

High-performance, Recyclable Glass Fiber-reinforced Composites from a Camphoric Acid-based Vitrimer with UV-shielding and Damage Self-reporting Capability

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Electronic Supplementary Information

Abstract Glass-fiber-reinforced polymer composites (GFRCs) have a wide range of applications. However, under complex service conditions, GFRCs are prone to internal damage that is not readily detectable, thereby posing potential safety risks. In this study, camphoric acid diglycidyl ester (CADE) was synthesized from bio-based camphoric acid (CPA) and subsequently cured with 4,4'-diaminodiphenyl disulfide (APD) to construct a CADE-APD vitrimer featuring a dual dynamic network based on disulfide and β -hydroxy ester bonds. After compositing with glass fibers, the resulting composite exhibited excellent mechanical performance, recyclability, UV shielding, and damage self-reporting capability. Benefiting from disulfide exchange and transesterification reactions, the vitrimer network demonstrates rapid stress relaxation, with a relaxation time of 50 s and an activation energy (E_a) of 74.66 kJ/mol. Notably, mechanical damage induces disulfide bond cleavage, generating thiyl radicals that result in a visible green color change, enabling real-time damage. The composites could be rapidly degraded in β -mercaptoethanol via thiol-disulfide exchange. Owing to the introduction of a rigid five-membered ring derived from camphoric acid, both the vitrimers and GFRCs exhibit excellent mechanical properties. This study provides a new approach for the preparation of multifunctional bio-based glass-fiber-reinforced polymer materials.

Keywords Glass-fiber-reinforced polymer; Damage self-reporting; UV-shielding; Recyclable; High performance

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INTRODUCTION

Glass-fiber-reinforced polymer composites (GFRCs) are widely used in industrial robotics, wind-energy systems, and sports equipment.^[1–3] However, when used as structural materials, GFRCs are susceptible to internal damage that is not visible to the naked eye, making it challenging to maintain structural reliability under complex service conditions and potentially posing safety risks.^[4] In addition, conventional GFRCs typically rely on thermosetting resin matrices derived from fossil resources. Their permanently cross-linked networks hinder reprocessing and degradation, thereby complicating the glass fiber recovery.^[5,6] Moreover, fossil-based thermosets have raised increasing environmental and health concerns.^[7,8] Therefore, the development of sustainable resin matrices derived from biomass that simultaneously exhibit high mechanical performance, damage self-reporting capability, and degradability is of great importance for

advancing next-generation multifunctional GFRCs.

Vitrimers^[9–11] contain dynamic exchange bonds, such as ester bonds,^[12,13] disulfide bonds,^[14–16] and imine bonds.^[17,18] These dynamic bonds can reversibly undergo bond exchange reactions under external stimuli, such as heat, light, or pH, enabling network rearrangement without compromising the cross-link density. As a result, vitrimers combine the mechanical robustness of thermosets with reprocessability and recyclability, making them highly attractive matrices for sustainable GFRCs. Vitrimers incorporating disulfide bonds can exhibit damage self-reporting behavior because bond cleavage generates thiyl radicals that induce visible color changes. Recent studies have demonstrated the feasibility of using disulfide-based vitrimer systems for damage detection. For example, Gong *et al.* synthesized a vitrimer using aliphatic cyclic diglycidyl esters and 4,4'-dithiodiphenylamine, which exhibited a visible green color upon damage owing to disulfide bond cleavage.^[19] Similarly, Chen *et al.* prepared a series of disulfide-containing vitrimers from different epoxy systems and 4,4'-diaminodiphenyl disulfide, which displayed distinct mechanochromic responses depending on the network structure.^[20] However, these systems are primarily derived

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from fossil-based monomers, which limits their sustainability and increases the environmental burden.

To address these issues, increasing attention has been directed toward bio-based vitrimer systems derived from renewable resources such as vegetable oils.^[21] For instance, Liu *et al.* synthesized a bio-based vitrimer material containing dynamic imine bonds using epoxidized soybean oil, vanillin, and 4-aminophenol as the raw materials. The carbon-fiber-reinforced polymer composite prepared from this vitrimer exhibited a relatively low tensile strength of 145.4 MPa.^[22] Hu *et al.* reported a urethane-based vitrimer derived from castor oil, which yielded composites with a tensile strength of 207.3 MPa.^[23] However, the intrinsic long-chain aliphatic structure of vegetable oils generally leads to low rigidity, resulting in inferior mechanical properties of both vitrimer matrices and their corresponding composites.^[24]

Recent studies have demonstrated that incorporating rigid ring structures into vitrimer networks is an effective strategy for enhancing mechanical performance.^[25–27] Xu *et al.* reported a high- T_g vitrimer incorporating a terpene-derived rigid ring structure, yielding glass fiber composites with a tensile strength of up to 495 MPa.^[28] Hong *et al.* utilized tannic acid to introduce aromatic rigidity into vitrimer networks, achieving carbon fiber composites with tensile strengths as high as 570 MPa.^[29] Similarly, Parisa *et al.* designed imine-based vitrimers containing rigid benzene and triazine rings, resulting in composites with tensile strengths of up to 427 MPa.^[30] These studies collectively demonstrate that the introduction of rigid cyclic structures is crucial for improving the mechanical properties of bio-based vitrimer composites.

Camphoric acid, a derivative of renewable camphor, has a rigid five-membered ring structure and multiple reactive functional groups, making it an attractive bio-based building block for high-performance polymer systems. In this study,

camphoric acid diglycidyl ester (CADE) was synthesized from biomass-derived camphoric acid (CPA) and subsequently cured with 4,4'-diaminodiphenyl disulfide (APD) to construct a CADE-APD vitrimer (Scheme 1a). By incorporating alkali-free glass fiber fabric as reinforcement, a high-performance and recyclable GFRC was fabricated (Scheme 1b). The CADE-APD-GF composites exhibited damage self-reporting capabilities (Scheme 1c). Furthermore, the complete and nondestructive recovery of glass fibers can be achieved via degradation of the composite in β -mercaptoethanol through thiol–disulfide exchange.

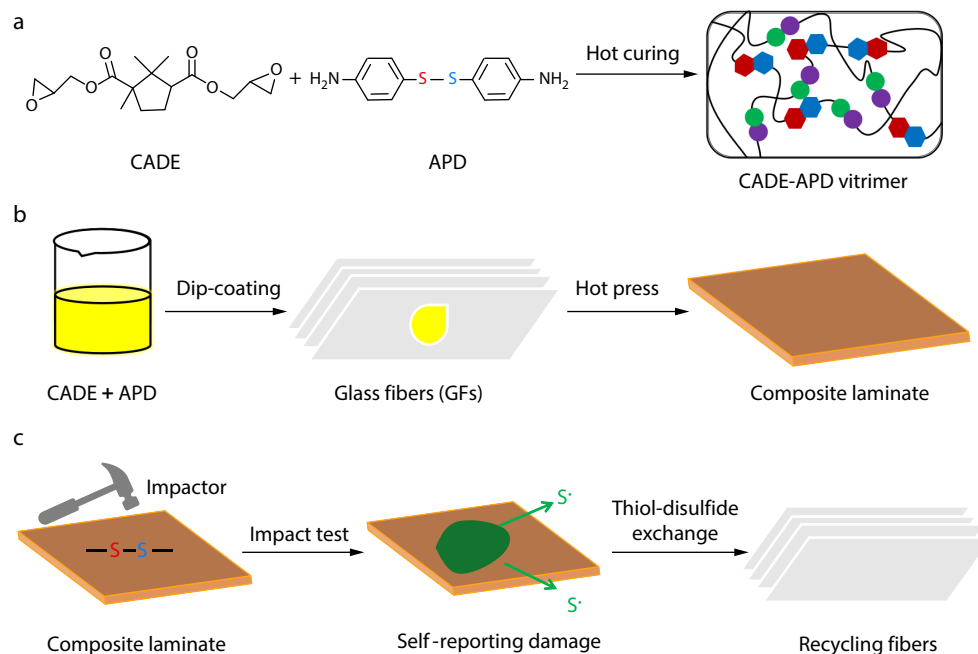
EXPERIMENTAL

Experimental Materials

Camphoric acid (CPA) and epichlorohydrin were purchased from Aladdin Reagent Co., Ltd. (China). 4,4'-Diaminodiphenyl disulfide (APD) was obtained from Macklin Biochemical Co., Ltd. (China). The alkali-free glass fiber cloth (GF) was supplied by Suzhou Dongxuan Plastic Products Co., Ltd. Bisphenol A-type epoxy resin (E51) was purchased from Kunshan Jiulimei Electronic Materials Co., Ltd. All the chemicals were used as received without further purification.

Fabrication of CADE

CPA (10.00 g) and epichlorohydrin (92.50 g) were added to a three-necked flask, followed by the addition of benzyltriethylammonium chloride as a catalyst. The mixture was stirred at 117 °C for 4 h and subsequently cooled to 75 °C, after which NaOH (4.00 g) was added. The reaction was continued for 2 h. The resulting solid-liquid mixture was filtered to collect the supernatant. The supernatant was subsequently concentrated by rotary evaporation and degassed under vacuum to afford a yellow viscous liquid identified as camphoric acid diglycidyl ester (CADE).



Scheme 1 (a) Fabrication of CADE-APD vitrimer; (b) Fabrication of CADE-APD-GF composites; (c) Impact testing of CADE-APD-GF composites and nondestructive recovery of GFs.

Fabrication of the CADE-APD Vitrimer

APD (0.30 g) and CADE (1.00 g) were weighed and dissolved in an appropriate amount of DMF followed by thorough mixing at room temperature. The resulting homogeneous solution was degassed in a vacuum oven and poured into a mold. The sample was subjected to a programmed curing schedule: 100 °C for 1 h, 120 °C for 2 h, 140 °C for 2 h, and 160 °C for 2 h, yielding a CADE-APD_{1.0} network with a stoichiometric ratio (R) of 1.0, where R is defined as the ratio of amino-active hydrogen to epoxy groups. the APD content ($R=0.6$ and 0.8), CADE-APD_{0.6} and CADE-APD_{0.8} networks were prepared following the same procedure. The E51-APD vitrimer was synthesized following the procedure used for the CADE-APD vitrimer.

Fabrication of CADE-APD-GF Composites

APD (0.30 g) and CADE (1.00 g) were then added to the DMF solution. Four layers of glass fiber cloth were then sequentially impregnated. The impregnated glass fiber fabric was placed into a stainless-steel mold (70 mm × 70 mm × 1 mm) and cured at 120,

140, and 160 °C for 3 h under a pressure of 10 tons to yield the CADE-APD-GF composites. The E51-APD-GF composites were fabricated via the same route.

RESULTS AND DISCUSSION

Synthesis and Characterization of the CADE-APD Vitrimer

The synthesis of CADE is as follows. CPA, a small amount of water, and excess epichlorohydrin were introduced into a flask and allowed to react, followed by treatment with NaOH to obtain CADE. The epoxidation mechanism was investigated using ¹H- and ¹³C-NMR spectroscopy. As shown in Fig. 1(a), the ¹H-NMR spectrum of CPA exhibited a characteristic signal at 12.1 ppm, corresponding to the carboxylic proton. After epoxidation, this signal disappears in CADE, while new signals assigned to methylene protons in the epichlorohydrin moiety appear at 3.8–4.5 ppm, confirming successful epoxidation. Fig. 1(b) shows

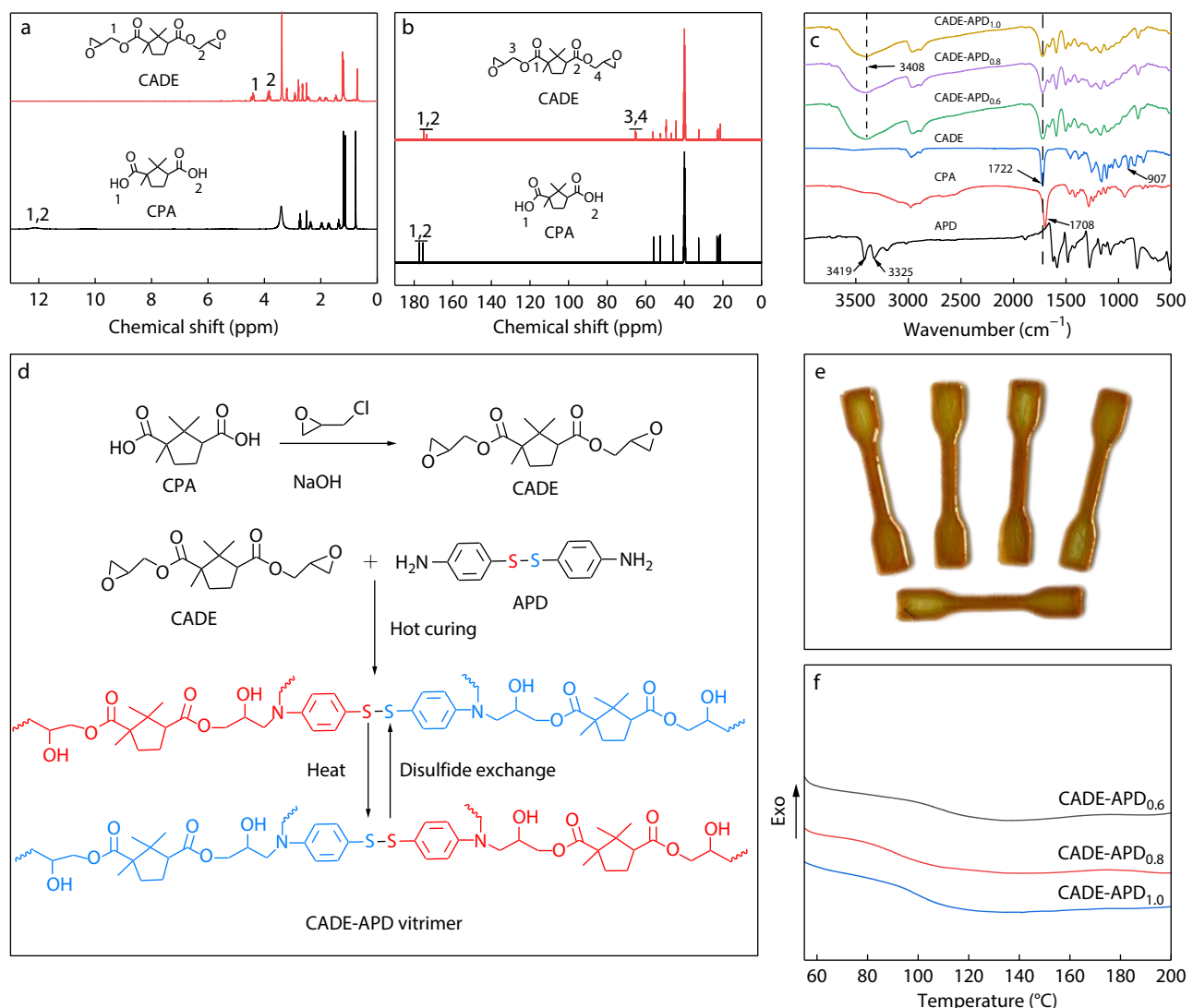


Fig. 1 (a) ¹H-NMR spectra of CPA and CADE; (b) ¹³C-NMR spectra of CPA and CADE; (c) FTIR spectra of APD, CPA, CADE, and CADE-APD; (d) Synthesis routes for CADE and CADE-APD vitrimers; (e) Digital photographs of CADE-APD_{1.0}; (f) DSC curves of CADE-APD at a heating rate of 10 °C/min.

the ^{13}C -NMR spectra of CPA and CADE. In both spectra, the peaks at 175.5–177.6 ppm correspond to the carbonyl carbon atom of the carboxyl group (1, 2), while the ^{13}C -NMR spectrum of CADE shows a characteristic peak at 65.2 ppm corresponding to the methylene carbon atom in the epichlorohydrin structure. ^1H -NMR and ^{13}C -NMR spectra confirmed the successful synthesis of CADE. FTIR spectroscopy was used to verify the formation of the CADE-APD vitrimer (Fig. 1c). APD shows characteristic ($-\text{NH}_2$) stretching vibrations at 3419 and 3325 cm^{-1} , whereas the CADE-APD network exhibits a broad ($-\text{OH}$) absorption at 3408 cm^{-1} , attributed to the formation of β -hydroxy ester linkages. The carbonyl stretching vibration appeared at 1708 cm^{-1} in CPA and was shifted to 1722 cm^{-1} in both CADE and CADE-APD. In addition, the epoxy characteristic band of CADE at 907 cm^{-1} disappears after curing. These results confirmed the successful formation of the CADE-APD vitrimer network.

The synthetic route from CPA to the CADE-APD vitrimer is shown in Fig. 1(d). CPA was first converted to CADE via epoxidation, followed by curing with APD to construct a vitrimer network containing dual dynamic covalent bonds, namely, β -hydroxy ester bonds and disulfide bonds. The curing process was carried out under a programmed temperature schedule of 100 $^\circ\text{C}$ for 1 h, 120 $^\circ\text{C}$ for 2 h, 140 $^\circ\text{C}$ for 2 h, and 160 $^\circ\text{C}$ for 2 h. The dumbbell-shaped specimens of the cured vitrimer are shown in Fig. 1(e). Differential scanning calorimetry (DSC) analysis (Fig. 1f), conducted at a heating rate of 10 $^\circ\text{C}/\text{min}$, revealed no residual exothermic peaks in the range of 60–200 $^\circ\text{C}$, indicating the complete curing of the CADE-APD vitrimer.^[31]

Thermal, Mechanical, and UV Shielding Properties of CADE-APD Vitrimer Networks

Thermogravimetric analysis (TGA) was performed to evaluate the thermal stability of CADE-APD vitrimers. As shown in Fig. 2(a), all the samples with different APD contents exhibited negligible mass losses below 200 $^\circ\text{C}$. At 276.44 $^\circ\text{C}$, the CADE-APD vitrimers ($R=0.6$, 0.8, and 1.0) retained more than 90% of their initial masses, indicating excellent thermal stability. The derivative thermogravimetric (DTG) curve (Fig. 2b) shows the maximum decomposition rate at approximately 300 $^\circ\text{C}$, further confirming the good thermal stability of the vitrimer network. Dynamic mechanical analysis (DMA) was conducted to investigate the thermomechanical properties. As shown in Fig. 2(c), the storage modulus (E') progressively decreased with increasing temperature. At 50 $^\circ\text{C}$, the values of CADE-APD_{0.6}, CADE-APD_{0.8}, and CADE-APD_{1.0} were 1696, 2262, and 2454 MPa, respectively, compared with 4587 MPa for petroleum-based E51-APD_{1.0}. The decrease in E' with temperature is attributed to the enhanced segmental mobility. At low temperatures, the restricted chain motion and the presence of rigid five-membered and aromatic ring structures confer high stiffness. As the temperature increases, the accelerated chain mobility leads to a gradual reduction in E' . Near the glass transition temperature (T_g), E' decreases sharply and the vitrimer transitions into a rubbery state with good elasticity. T_g values were determined from the peaks of the $\tan\delta$ curves (Fig. 2d).^[32] T_g increased with increasing APD content, reaching 132.37, 147.46, and 154.56 $^\circ\text{C}$ for CADE-APD_{0.6}, CADE-APD_{0.8}, and CADE-APD_{1.0}, respectively. This trend could be attributed to two factors. First, a higher APD content introduced more rigid aromatic structures into the network, re-

stricting chain mobility. Second, the increased number of amino-active hydrogens promoted the formation of additional cross-linking sites, resulting in a higher cross-linking density. Although the T_g of CADE-APD_{1.0} is slightly lower than that of E51-APD_{1.0} (180.56 $^\circ\text{C}$), the difference is relatively small, indicating that the bio-based vitrimer maintains comparable thermal rigidity.

The cross-linking density (V_e) of the CADE-APD vitrimer was estimated based on rubber elasticity theory using the following equation:^[33,34]

$$E' = 3V_eRT \quad (1)$$

where E' is the storage modulus in the rubbery plateau region ($T_g + 30$ $^\circ\text{C}$), R is the gas constant (8.314 J/(mol·K)), and T is the absolute temperature. The calculated V_e values are 0.75×10^{-3} , 0.89×10^{-3} , and 1.60×10^{-3} mol/ cm^3 for CADE-APD_{0.6}, CADE-APD_{0.8}, and CADE-APD_{1.0}, respectively, confirming the formation of a highly cross-linked network at higher APD content. With increasing APD loading, the reaction between the active amino hydrogens and epoxy groups becomes more complete, reaching full conversion at $R=1$ (molar ratio of epoxy groups to active amino hydrogens = 1:1). At this stoichiometry, all epoxy groups undergo ring-opening reactions with active hydrogens, leading to an improved modulus and thermal stability of the vitrimer, as well as increased crosslinking sites. As R increases from 0.6 to 1.0, the tensile strength, glass transition temperature (T_g), crosslink density, and storage modulus of the vitrimer increase.

The mechanical properties of the CADE-APD vitrimers are shown in Fig. 2(e). The tensile strengths of CADE-APD_{0.6}, CADE-APD_{0.8}, and CADE-APD_{1.0} are 57.8, 60.0, and 65.3 MPa, respectively, with corresponding elongations at break of 9.7%, 8.2%, and 7.6%. The mechanical performance improves with increasing APD content, reaching an optimum value at $R=1.0$. This enhancement was attributed to the increased number of reactive amino groups, which facilitated a more complete curing and the formation of a dense cross-linked network. In comparison, the petroleum-based E51-APD_{1.0} exhibits a tensile strength of 57.35 MPa and an elongation at break of 13.1%, indicating that the bio-based vitrimer achieves comparable mechanical performance. Toughness refers to the ability of a material to absorb energy prior to fracture, which is equivalent to the area enclosed by the stress-strain curve and strain axis. A larger area indicates a stronger impact resistance and fracture resistance. Accordingly, the areas under the stress-strain curves of CADE-APD_{1.0} and E51-APD_{1.0} were calculated as $S_{\text{CADE}}=2.62$ MJ/ m^3 and $S_{\text{E51}}=3.21$ MJ/ m^3 . A comparison of the tensile strength and toughness of the two samples shows that the initial slope of the curve for CADE-APD_{1.0} is significantly larger than that for E51-APD_{1.0}, indicating a higher crosslink density and greater rigidity of CADE-APD_{1.0} which is the main reason for its higher tensile strength and lower elongation at break.

These results demonstrate that the incorporation of rigid ring structures effectively enhanced the mechanical properties of bio-based vitrimer systems. As shown in Fig. 2(f), the CADE-APD vitrimer exhibited superior mechanical and thermal performance compared to previously reported bio-based vitrimers,^[35–45] along with enhanced UV shielding.

The UV shielding performance of the CADE-APD network was evaluated. As shown in Fig. 2(g), the transmittance is

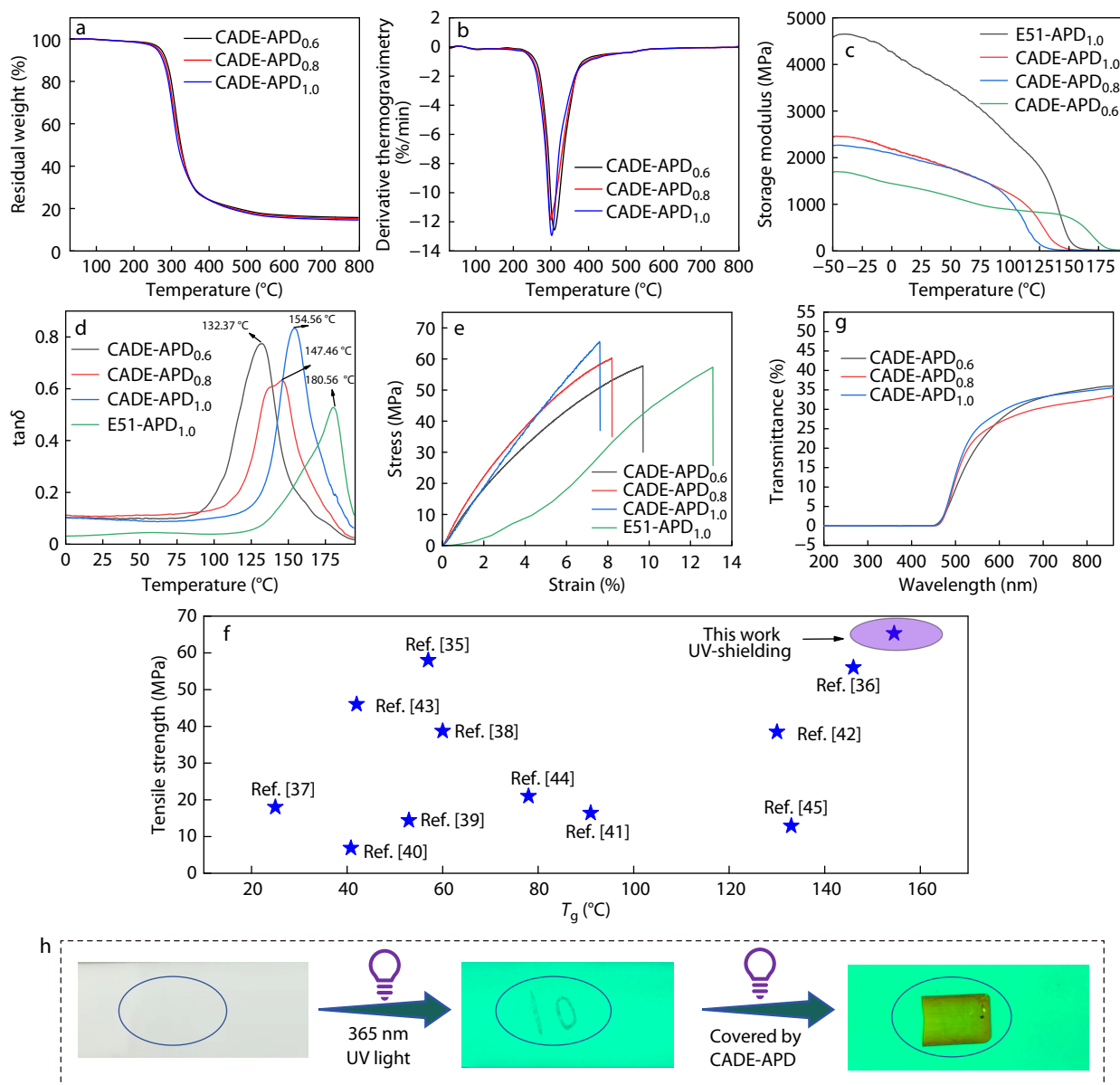


Fig. 2 (a) TGA curves of CADE-APD vitrimer; (b) DTG curves of CADE-APD vitrimer; (c) Curves showing the temperature dependence of the storage modulus for CADE-APD and E51-APD; (d) Curves showing the temperature dependence of $\tan\delta$ for CADE-APD and E51-APD; (e) Stress-strain curves for CADE-APD and E51-APD; (f) Tensile strength and glass transition temperature of reported bio-based vitrimers; (g) Transmission spectra of CADE-APD; (h) Digital photograph of the number “10” covered by a CADE-APD film under 365 nm UV lamp irradiation.

nearly zero below 450 nm, covering the entire UV region (200–400 nm), indicating an excellent UV-shielding capability. This behavior was further verified through a practical demonstration (Fig. 2h). The pattern on a silica plate is clearly visible under irradiation with a 365 nm UV lamp. However, it becomes obscured when covered by a semitransparent CADE-APD film. This result confirmed the excellent UV-shielding performance of the vitrimer network.

Color-changing Behavior of CADE-APD Vitrimers

Based on the distinct mechanochromic behaviors of the CADE-APD vitrimer and CADE-APD-GF composites, the underlying color-change mechanism was systematically investigated. As shown in Fig. 3(a), mechanical damage induced an immediate color change in the CADE-APD vitrimer, which rapidly turned

green when the surface was scratched. After 24 h at room temperature, the green coloration gradually faded and returned to the original yellow state. Similarly, the cross-section of the CADE-APD-GF composites exhibited a green color upon shearing (Fig. 3b). In contrast, no noticeable color change was observed on the fracture surfaces after the tensile or flexural failure.

To elucidate the origin of this behavior, a green powder was collected from the damaged CADE-APD surface and analyzed by electron paramagnetic resonance (EPR), Raman spectroscopy, and UV-Vis spectroscopy. The EPR spectrum (Fig. 3c) displays three anisotropic signals at $g_1=2.005$, $g_2=2.043$, and $g_3=2.022$, which are assigned to sulfur-centered radicals generated by disulfide bond cleavage.^[46] Ra-

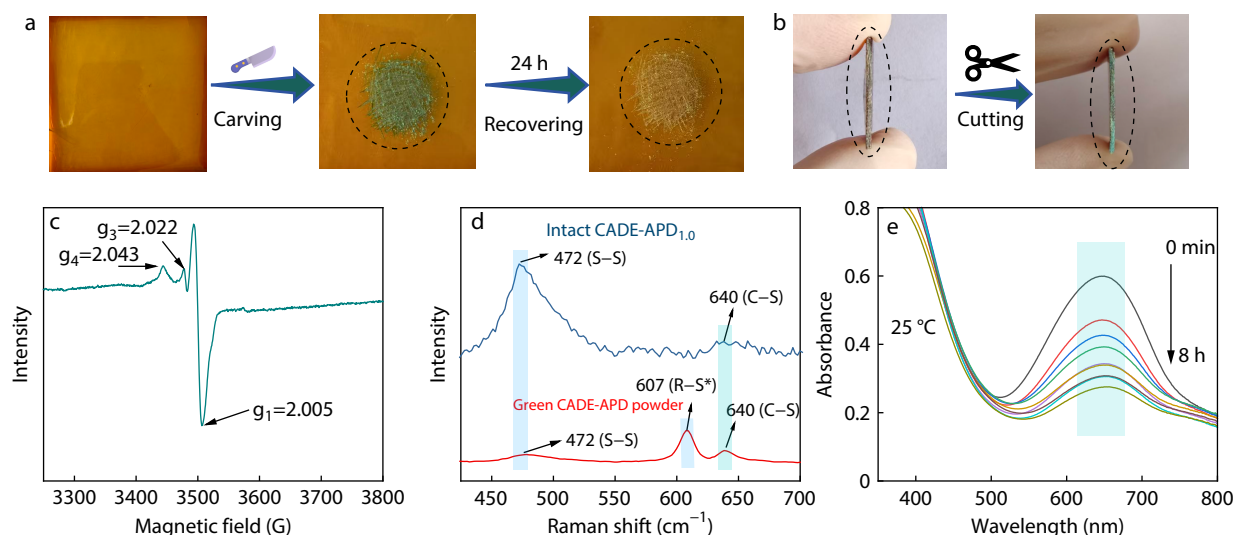


Fig. 3 (a) Color change behavior of the CADE-APD vitrimer upon scratching; (b) Color change behavior of the CADE-APD-GF upon shearing; (c) EPR spectrum of the green powder; (d) Raman spectra of the green powders of CADE-APD and solid of CADE-APD; (e) UV-Vis absorption spectrum of the green powder of CADE-APD.

man spectra (Fig. 3d) further supported this mechanism. Compared to the characteristic S—S and C—S stretching bands at 472 and 640 cm⁻¹ in the intact network, the green powder exhibited an additional peak at 607 cm⁻¹, which was attributed to thiyl radicals.^[47] Under ambient conditions, these radicals gradually recombine to form disulfide bonds, leading to the disappearance of the green color. The temporal evolution of the radicals was further examined by UV-Vis spectroscopy (Fig. 3e). The green powder exhibited absorption in the range of 500–800 nm, which progressively decreased over time (from 0 min to 8 h), indicating a gradual radical decay. This result confirms that color fading is associated with the recombination of sulfur-centered radicals. Overall, the mechanochromic self-reporting capability of the CADE-APD-GF composites originates from the generation and subsequent decay of radicals formed *via* disulfide bond cleavage, enabling real-time and reversible damage.

Exchange and Degradation of the CADE-APD Vitrimer Network

The CADE-APD vitrimer is a macromolecular network containing rigid benzene rings linked *via* disulfide bonds that enable dynamic disulfide exchange reactions. Additionally, it incorporates β -hydroxy esters and tertiary amines, which facilitates dynamic ester exchange. Based on these structural features, a systematic investigation of the dynamic bond exchange and degradation behavior of the vitrimers was conducted. The stress relaxation time (τ) is defined as the time required for the stress to decay from its initial value to $1/e$ (about 0.37) under a constant strain, reflecting the rate of dynamic covalent bond exchange within the vitrimer network. Fig. 4(a) presents the stress-relaxation behavior of CADE-APD_{1.0} at various temperatures, measured *via* dynamic thermomechanical analysis. At 120, 140, 160, and 180 °C, the τ values are 760, 286, 55, and 50 s, respectively. The rapid decrease in relaxation time can be attributed to several factors. The tertiary amine and adjacent hydroxyl groups form a five-membered ring intermediate *via* intramolecular hydrogen bonding, which enhances the nucleophilicity of the hy-

droxyl group and promotes both ester and disulfide exchange. As the temperature increased, the vitrimer network transitioned to a highly elastic state, enabling rapid polymer segment mobility, thereby accelerating dual dynamic bond exchange. In addition, the dual dynamic covalent network exhibited faster exchange kinetics than the individual dynamic bonds at elevated temperatures.

Analysis of the stress-relaxation data indicated that the CADE-APD_{1.0} vitrimers follows Maxwellian behavior. The activation energy (E_a) of bond exchange was determined using the Arrhenius equation:^[48]

$$\ln \tau = \frac{E_a}{RT} - \ln A \quad (2)$$

where R is the gas constant, τ is the relaxation time, T is the absolute temperature, and A is a pre-exponential factor. Fig. 4(b) shows the linear Arrhenius fit of $\ln \tau$ versus $1000/T$. From the slope, E_a was calculated to be 74.66 kJ/mol, indicating that CADE-APD_{1.0} can undergo rapid self-healing, reshaping, and recovery while maintaining structural stability.

Creep-recovery experiments provide insight into the dimensional stability and reprocessability of the network, which are key aspects of its dynamic reversibility. As shown in Fig. 4(c), under a constant stress of 0.2 MPa at 160 °C, the CADE-APD_{1.0} network exhibited an initial elastic response, followed by rapid molecular chain mobility driven by ester and disulfide exchange. The strain increased to 12%, and after 60 min, it decreased slightly and stabilized at 8%, reflecting partial irreversible deformation due to chain slippage and untangling. The degradability of the vitrimers was evaluated in β -mercaptoethanol, which cleaves disulfide bonds *via* thiol-disulfide exchange. Fig. 4(d) shows the residual mass of CADE-APD_{1.0} during degradation, decreasing from 100% to 0% within 5 h, indicating complete dissolution.

The proposed degradation mechanism is shown in Fig. 4(e). The CADE-APD vitrimer prepared in this study uses disulfide bonds as the core interchain crosslinking chemical bonds, which bridge and support polymer chains to con-

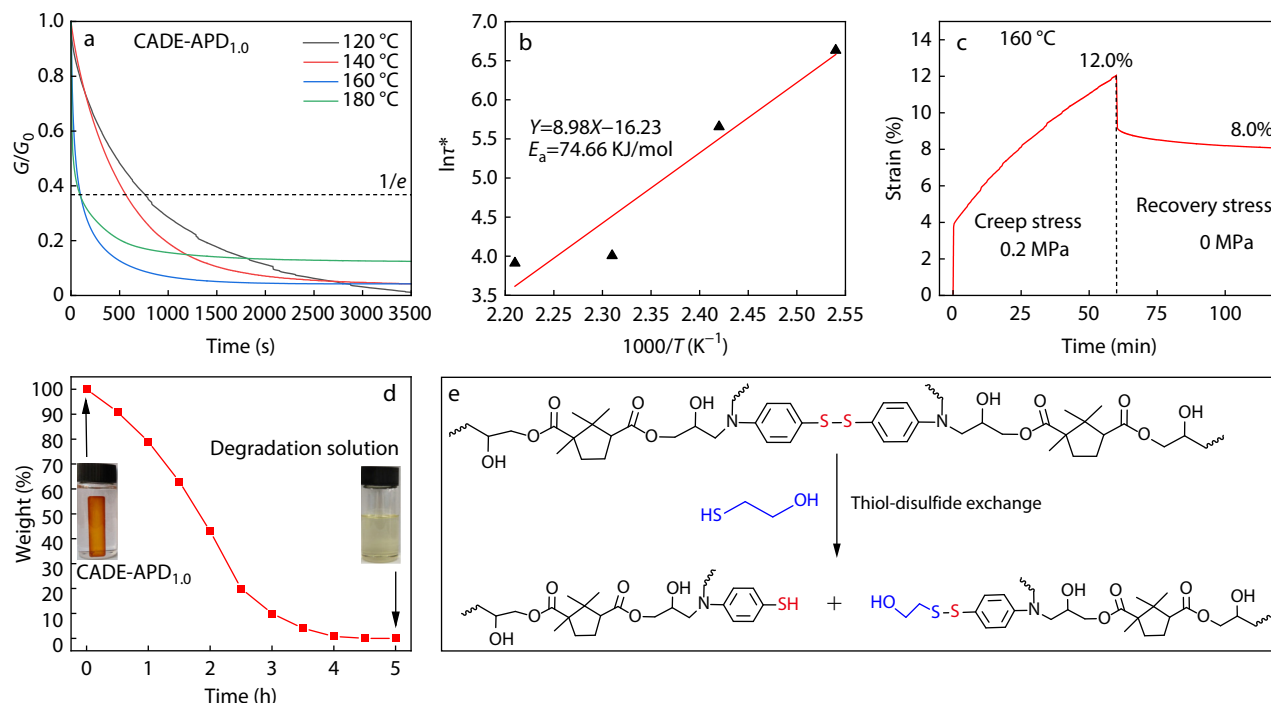


Fig. 4 (a) Stress-relaxation curves of CADE-APD_{1.0} at different temperatures; (b) Relationship between relaxation time and temperature for CADE-APD_{1.0}; (c) Creep recovery behavior of CADE-APD_{1.0} at 160 °C; (d) Relative weight change of CADE-APD_{1.0} during degradation; (e) Degradation mechanism of CADE-APD_{1.0} in a β-mercaptoethanol solution.

struct a complete three-dimensional crosslinked network, whereas β-hydroxy ester bonds mainly exist in the polymer backbone. Therefore, this vitrimer can be effectively degraded by mercaptoethanol via thiol-disulfide exchange reactions. In the weakly alkaline environment of the material system, the thiol groups of mercaptoethanol are activated into thiolate anions, which trigger the cleavage and recombination of interchain disulfide bonds through nucleophilic attack. Under the continuous action of excess mercaptoethanol, many disulfide crosslinking sites in the network are broken down, and the three-dimensional covalent crosslinked network originally supported by disulfide bonds gradually decrosslinks, ultimately achieving dissolution and degradation of the vitrimer.

Preparation and Structural Properties of CADE-APD-GF Composites

Owing to its excellent mechanical properties, CADE-APD_{1.0} ($R=1.0$) was selected as the resin matrix, and glass fiber fabric (GF) was used as the reinforcing phase. CADE-APD-GF composites were fabricated by wetting the glass fiber cloth with a liquid resin matrix, followed by hot pressing. In the resulting laminate, the four layers of glass fiber cloth accounted for approximately 60% of the total composite mass. A representative composite sample is shown in Fig. 5(a). The surface morphologies of the composites were examined by scanning electron microscopy (SEM). As shown in Fig. 5(b), the CADE-APD resin matrix tightly coated the glass fibers during hot pressing, forming a strong interfacial bond. The resin fully enveloped the fiber surfaces, causing adjacent filaments to adhere and establish a dense fiber-matrix interface. The internal structure of a 20 mm × 20 mm × 1 mm specimen was further investigated using mi-

cro-computed tomography (micro-CT). The 3D reconstruction revealed the overall architecture of the glass fiber fabric (Fig. 5c). Cross-sectional images in the XY, XZ, and YZ planes (Figs. 5d–5f) show well-aligned fibers with no evidence of delamination or voids, confirming a dense internal structure and excellent interfacial adhesion. The robust fiber-matrix interface translates into superior mechanical performance. Tensile and flexural tests (Figs. 5g and 5h) indicate that the CADE-APD-GF composites exhibit a tensile strength of 496.02 MPa with 8.05% elongation at break and a flexural strength of 370.07 MPa with 10.14% elongation at break. The E51-APD-GF composite shows a tensile strength of 423.97 MPa, an elongation at break of 11.81%, a flexural strength of 357.67 MPa, and a flexural elongation at break of 9.30%. These results demonstrate that the camphoric acid-based glass fiber composite exhibits superior mechanical properties compared to its petroleum-based counterpart with the same curing agent APD employed.

Impact Test of CADE-APD-GF Composite Materials

To investigate the self-reporting capability of damage in the CADE-APD-GF composite, a customized hammering test was conducted. Composite specimens were placed on a table and struck with a 0.6 kg hammer dropped from a height of 0.1 m, corresponding to an impact energy of approximately 0.588 J per strike. The surface color and damage were documented after each impact, and representative images were captured every 10 strikes (Fig. 6a). As the number of strikes increased, the impacted area changed from black to green and the color intensified as the cumulative impact energy increased. Surface damage and visible cracks were observed alongside this progressive color change (Fig. 6b). SEM analysis was performed before and after hammering. Fig. 6(c) shows the pristine composite, with a

distinct resin matrix tightly coating the glass fibers and forming a strong interface. After 110 hammer cycles, Fig. 6(d) reveals fiber breakage and matrix damage, demonstrating that color changes occurred prior to material failure. Notably, the CADE-APD-GF composites exhibited a pronounced color change under impact, whereas the pure resin did not change color upon tensile fracture, and only minor discoloration occurred at stress points during bending. This behavior arises because disulfide

bond cleavage requires specific stresses, such as shear, impact, or scratching, to generate sufficient thioxy radicals, which form a green powder and provide visible damage self-reporting.

Degradation and Recycling of CADE-APD-GF Composites

β -Mercaptoethanol contains both hydrophilic hydroxyl ($-\text{OH}$) and reducing thiol ($-\text{SH}$) groups, enabling dynamic bond-ex-

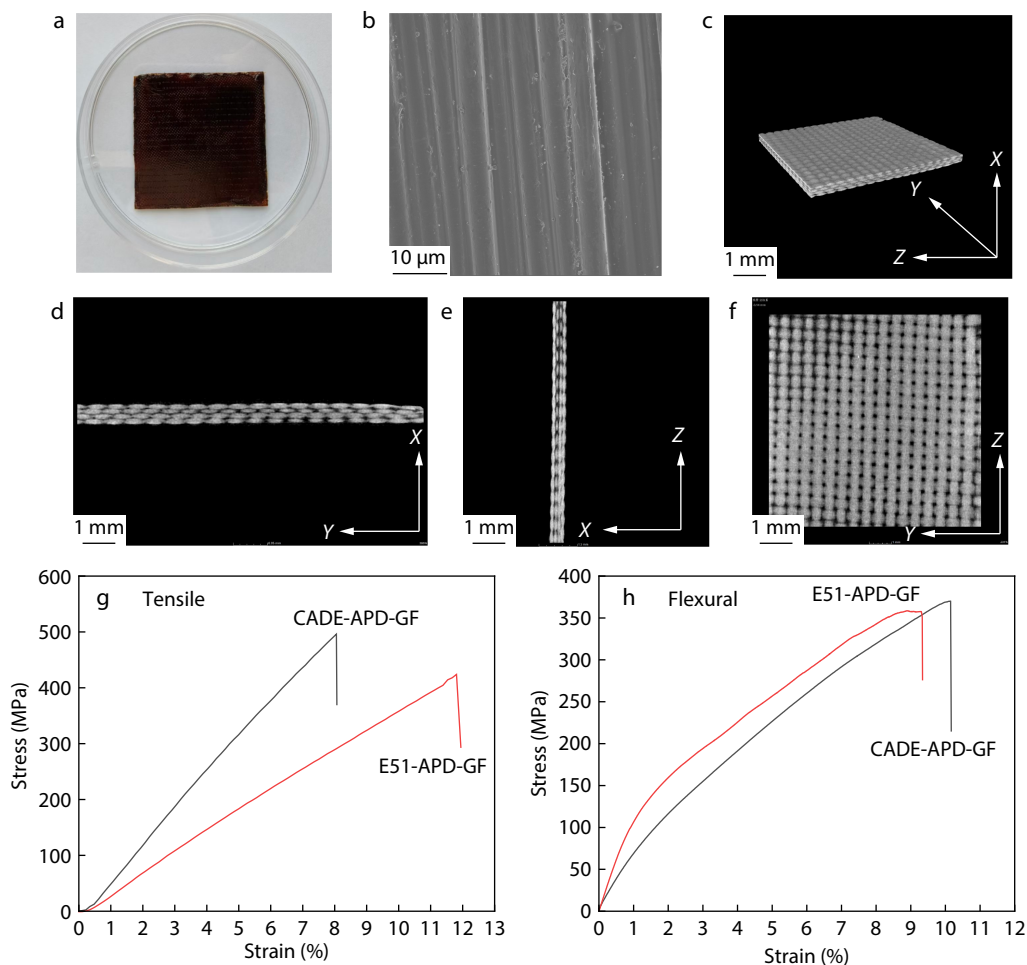
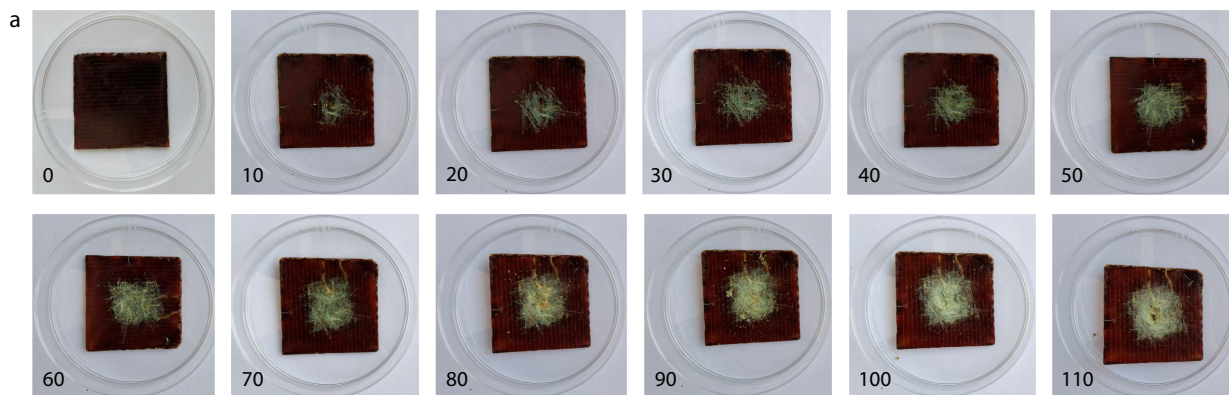


Fig. 5 (a) Image of a square composite sample; (b) SEM image of the CADE-APD-GF composite; (c) 3D microstructure of the CADE-APD-GF composite obtained by micro-CT; (d–f) Microstructural cross-sectional images in the XY, YZ, and XZ directions, respectively; (g) Tensile strength curves of CADE-APD-GF and E51-APD-GF composites; (h) Flexural strength curves of CADE-APD-GF and E51-APD-GF composites.



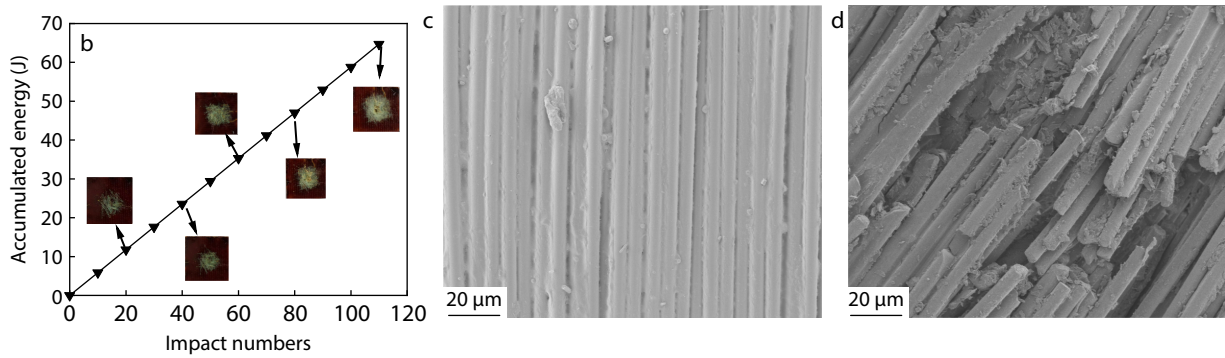


Fig. 6 (a) Cyclic hammering of the CADE-APD-GF composites, the numbers indicate the number of hammering cycles; (b) Energy accumulation in the CADE-APD-GF composites after hammering; (c) SEM image of the CADE-APD-GF composites before hammering; (d) SEM image of the CADE-APD-GF composites after 110 hammering cycles.

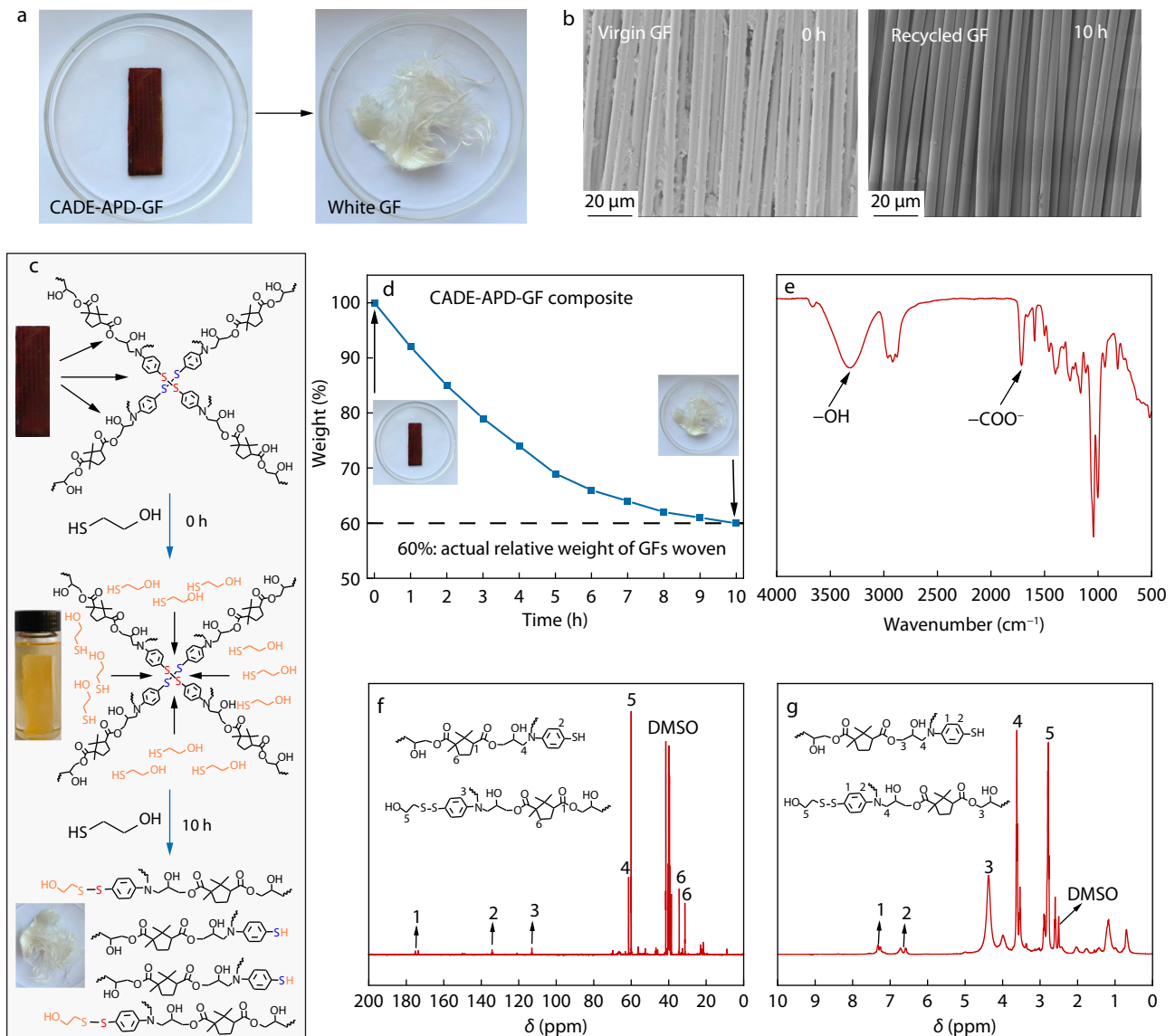


Fig. 7 (a) Photographs of the CADE-APD-GF composite before and after degradation; (b) SEM images of surface fibers in the CADE-APD-GF composite before and after degradation; (c) Schematic diagram of the degradation mechanism of the CADE-APD-GF composite; (d) Relative weight change of the CADE-APD-GF composite during degradation; (e) FTIR spectrum of the degradation products of CADE-APD-GF; (f) ¹³C-NMR spectrum of the degradation products of CADE-APD-GF; (g) ¹H-NMR spectrum of the degradation products of CADE-APD-GF.

change reactions with disulfide bonds. Therefore, it was selected as the degradant for the CADE-APD-GF composites. Fig. 7(a) shows the composites before and after degradation. Upon treatment with β -mercaptoethanol, the resin matrix was completely removed from the glass fiber fabric. The composite gradually lightened over time, and after 10 h, the CADE-APD matrix was entirely removed, restoring the white glass fiber fabric and enabling the full recovery of the fibers. SEM images of the composite surface before and after degradation (Fig. 7b) confirmed this process. Prior to degradation, a strong interface existed between the CADE-APD matrix and the fibers. After 10 h, the resin was fully removed, leaving smooth, clean fiber surfaces, further validating complete matrix removal.

The degradation mechanism is illustrated in Fig. 7(c). Owing to the strong interfacial bonding, heating and stirring facilitated the penetration of β -mercaptoethanol molecules into the composite, where thiol-disulfide exchange and ester exchange reactions occurred. Fig. 7(d) schematically depicts the process, intact CADE-APD macromolecules, initiation of degradation by mercaptan molecules, and the formation of low-molecular-weight products. Complete disulfide bond cleavage yielded clean, recoverable fibers, allowing 100% recycling of the glass fiber fabric without damage. Quantitative degradation was tracked by measuring the residual composite mass. The mass fraction of the CADE-APD-GF composites (2.36 g) decreased to 85% (2.01 g) after 2 h, 69% (1.63 g) after 5 h, and 60% (1.42 g) after 10 h, which is consistent with the mass fraction of the four layers of GF (60%).

FTIR, $^1\text{H-NMR}$, and $^{13}\text{C-NMR}$ analyses of the degradation solution confirmed complete removal of the resin matrix (Figs. 7e–7g). In the FTIR spectrum, the bands at 3320 and 1714 cm^{-1} correspond to terminal hydroxyl and ester groups, respectively. $^{13}\text{C-NMR}$ signals at 175 and 171 ppm corresponded to ester carbons, 134 ppm to carbon adjacent to thiol, 113 ppm to aromatic carbons, 61 and 60 ppm to methylene carbons linked to nitrogen and disulfide, and 34 and 31 ppm to methylene carbons in the five-membered ring. $^1\text{H-NMR}$ signals at 7.3 ppm (adjacent to thiol), 6.6–6.7 ppm (aromatic), 4.3 ppm (methylene near ester), 3.6 ppm (methylene near nitrogen), and 2.8 ppm (methylene near terminal hydroxyl) confirm the degradation product structure. These analyses verified that the CADE-APD matrix could be fully removed from the fibers, and that the CADE-APD-GF composite could be rapidly degraded in β -mercaptoethanol solution, enabling the complete, damage-free recovery of the glass fiber fabric.

CONCLUSIONS

In summary, CADE, synthesized from biomass-derived camphoric acid (CPA), was crosslinked with APD to form a high- T_g high-performance dual-dynamic covalent network CADE-APD vitrimer. The incorporation of a rigid ring structure into CPA endows the vitrimer with excellent mechanical and thermal properties, including a tensile strength of 65.32 MPa, glass transition temperature (T_g) of 154.56 $^{\circ}\text{C}$, and 10% weight loss temperature ($T_{10\%}$) of 279.41 $^{\circ}\text{C}$. The vitrimer also provides effective UV shielding across the 200–450 nm range. The CADE-APD vitrimer underwent rapid stress relaxation and topological rearrangement via disulfide and ester exchange reactions, with a relaxation time of 50 s at 180 $^{\circ}\text{C}$ and an activation energy (E_a) of 74.66

kJ/mol. Leveraging its robust mechanical performance and damage self-reporting capability, composites prepared from this matrix with glass fibers (CADE-APD-GF) exhibited outstanding properties, including a tensile strength of 496.02 MPa and a flexural strength of 370.07 MPa. The composites can self-report damage; impacted areas turn green, allowing accurate assessment of damage extent and serving as a visual indicator of material failure; furthermore, the CADE-APD-GF composites can be fully recycled. Immersion in β -mercaptoethanol enabled complete degradation of the resin within 10 h while recovering intact glass fibers. This study demonstrates a novel approach for the fabrication of high-performance, UV-shielding, self-damage-reporting, and fully recyclable glass fiber-reinforced composites from biomass-derived camphoric acid.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://doi.org/10.1007/s10118-026-3858-4>.

Data Availability Statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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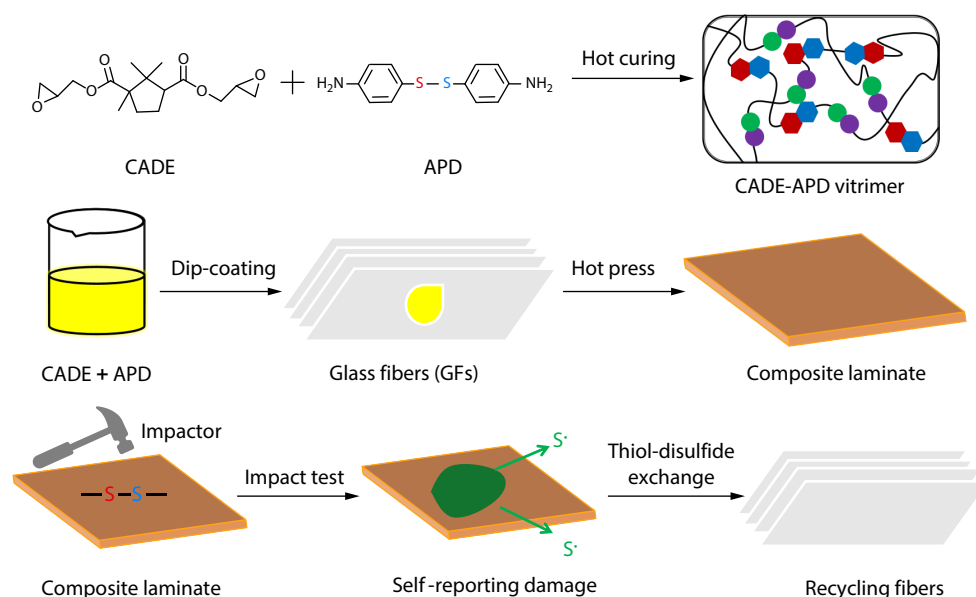
Graphical Abstract

High-performance, Recyclable Glass Fiber-reinforced Composites from a Camphoric Acid-based Vitrimer with UV-shielding and Damage Self-reporting Capability

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Camphoric acid diglycidyl ester (CADE) derived from camphoric acid was used to fabricate a CADE-4,4'-diaminodiphenyl disulfide (APD) vitrimer with UV-shielding property and dual dynamic covalent bonds. The glass fiber-reinforced composites exhibit damage self-reporting and recyclable performance.



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