cellular HSPs and hence promote osteogenesis through distant NIR irradiation.

Mechanical systems can suffer surface and internal damage due to processing and use, affecting safety and service life. The development of composites or coatings with self-lubricating and self-healing functions offers a new way to develop safer and more durable products (Imani et al., 2018; Xiong et al., 2022). Self-lubricating composites based on oil-containing microcapsules have shown excellent lubrication performance, comparable to liquid lubrication. However, increasing microcapsule size or content can decrease the mechanical properties of composites, affecting their practical applications. Polymer materials have the ability of in situ multiple self-healing, but healing efficiency depends on the responsiveness of dynamic bonds and the fluidity of chain segments. Currently, there are few studies on self-lubricating and self-healing bifunctional polymer materials. Various strategies have been proposed, such as the swelling effect of polymer in polar solvent and an intrinsic bifunctional

coating (Cao et al., 2020). Gong et al. (2023) synthesized a series of multishell oil-containing microcapsules with enhanced solvent resistance and interfacial adhesion as lubricant fillers, inspired by the high strength and hydrogen bond structure of spider silk (Gong et al., 2023).

Bifunctional composites were synthesized by integrating intrinsic self-healing matrices with external self-lubricating microcapsules. At a standard load of 6 N and a sliding velocity of 36 mm/s, the coefficient of friction (COF) for self-healing polymer (EP-U) (0.64) is comparable to that of modified self-healing polymer (EP-D230); however, the wear rate decreases by approximately 41%, indicating superior wear resistance (Gong et al., 2023). The superior mechanical properties of the matrix material might impede the initiation and advancement of cracks during friction, hence enhancing wear resistance (Huang et al., 2016). Following the incorporation of self-lubricating microcapsules, various composites have a pronounced lubrication effect, signifying the successful integration of oil-containing microcapsules into the synthesized EP-U, which release during the friction process. The lubricating efficacy of bifunctional composites incorporating metal microcapsules is constrained by insufficient oil content and the substantial mass fraction of silver (Ag) within the microcapsules. Nonetheless, the bifunctional composites, including polymethyl methacrylate/hydroxyethyl cellulose (PMMA/HEC) microcapsules, demonstrate an exceptionally low COF (0.032) and wear rate (8.09×10^{-8} mm^3/Nm) with a microcapsule mass fraction of merely 5%. In comparison to the pure epoxy resin EP-D230 (COF of 0.66, wear rate of 3.44×10^{-8} mm^3/Nm), the COF and wear rate of EP-U-5%PMMA/HEC decrease by one and three orders of magnitude, respectively. The scratch damage self-healing tests are utilized to assess the self-healing capabilities of bifunctional polymer composites. After 3 hours at 80°C, the scratch depth and width of EP-U-5%PMMA/HEC diminish from 90 and 150 to 30 and 60 μm, respectively, with a self-healing efficiency rate of around 58%. At a healing temperature, the efficiency rate attains 78%, comparable to that of EP-U. To assess the impact of microcapsules on self-healing properties, the scratch damage self-healing efficacy of EP-D230-5%PMMa/HEC was examined under identical conditions. The volume of the scratch remains mostly constant or may even increase upon heating. The self-healing characteristics of bifunctional polymer composites are attributed to the EP-U matrix. An increase in the content or size of microcapsules adversely affects the self-healing performance of composites (Sueraya et al., 2024; Rahman, Hamdan, Hashim, et al., 2015; Rahman et al., 2011; Lai et al., 2015; Kuok et al., 2024; Rahman et al., 2019, 2017, 2024). The self-lubricating mechanism of bifunctional polymer composites primarily derives from the release of lubricating oil contained in microcapsules when subjected to an applied force. The microcapsules rupture during the wear process, allowing the lubricant to flow to the friction surface and execute its self-lubricating function. Discernible oil droplets are visible on the friction surface. The self-healing capability of bifunctional polymer composites is ascribed to the movement and reorganization of reversible hydrogen bonds within the matrix when subjected to elevated temperatures. The 3D covalent cross-linking network of epoxy resin is relatively rigid, preventing the molecular chain from flowing flexibly, which guarantees the material's dimensional stability and strength.

In contrast, the supramolecular network can fracture and reform flexibly without a catalyst, imparting self-healing properties and toughness to the polymer. Simultaneously, HEC with active hydroxyl groups in the microcapsule shell can establish noncovalent interactions with both PMMA and EP-U, facilitating an effective equilibrium of the mechanical, lubricating, and healing properties of the composites.

6.5 Intelligent piezoelectric

The urgent need for alternative energy sources due to depleting fossil fuel reserves, global warming, and environmental pollution has led to significant research and effort in developing clean energy sources such as solar, wind, nuclear, fuel cells, and batteries (Dowarah et al., 2024). Energy harvesting, which involves scavenging waste ambient energy and converting it into useful electrical energy, is an attractive approach due to its cleanliness, sustainability, and easy accessibility. However, only small levels of power are obtained through energy harvesting, making it primarily recognized for low-power applications/devices (Watabe & Leaver, 2023). Advancements in semiconductor manufacturing have resulted in technological developments in miniature electronic devices, which have broadened functionality and applicability while enhancing energy efficiency. This makes energy harvesting ideal for powering these devices. Additionally, advances in semiconductor manufacturing and medical sciences have led to the development of next-generation wearable or portable electronic devices, artificial electronic skins, sensors, personalized healthcare monitoring, real-time disease diagnosis, and medical implants (Li et al., 2014). Energy harvesting has attracted significant academic and industrial interest, as it is considered an attractive proposition for the realization of "battery-free" self-powered devices. Self-powered devices with integrated power harvesters/generators can convert surrounding energy into electrical energy, making them fully independent. Energy harvesting is an economical, environment-friendly, and sustainable alternative energy source, reducing battery costs, limiting battery disposal, and utilizing waste ambient energies (Liu et al., 2018).

Mechanical energy, including vibrations and random displacements from human activities, is a versatile and ubiquitous source of energy. Conversion of mechanical energy to electrical energy can be achieved through four transduction principles (Jing & Kar-Narayan, 2018): electrostatic, electromagnetic, piezoelectric, and triboelectric. Recent research has focused on mechanical energy harvester based on these principles, with the relative advantages and disadvantages documented (Wang, 2013; Jing & Kar-Narayan, 2018; Li et al., 2023). However, mechanical energy harvesting faces challenges such as low output power, low energy conversion efficiency, variation in mechanical vibrations, difficulty in integrating harvesters into portable and miniature devices, and the need for a single material that can effectively harvest a wider range of ambient vibrations (Toprak & Tigli, 2014; M′boungui et al., 2015; Todaro et al., 2017). Traditional piezoelectric materials, such as lead zirconate titanate (PZT), zinc oxide (ZnO), and lead manganese niobate-lead titanate (PMN-PT), are extensively used in the fabrication of nanogenerators due to their higher

output power but face issues like being hard, brittle, and toxic. Piezoelectric materials have gained widespread attention in recent years due to their sensitivity to external/ambient vibrations, inherent reciprocal conversion capability, and simpler architecture (Ogbonna et al., 2022). Many studies have designed various energy harvesters for converting mechanical vibrations into useful electrical outputs. New materials like biowastes and their hybrids with polymers have also been reported for energy harvesting applications (Maiti et al., 2017; Sahu et al., 2022). It is evident that harvesting waste/ambient mechanical energy and transforming it into electrical energy via piezoelectric materials is a progressively evolving technology of the future.

Nearly 18 years ago, Z.L. Wang of the Georgia Institute of Technology presented the concept of self-powered nanotechnology, leading to a revolution in energy harvesting and sensing systems (Wang & Song, 2006). The concept of a self-sustaining sensing system was originally introduced utilizing piezoelectricity, and then expanded to incorporate pyroelectricity and triboelectricity a few years later. The three principles led to the development of three distinct energy harvesting technologies: piezoelectric nanogenerator (PENG) (Wang & Song, 2006), TENG (Wang, 2013), and pyroelectric nanogenerator (Yang et al., 2012). The initial two proposals transform kinetic energy into usable electrical power, whereas the last concept generates electrical power from thermal energy (Askari et al., 2018). Nearly 140 years ago, Pierre and Jacques Curie identified the piezoelectric phenomenon, leading to the creation of various sensing and energy harvesting systems (Katzir, 2018). Nearly century later, Z.L. Wang discovered the PENG (Wang & Song, 2006). His study demonstrated that nanoscale mechanical energy may be transformed into electricity via piezoelectric zinc oxide nanowire (NW) arrays. The notion of a nanoscale PENG, capable of harnessing ambient motion to energize nano electronics and provide battery-free operation, was highly attractive. By employing arrays of nanoscale zinc oxide piezoelectric NWs oriented perpendicular to a sapphire substrate, they were able to integrate the piezoelectric and semiconducting properties. Mechanical energy may be transformed into electricity by the vibration of NWs. The electricity generation process of Wang's first nanogenerator relies on the concentration of charge on the side and at the bend during deflection, which triggers current generation upon its release in a reciprocating motion (Wang et al., 2007). Since that time, the PENG has undergone ongoing enhancements regarding electrical output and integration into novel applications. Over the past 18 years, we have observed significant advancements in the electrical output of PENG. The initial PENG devices were capable of producing electrical currents and voltages limited to the scale of nanoamperes and several millivolts. The electrical output has significantly escalated to hundreds of volts and hundreds of microamperes over a span of less than two decades (Xu et al., 2021). The second technology, known as TENG, was identified as a novel energy harvesting method in 2012. Since then, they have been utilized to create various energy harvesting devices and autonomous sensing systems (Gao et al., 2021). They have been utilized in several applications, including biomedical devices, intelligent transportation, ocean wave energy collecting, wind farms, automotive systems, and human-to-machine interfaces.

A primary problem in the utilization of electric cars is electrical power and its reliance on batteries. The implementation of energy harvesting devices in charging stations can contribute to the establishment of a pollution-free environment (Kumar et al., 2015). The implementation of wireless energy transfer or energy harvesting for electric vehicles aligns with Nikola Tesla's vision of wireless power transmission and harvesting. Nanogenerators have demonstrated the potential to supply power for in-car sensors and electric vehicle charging infrastructure, owing to recent advancements. Several researchers have attempted to utilize tire rotation to harvest energy for powering various sensors in automobiles. Maurya et al. (2018) developed a flexible organic piezoelectric material capable of harnessing energy from rotating tires, sufficient to illuminate 78 light-emitting diodes (LEDs) simultaneously (Maurya et al., 2018). The output escalates as the velocity amplifies. Mao et al. (2015) created a single-electrode triboelectric nanogenerator capable of immediately powering six conventional green LEDs, with a maximum energy conversion efficiency of 10.4% (Mao et al., 2015). Recently, Bhamre et al. (2020) used a sheet of TENG-based devices on electric car tires to capture the vehicle's lost energy. They produced electrical power of up to 2 W each rotation (Bhamre et al., 2020). This is sufficient to energize many sensors within a wheel. To further examine the capacity of NGs to enhance the energy efficiency of electric cars, the whole energy flow of the electric vehicle is evaluated. Eliminating the losses caused by the internal resistance of the battery, electric drive system, and auxiliaries results in around 70% of the battery energy being sent to the wheels (Woo et al., 2024). In a traditional vehicle, the energy originally used to overcome inertia and accelerate the vehicle is dissipated as heat through friction at the brake site when the brakes are engaged. Nonetheless, electric vehicles can employ regenerative braking methods to recuperate a fraction of this energy. Aerodynamic drag constitutes a significant source of energy dissipation. A vehicle expends energy to displace are as it traverses the road; at reduced speeds, it consumes less energy. As vehicle speed escalates, the energy loss due to aerodynamic drag intensifies. Rolling resistance is a force that opposes motion, arising from the deformation of tires as they rotate on a level surface. It is a significant source of energy loss that may be harnessed by nanogenerators. A 5%–7% reduction in rolling resistance is anticipated to enhance fuel economy by 1%.

6.6 Biosmart actuator

Organism in nature may modulate their osmotic pressure via cell hydration or dehydration in response to external stimuli, facilitating transformation or movement and adapting to dynamic settings. Mimosas may alter their form in response to external stimuli, whereas Venus flytraps ensnare intruding insects by shutting their leaves (Zhang et al., 2023). Motivated by natural creatures, researchers are progressively concentrating on the creation of autonomous biomimetic soft actuators and soft robots (Chen, Valenzuela, et al., 2023). The objective is to enhance motion agility integration, broaden adaptability to unstructured surroundings, and accomplish intricate tasks. Soft robots can navigate intricate surroundings and have benefits,

including material flexibility, human-like interaction, and biological adaptation, which have garnered significant interest from fundamental study to technological applications. Liquid crystal elastomers and hydrogels are optimal materials for soft actuators due to their superior flexibility, reconfiguration, and adaptability (Yang et al., 2022). In contrast to metals or elastomers, hydrogels have been extensively researched for the development of biomimetic soft robots owing to their intrinsic softness, exceptional stretchability, outstanding biocompatibility, favorable permeability, and response to diverse stimuli (Chen, 2023). Smart hydrogels, as significant smart materials, may undergo predictable and reversible shape transformations in response to diverse external stimuli, including temperature, pH, light, chemicals, electricity, and magnetism. In comparison to other polymer actuators, such as liquid crystal elastomers and shape-memory polymers, smart hydrogel actuators are soft and aqueous materials, resembling biological creatures. This characteristic allows them to adapt to varying surroundings and stresses, facilitating flexible movement and operation (Xue et al., 2021). Furthermore, smart hydrogels exhibit significant controllability and programmability. The mechanical properties, deformation behavior, and functional characteristics of hydrogels can be modified by altering their composition, structure, and external stimuli. This offers several opportunities for the design and production of soft robots and soft actuators with varied functionalities and performance levels. Consequently, they have garnered significant interest in soft robotics, biomedicine, and artificial muscles, demonstrating promising application potential (Goudu et al., 2020; Fusi et al., 2023). The design of the majority of hydrogel actuators is predominantly inspired by natural creatures.

Biomass denotes a diverse array of species generated via photosynthesis, encompassing all flora, fauna, and microbes. Biomass materials originate from living beings, including animals, plants and microbes, and are sourced from plentiful resources, including energy crops, agricultural residues, wood and wood by-products, municipal solid waste, aquatic vegetation, and algae (Wang et al., 2024). They are biodegradable, renewable, eco-friendly, and nontoxic, representing a viable alternative to petroleum-derived goods. The majority of natural biopolymers are macromolecules characterized by repetitive structures, including chitosan, chitin, cellulose, and starch (Appiah et al., 2022). Biomass resources have diverse varieties, extensive dispersion, and substantial reserves. Biomass materials have superior biodegradability in comparison to synthetic polymer materials. The bulk of biomass materials is rapidly and entirely decomposed by microbes in the natural environment into water, carbon dioxide, and other minor compounds. In the last 20 years, research has predominantly concentrated on natural hydrogels, which have increasingly supplanted synthetic hydrogels. Biopolymers exhibit significant potential, particularly owing to the abundance of hydrophilic groups, such as hydroxyls, contained within their structure. Biomass-derived hydrogels are composed of natural polymer materials, including starch, cellulose, chitosan, and SA, which undergo specific modifications through various physical and chemical cross-linking techniques, resulting in a characteristic anisotropic structure, along with defined strength, biocompatibility, and biodegradability. Due to the superior strength characteristics and anisotropic structural orientation of natural biomass polymers, biomass-derived

hydrogels exhibit significant strength and intricate actuation behavior. Consequently, the development of functional hydrogels with intelligent actuators utilizing biomass polymers has garnered significant interest in recent years.

Given the escalating depletion of nonrenewable resources such as coal, oil, and natural gas, the comprehensive development and utilization of renewable resources has become an unavoidable trend. Cellulose is a crystalline natural polymer with excellent mechanical qualities, sourced from various materials such as wood, cotton, flax, bamboo, algae, and bacteria (Nechyporchuk et al., 2016). Cellulose NFs, characterized by their nanometer width and many micrometers in length, serve as exceptional nanoscale building blocks. They possess elevated strength, significant stiffness, little thermal expansion coefficient, high crystallinity, hydrophilicity, and readily alterable surfaces (Guan, 2024). Due to these exceptional qualities, cellulose has emerged as an optimal precursor for the synthesis of functional composite hydrogels. CNC are needle-shaped nanomaterials derived from natural cellulose of film, characterized by high strength and unique self-assembly capabilities, making them suitable nanofillers for the preparation of composite hydrogels (Montero de Espinosa et al., 2017). Owing to their favorable hydrophilicity and elongated morphology, they serve as reinforcing agents to enhance the 3D framework of composite hydrogels (Kontturi et al., 2018). CNC, as an eco-friendly biomass material, exhibits nanoparticle characteristics and garners significant interest due to its high aspect ratio and noncovalent intermolecular interactions, rendering it an optimal candidate for the formation of self-assembled 3D nanostructures with distinctive optical properties (Li et al., 2023). Recently, both primitive and modified CNC have been incorporated into diverse polymer matrices. Leray et al. (2022) synthesized CNC-based hydrogels featuring a double network topology (Leray et al., 2022). The first network comprises CNC and xylan (XG). The entropy adsorption of XG and CNC surface enables the second network to use the electrostatic interaction between the cationic chitosan (CHI) and anionic CNC for the effective creation of hydrogels with varying components. Their rheological characteristics, stability, and swelling capacity in acidic and alkaline solutions were assessed. The hydrogels exhibited superior mechanical characteristics and customizable swelling capacity. Subsequently, they utilized the distinct rheological and swelling characteristics of the double-layer water-swelling actuated hydrogel actuator. Three varieties of hydrogels with varying XG/CNC ratios were synthesized. Upon immersion of the hydrogels in water, the actuator exhibited bending and twisting as a result of the differential swelling characteristics of the two layers. CNCs and SA can be oxidized to generate aldehyde functional groups, after which amine-containing vinyl functionalized monomers are incorporated into the CNC or the oxidized SA backbone via a dynamic Schiff base reaction, culminating in the preparation of biomass-based hydrogels through one-pot in situ polymerization. The bilayer actuator is constructed utilizing temperature-sensitive di(ethylene glycol) methyl methacrylate (MEO2MA) and poly(ethylene glycol) methyl methacrylate (OEGMA) as responsive polymer layers, enabling it to bend in water at 55°C.

The activation of biomass-based hydrogels fundamentally depends on the differential internal stress resulting from the irregular expansion or contraction of

the hydrogel. Biomass-derived hydrogels can exhibit nonuniform expansion or contraction by employing diverse architectures, including bilayer, gradient, and anisotropic patterns, enabling deformation in response to external stimuli. The activation of biomass-based hydrogel fundamentally relies on the differential internal stress induced by the irregular expansion or contraction of the hydrogel. The architecture dictates the efficacy. Biomass-derived hydrogels may be engineered into several configurations, including bilayer, gradient, and anisotropic patterns, enabling the hydrogel to undergo nonuniform expansion or contraction in response to external stimuli, so inducing deformation. The bilayer structure consists of two hydrogel sheets with distinct expansion rates or ratios. The bilayer hydrogel actuator may attain regulated deformations, including bending and buckling, due to the asymmetric response properties of its bilayer structure (Hou et al., 2020). Despite substantial research on biomass-based hydrogel actuators with a bilayer structure, their functionality is limited to shape deformation, namely bending. In hydrogels with a gradient structure, the gradient distribution of nanofillers is often established during polymerization to provide diverse "programmed" shape modification behaviors (Yu et al., 2022). Moreover, a gradient distribution of polymer chains exists, and both the gradient distributions of polymer chains and fillers may successfully produce intricate shape deformations. In anisotropic patterned hydrogels, the gradient structure of a designated region may be attained by modulating the cross-linking density within that specific area of the hydrogel network, enabling the hydrogel to undergo deformation into a complex 3D structure (Podstawczyk et al., 2021).

Biomass-derived hydrogels are frequently employed in the fabrication of bionic walking robots, typically achieved by manipulating the asymmetric deformation of the hydrogel elements and the friction of the substrate. A biomimetic walker was built, inspired by the biological behavior of praying mantises in nature, utilizing a 3D-weldable multistimulus response (WMR) actuator and commercial printing paper (Sun et al., 2024). The 3D-WMR actuator was produced by applying a combination of PDMS and iron(II, III) oxide (PIC) onto a SA layer. Utilizing the SA solution, the biomimetic legs, crafted from printing paper, were judiciously developed and affixed to the SA side of the PIC/SA film. Following several cycles of alternating light and humidity stimulation, the walker will persist in its forward movement due to the differential friction between the anterior and posterior limbs and the base. Zhang et al. (2022) created gradient hydrogels exhibiting both photoresponsive (nonthermal) shape-memory and modifiable thermal response actuation capabilities (Zhang et al., 2022). The hydrogel employs an alginate/polyacrylamide substrate, then copolymerizing *N*-isopropylacrylamide and sodium acrylate to form a chemically cross-linked network with linear SA chains. The integration of shape-memory and thermal response properties enables programmable actuation, allowing the hydrogel to be configured into an asymmetric geometric form. Applying hot water to the upper surface of the sample induces a shift in the center of gravity and reversible bending deformation of the two legs with varying curvatures, enabling the hydrogels to traverse laterally, akin to the locomotion of earthworms. The engineered biomimetic walkers can facilitate remote and controlled freight transfer; nonetheless, challenges persist in executing intricate walking or crawling maneuvers. Should the future

hydrogel walker include numerous responsive capabilities to provide programmable structural design and controlled technological methods, it will broaden the scope of its applications.

6.7 Conclusions

Matrix materials, such as polymers and metals, are used as load-bearing components in intelligent materials. Sensing materials detect and measure environmental changes, while actuating materials are used for reaction and control purposes. Smart composites serve as an intermediate stage of connection, enabling the creation and advancement of intelligent materials with diverse characteristics. To create intelligent materials, rigorous standards, scientific procedures, and precise processing are necessary. The integration of software functions with composites is the most appealing technique, combining various software functionalities in diverse structural hierarchies. These composites have similar characteristics to information processing units found in animals, specifically neurons. Various technological challenges must be addressed during the design and production of intelligent composites, including compatibility between sensing/actuation components and the materials they are integrated. The quality of the interface between different materials is essential for effectively transferring substantial shear force. Inadequate interface stress transmission can occur due to chemical incompatibility or elevated temperature produced by heating the actuation element. Precision is crucial in the production of composite materials for sensors and actuators, as they must remain unaffected by mechanical, thermal, or chemical reactions during the manufacturing process. Manual laying and bonding provide sufficient care but are the costliest method. Preliminary research is being conducted to include active fibers, such as those composed of SMA or piezoelectric materials, using automated processes. Automated fiber and tape laying can be considered as an option, but these technologies must be modified to suit the unique characteristics of these novel composite materials. Additional concerns include the link between components, mathematical models, simulations based on biological principles, computation techniques for intelligent systems, the mechanics of systems at both macro and meso scales, the interaction between many parts, phase transition features, and control of hysteresis. Further, an extensive, methodical, and thorough investigation is required to address these challenges.

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