

Biobased Polyimine Vitrimers: Promising Materials for Fluorescence Quenching, Anticounterfeiting, and Plastic Degradation

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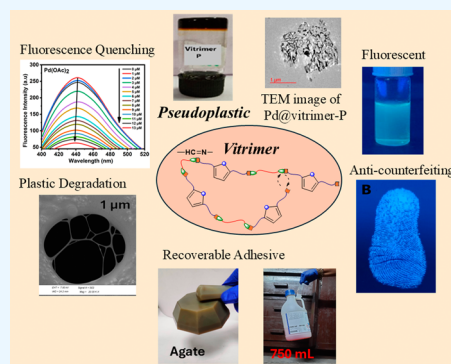


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ABSTRACT: Using glucose, one of the readily available biomasses provides an industrially relevant derivative 5-hydroxymethyl furfural (5-HMF) containing two versatile functionalities. 2,5-Furandicarbaldehyde (DFF) has been synthesized in high yields via chemo-selective oxidation of 5-HMF. Biobased thermoset vitrimers were prepared by the condensation of DFF with various amines such as polyethylenimine, Bis[4-(4-aminophenoxy)-phenyl] propane (BAPP), 1,4-diaminobutane, and (L)-Lysine. This paper reports the design, synthesis, and characterization of fluorescent biobased vitrimers and poly imines for sensing of various aromatic monoamines, diamines, and also Pd metal. It was further demonstrated that biobased polyimine vitrimers are apt as anticounterfeiting material, for plastic degradation, and for synthesis of nanoparticles in an effective way. The fluorescent material offered good thermal stability and obeyed the property of pseudoplasticity having Non-Newtonian fluids.



1. INTRODUCTION

Thermoset polymers are widely used as an irreplaceable material to produce plastic components having high thermal and chemical resistance.^{1–3} A family of thermoset materials are known for the last couple of years in various applications including in reprocessing.⁴ These materials possess high chemical stability, retain their structural integrity, and behave like vitreous silica even at elevated temperatures.⁵ Thermoplastics are made up of linear chains, which are not easily degradable and show a highly resistant property toward oxidation.⁶ A class of new materials that acts as a bridge between thermosets and thermoplastics are represented as vitrimers.⁷ Extensive development of research on vitrimers has been carried out to find an alternative sustainable material to replace fossil-based ones.⁸ In addition, they behave like thermosets, having the properties of mechanical durability and thermoplastic-like malleability/processability at the same time. One important and intrinsic characteristic of vitrimers is their persistent network with dynamic covalent bonds, which are responsible for their chemical and physical properties.^{9,10} Reactants such as epoxy monomers, bisphenol monomers, diols, and disulfides are widely used for the synthesis of vitrimers.^{11–14} In addition, potential dynamic vitrimer reactions also include transesterification,¹⁵ transamination,¹⁶ imine exchange reaction,¹⁷ and disulfide exchange reaction.¹⁸ Recently, imine based vitrimers are also used in self-healing, as adhesives, and as composite materials.¹⁹ The development of biobased materials with reversible imine groups has been gaining attention due to additional potential reduction in environmental impact.²⁰ Biobased formyl furan derivatives with butylamine were reported and they observed that imine

exchange reactions could take place in the presence of excess of amine.²¹ Further Liu and co-workers also reported vanillin-based bio vitrimers with dynamic imine bonds have been used for good solvent resistance and superior mechanical properties.²² Later, vanillin-based bio vitrimers were prepared to get materials with high mechanical strength for good recycling and self-healing abilities.²³ Additionally, other biobased vitrimers had been reported from epoxidized soybean oil and citric acid for retention of tensile strength and programmability.²⁴ Dynamic amine-imine exchange reaction of flexible network vitrimers has been used for UV-shielding performance.²⁵

Herein we report the biobased polyimine vitrimers using 2,5-furandicarbaldehyde (DFF) and various amines polyethylenimine (PEI, branched), 1,4-diaminobutane, (L)-Lysine, and 2,2-Bis[4-(4-aminophenoxy)phenyl]propane (BAPP) (Scheme 1). The monomer DFF was obtained from oxidation of 5-hydroxymethyl furfural (HMF) using our reported protocol.

2. RESULTS AND DISCUSSION

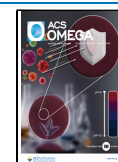
The polyimine vitrimers were studied by various characterization techniques such as FT-IR, UV-visible, NMR, and TGA (Scheme 1).

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Scheme 1. Synthesis of Bio-Based Vitrimers/Polyimines from Glucose-Derived DFF

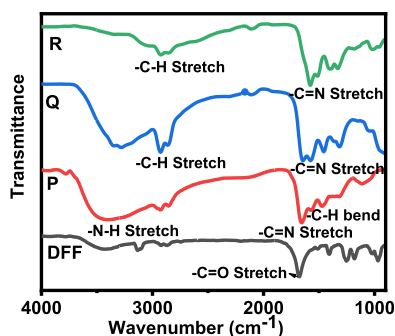
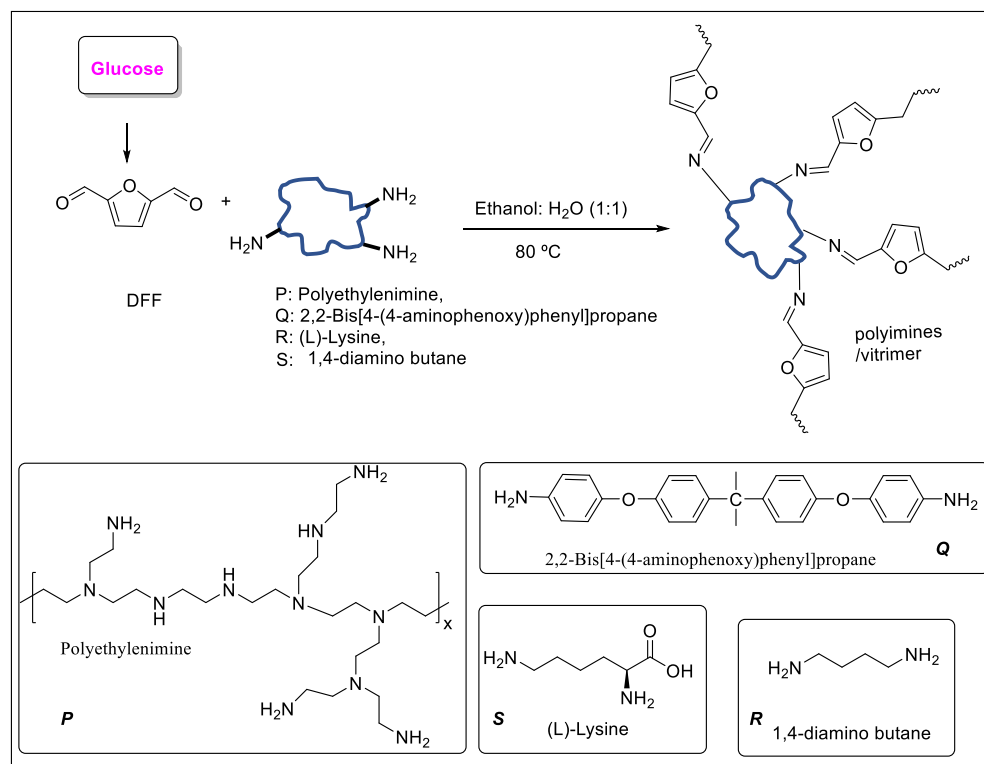


Figure 1. FT-IR spectra of DFF and polyimine vitrimers P, Q, R.

In our experiment, DFF was mixed with various amines separately to create a set of cross-linked networks (in a stoichiometric ratio of aldehyde and amine). The reaction was performed in ethanol/water at 80 °C and the formation of the vitrimer was confirmed from FT-IR analysis. The disappear-

ance of the --HC=O band at 1674 cm^{-1} and the appearance of the band related to the --HC=N band at 1657 cm^{-1} is an indication of the vitrimer. Additional peaks related to a long-chain --C--H rock vibration present in polyethylenimine appeared at 628 cm^{-1} and a broad peak at 3384 cm^{-1} corresponding to the --N--H band of imine. Similarly, formation of vitrimers using various other amines was confirmed through FT-IR analysis (Figure 1).^{26,27}

2.1. UV–Visible Absorption. Initially polyimine vitrimer material “P” is collected as a sticky solid, and it was highly soluble in both THF and in ethanol. The bulky polyimine from “Q” was insoluble in most of the organic solvents and also in 1.0 M HCl and 1.0 M NaOH solutions. However, polyimine vitrimer materials obtained from R, S are highly soluble in ethanol at room temperature.²⁸

The UV–visible spectra of polyimine vitrimer P showed a peak at 368 nm (in ethanol) corresponding to $\pi \rightarrow \pi^*$ transitions. For DFF, the $\pi \rightarrow \pi^*$ transition appears at 280 nm whereas for polyethylenimine ($n \rightarrow \pi^*$), transition appears at

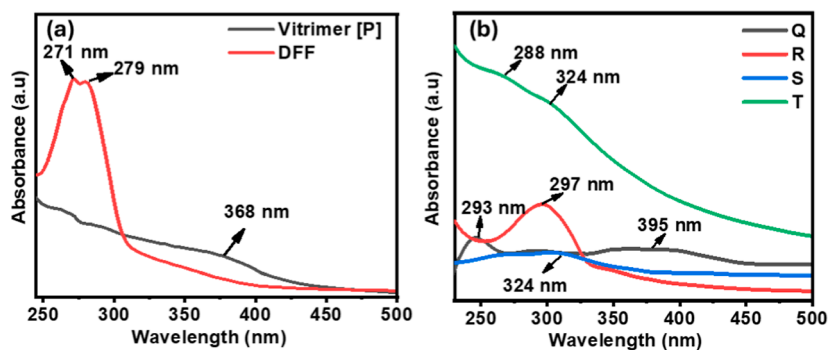


Figure 2. UV-spectra of DFF and polyimine vitrimer P (left side). UV-Spectra of polyimine Q, R, S, T (right side).

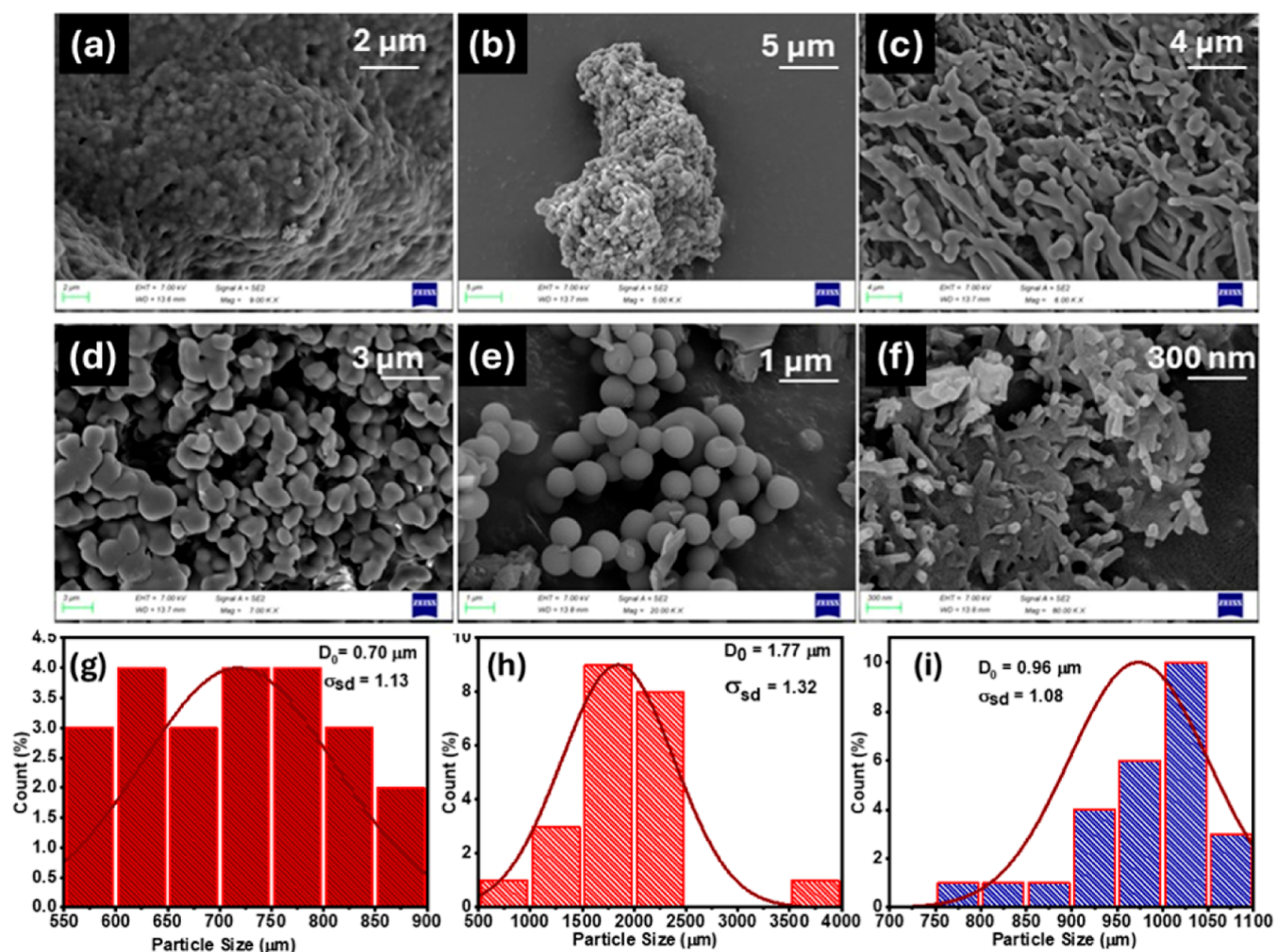
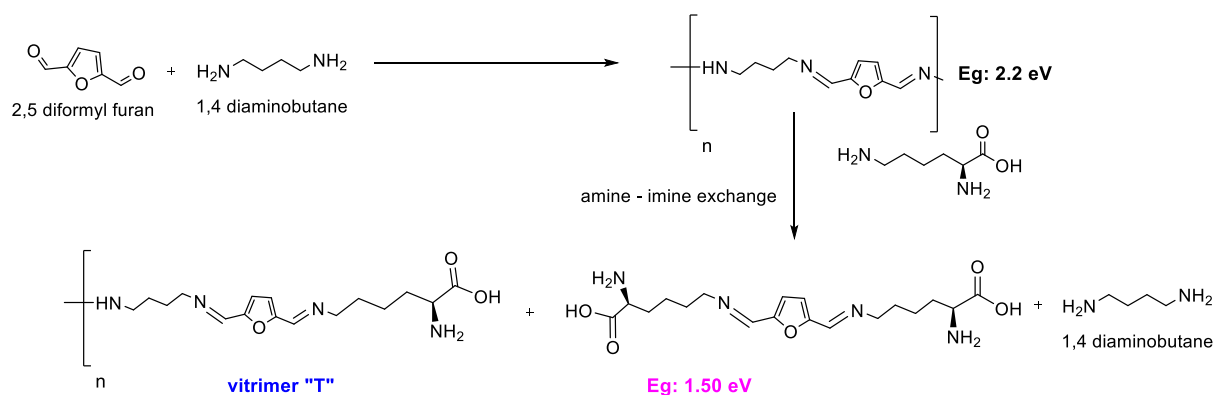


Figure 3. FE-SEM image of polyimine vitrimers of (a) P (2 μm); (b) P (5 μm); (c) Q (4 μm); (d) Q (3 μm); (e) S (1 μm); (f) S (300 nm). (g) Average particle size of polyimine vitrimer P. (h) Average particle size of polyimine vitrimer Q. (i) Average particle size of polyimine vitrimer S.

Scheme 2. Polyimine Vitrimer Exchange Reaction (Imine–Amine Exchange)



206 nm. These data indicate that there is a shifting of $\pi \rightarrow \pi^*$ transitions due to the formation of the imine bond (Figure 2). Polyimine vitrimer material "Q" showed a peak at 293 nm and a shoulder at 395 nm in an ethanolic solution. For vitrimer "P", the absorbance of DFF decreased, and at the same time, an imine peak appeared at 368 nm. However, in "S", imine transitions were observed at 324 nm.^{29,30} In vitrimer "T", imine transitions observed at 288 and 324 nm clearly indicate that an amine–imine exchange reaction took place.

2.2. Characterization of Vitrimers. **2.2.1. FE-SEM Analysis.** It was observed from FE-SEM (Figure 3) of polyimine vitrimer P that at 2 μm (Figure 3a), the small spherical particles could be seen in the layer. In the image of Figure 3b, the perfectly spherical particles are intact through each other at 5 μm . However, for polyimine vitrimer Q, at 4 μm (Figure 3c), the rod-type structure and also spherical particles could be seen in the image (Figure 3d). However, chiral imine from "S" showed a perfect spherical particle appeared at 1.0 μm

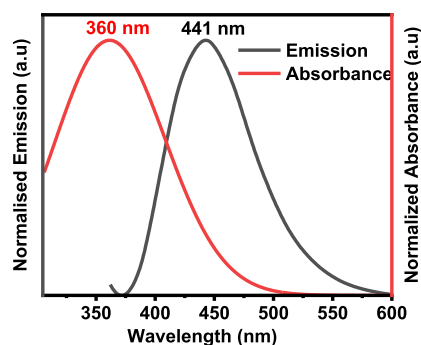


Figure 4. Excitation and emission spectra of the polyimine vitrimer.

(Figure 3e), whereas in Figure 3f, at 300 nm, it appeared as a rod structure with a hollow sphere. The average particle size calculation can be obtained from the FE-SEM image where graph Figure 3g is for vitrimer P, Figure 3h for vitrimer Q, Figure 3i for vitrimer S^{31–33} (Figure 3g–i).

2.3. Imine–Amine Exchange Reaction. Imine–amine exchange reactions are well-known due to their dynamic covalent nature. This reaction predominates and a reversible process take place in the presence of excess of amine.³⁴ Initially, the polyimine vitrimer “R” of DFF and 1,4-diaminobutane was collected as a powder. To the ethanolic solution of R, (L)-Lysine solution was added slowly for 30.0 min (Scheme 2). In the UV spectra, two absorption peaks appeared at 288 and 324 nm, indicating that (L)-Lysine slowly replaced 1,4-diaminobutane (Figure 2, Scheme 2). Similar absorption bands corresponding to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ of polyimines were displayed below 400 nm. These bands indicate the presence of transitions of their azomethine chromophore group and aromatic ring.³⁵ The UV–visible spectra show distinct absorption maxima at 288 and 324 nm after addition of (L)-Lysine. The Tauc plot has been developed for the allowed UV–visible transition spectra. The band gap of tvitrimer R was calculated to be 2.2 eV and shifted to 1.51 eV after addition of the (L)-Lysine solution (Figure S1). A separate band gap of chiral diimine (DFF with Lysine) was

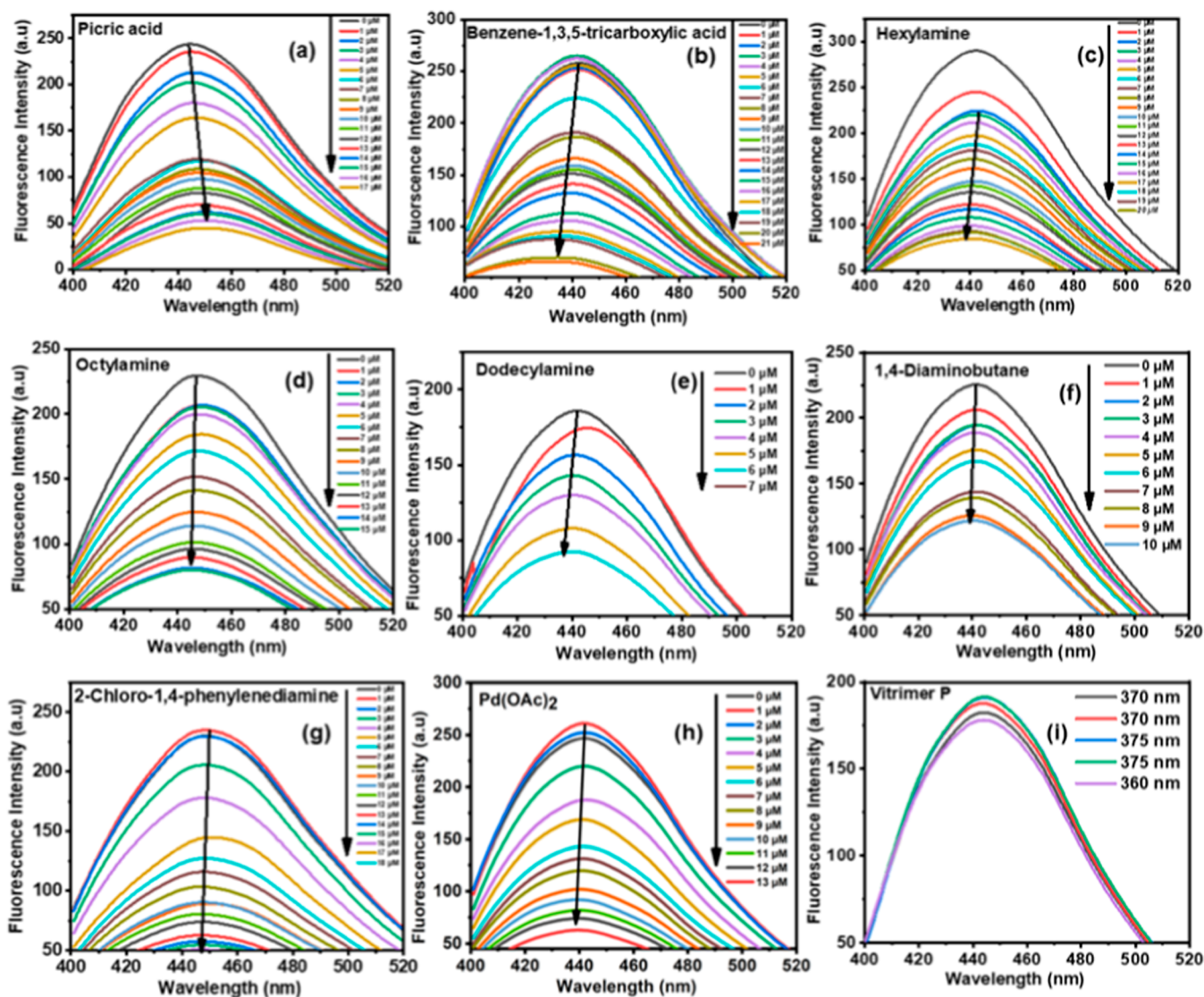


Figure 5. Fluorescence quenching of polyimine vitrimer P using (a) picric acid; (b) 1,3,5-benzenetricarboxylic acid; (c) hexylamine; (d) octylamine; (e) dodecylamine; (f) 1,4-diaminobutane; (g) 2-chloro-1,4-phenylenediamine; (h) palladium acetate; (i) emission spectrum of vitrimer P at different excitations.

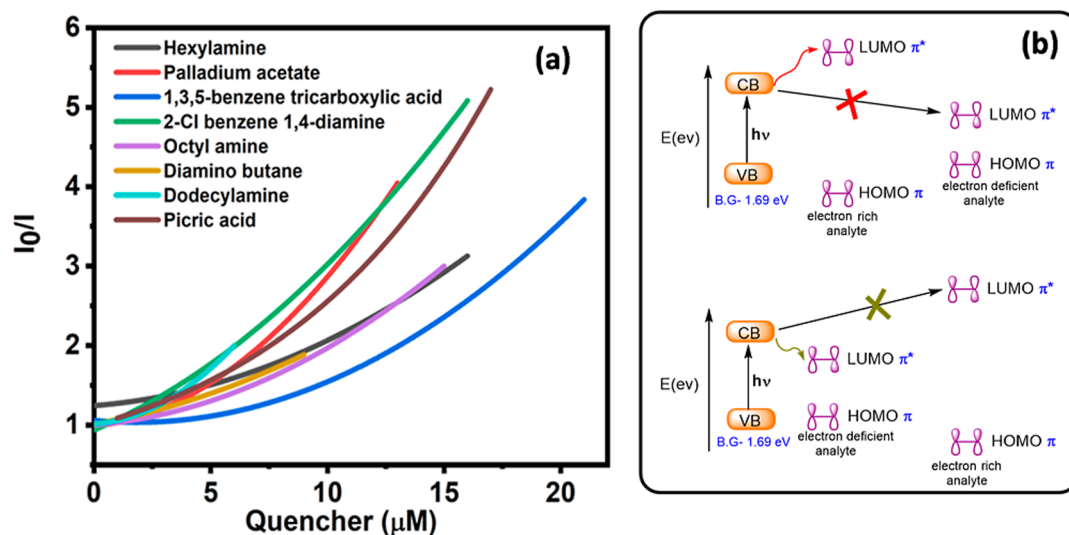


Figure 6. (a) Stern–Volmer plot between I_0/I and molar concentration of the quencher (in μM). (b) MOT to quench the fluorescence property.

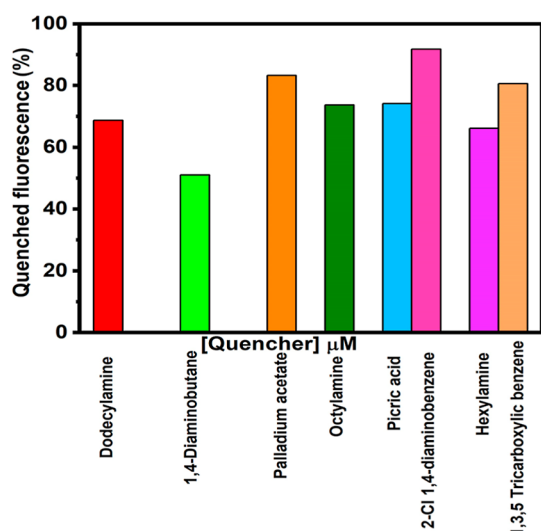


Figure 7. Comparison of percent quenched fluorescence with concentrations of different [quencher] μM .

found to be 1.50 eV (Scheme S1). This result confirms that the imine–amine exchange reactions take place in the presence of an excess of amine (Scheme S2).

2.4. Fluorescence Quenching. The fluorescence spectra of vitrimer “P” in ethanol is shown in Figure 4. It shows an

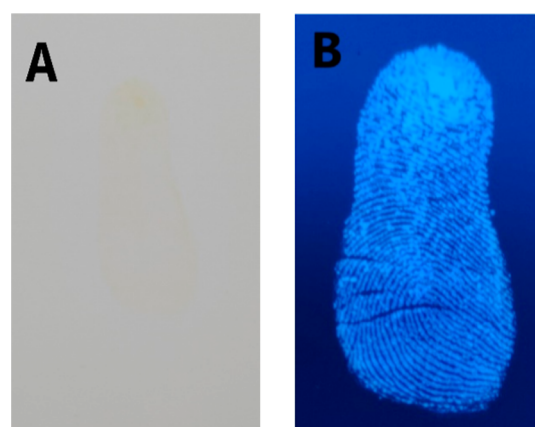


Figure 9. Exposure of polyimine vitrimer P in the presence of (a) visible light; (b) UV-light (365 nm).

emission maximum at 371 nm.^{36,37} The fluorescence quantum yield of vitrimer “P” is 45.85% using fluorescence as a standard.³⁸ Its fluorescence property was quenched using toxic picric acid, amines, and carboxylic acids. The fluorescence excitation and emission spectra of “P” appeared at 360 and 450 nm, respectively (Figure 4).³⁹

Picric acid is a nitroaromatic explosive that has been used in landmines and it is also an important feedstock in rocket fuel, pigment, and pesticide industries.⁴⁰ It is a highly environ-

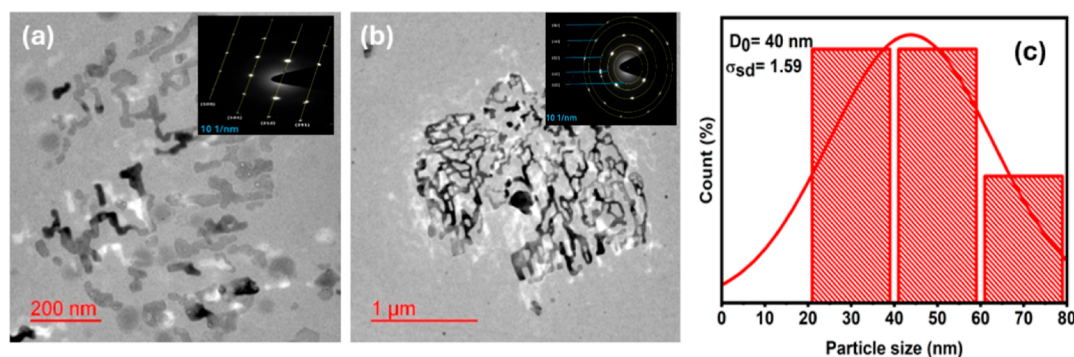


Figure 8. TEM analysis of palladium NPs stabilized by polyimine vitrimer P at (a) 200 nm (b) 1 μm ; (c) particle size distribution.

Table 1. Exposure of Vitrimer *P* to Various Environmental Conditions in the Presence of UV-Light (365 nm)

Entry no	Time (h)	0.1M HCl	0.1M NaBH ₄	0.1M NaOH	0.1M H ₂ O ₂	Toluene
1	0					
2	0.25					
3	6		solubilised	solubilised	solubilised	
4	18		solubilised	solubilised	solubilised	
5	48		solubilised	solubilised	solubilised	

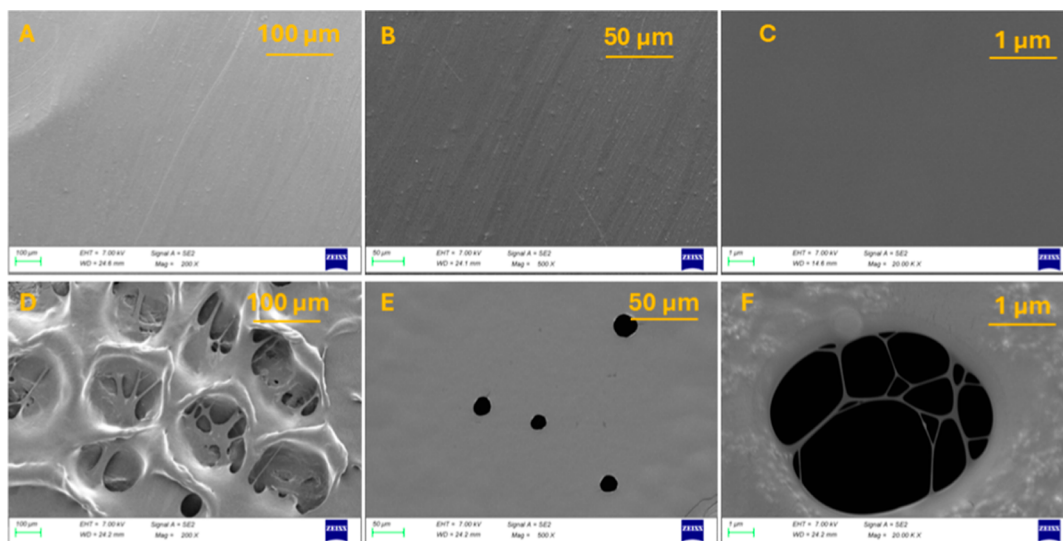


Figure 10. SEM image of plastic where (A–C) is the low-density thermoplastic at 100 μm , 50, and 1 μm and (D–F) are the same plastic after exposure to polyimine *vitrimer R* at the same scale.

mental pollutant material and causes many health issues. For the quenching experiment, 1.0 mmol of “P” was dissolved in ethanol (5.0 mL). To this vitrimer solution “P”, picric acid was added dropwise (1.0 mmol in 5.0 mL of ethanol). As shown in Figure 5, fluorescence quenching started from the addition of zero micro-molarity concentration of picric acid and continued until the solution reached a 1.0 μM concentration. After the addition of a 5.0 μM quencher, the fluorescence completely ceased. This significant decrease is due to the formation of a collisional encounter between fluorophore and the quencher called collisional quenching.

As shown in Figure 5, from fluorescence intensity versus wavelength plot, maximum emission obtained was at 450 nm (Figure 5a).⁴¹ Further, the interaction between the carboxylic acid group of the aromatic and imine makes it possible to use the vitrimer as a fluorescence probe to detect organic acids in aqueous solution. The fluorescence property of the vitrimer is strongly quenched in the presence of dicarboxylic acid.⁴² The vitrimer “A” in ethanol emits yellow fluorescence at 360 nm, and 1,3,5-tricarboxylic benzene also dims the light emitted by the vitrimer strongly (Figure 5b). On addition of the 20 μM solution, complete quenching of fluorescence with blue shift was observed.

2.5. Amines. The fluorescence of the vitrimer solution (*P*) is strongly quenched when aromatic amines were added.

Different fluorescence responses of the vitrimer to the ethanolic solutions of amines (aliphatic and aromatic) show high selectivity to aromatic amines. The aromatic π – π interactions and imine moiety of the vitrimer may play a significant role. In aliphatic amines,⁴³ hexyl amine, octyl amine, dodecyl amine, 1,4-diaminobutane did not quench the fluorescence completely. Aromatic amines quench fluorescence completely by the addition of a 18 μM solution. The quenching by the aliphatic monoamines and diamines is less effective than that of the aromatic amine (Figure 5c–f). Among all aromatic amines, aromatic diamine, i.e., 2-chloro-1,5-diaminobenzene, shows the significant π – π interaction with the vitrimer and aliphatic amines adopt an electron transfer mechanism (Figure 5g). Later we studied the fluorescence quenching property of the vitrimer using picric acid. A constant volume of the vitrimer was titrated against picric acid and changes in absorbance were observed. Complete fluorescence quenching was observed with a 20 μM solution.

2.6. Quenching by Palladium Acetate. The addition of palladium acetate (1.0 mM solution) led to a significant quenching of the vitrimer “P” to. Complete disappearance of the polyimine absorption band (Figure 5h) is also noted. Even as little as 0.010 mM palladium acetate caused the vitrimer to lose 92% of its initial value. A vitrimer solution was added to

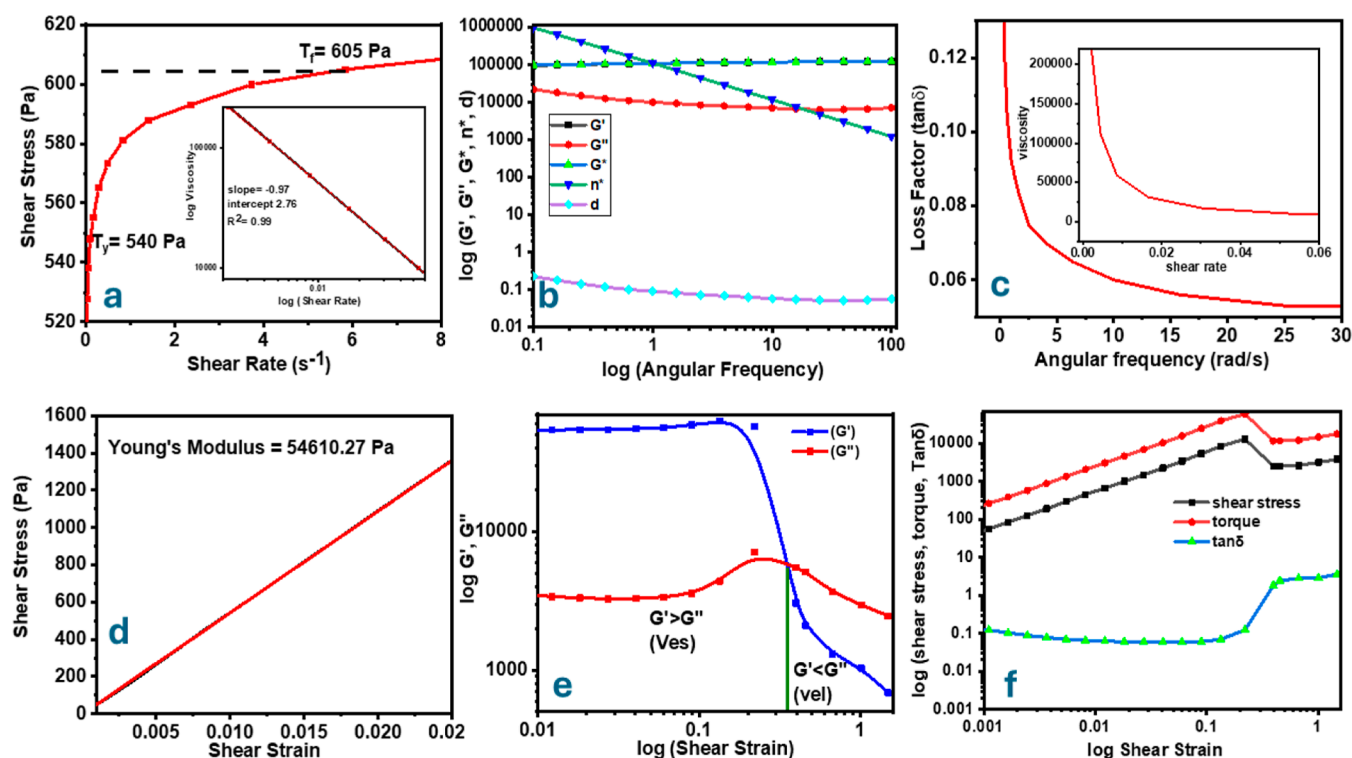


Figure 11. Rheological study of vitrimer P/ethanol: (a) flow curve (b) frequency sweep, and (c) angular frequency. (d) Young modulus, (e) viscoelasticity, and (f) phase deformation, whereby G' , G'' , T_y , T_f , G^* , d , and n^* are storage modulus, loss modulus, yield stress, fluid stress, phase difference, complex modulus, and complex viscosity, respectively.

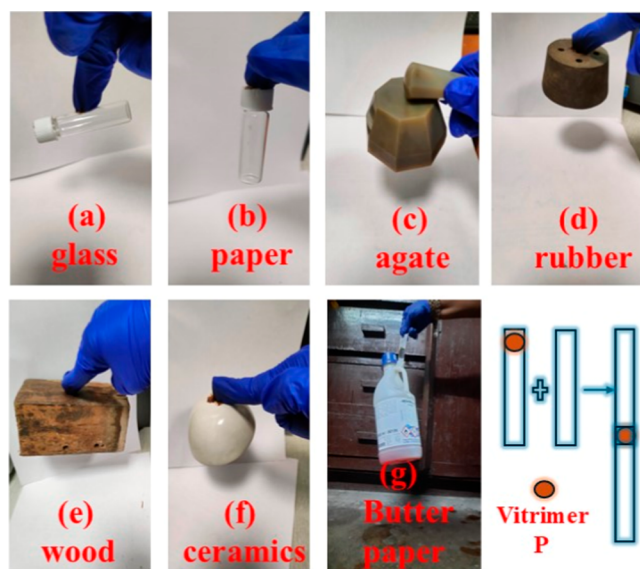


Figure 12. Schematic representation of elastomer *vitrimer P* on various substrates: (a) glass, (b) PTFE, (c) agate, (d) rubber, (e) wood, (f) ceramics, (g) butter paper.

metal salt solution dropwise (yellow to colorless) until its fluorescence completely disappeared (Figure 5i).

2.7. Stern–Volmer Plot. The interaction of the fluorescent emitter and a quencher is studied using the Stern–Volmer plot. A graph was plotted between I_0/I and quencher concentration [Quencher] in μM (Figure 6). It shows an upward deviation from linearity toward the y-axis at a higher concentration of the quencher. At a lower concentration, linearity is maintained to some extent. This may be due

to the fluorophore being quenched by the collision and complex formation. Evidently, picric acid enhances the absorption and quenches the fluorescence maxima. In approximately all cases, at lower concentrations of the quencher, a linear relationship is evident; however, as the quencher concentration increases, the relationship becomes nonlinear (Figure 6). This behavior is commensurate with the arrangement/density of π electrons in the aromatic rings of the respective amines. The fluorescence quenching studies of polyimines were also conducted in the presence of aliphatic amines such as hexyl, octyl, dodecyl amine, 1,4-diamino butane, where all of them show a quenching behavior due to their sigma electron density and direct interaction with the imine of the vitrimer. A long chain monoamine, hexylamine, showed better performance than other amines. Picric acid enhanced the absorption and quenched the fluorescence maxima.⁴⁴

The fluorescence quenching property was explained by molecular orbital theory (Figure 6b). For electro-deficient aromatic compounds like 1,3,5 tricarboxylic benzene and trinitrophenol, the lowest unoccupied MO (LUMO) is low-lying π^* -type, which is stabilized by the electron-withdrawing groups. This is due to conjugation, which has a lower energy than the conduction band (CB) of imine-based electron-rich vitrimers. The UV absorbance data (*Tauc* plot) show a band gap at 1.69 eV, representing the $\pi \rightarrow \pi^*$ transition. In the case of electron-rich aromatic amines, the fluorescence first increased and shifted toward the blue region, and after reaching a certain point, it decreased. The lowest unoccupied MO (LUMO) has a higher energy π^* -type orbital stabilized by the NH_2 group through + R type conjugation, and its energy is above the conduction band (CB) of the vitrimer in the case of electron-rich aromatic amines. The aliphatic amines have σ -

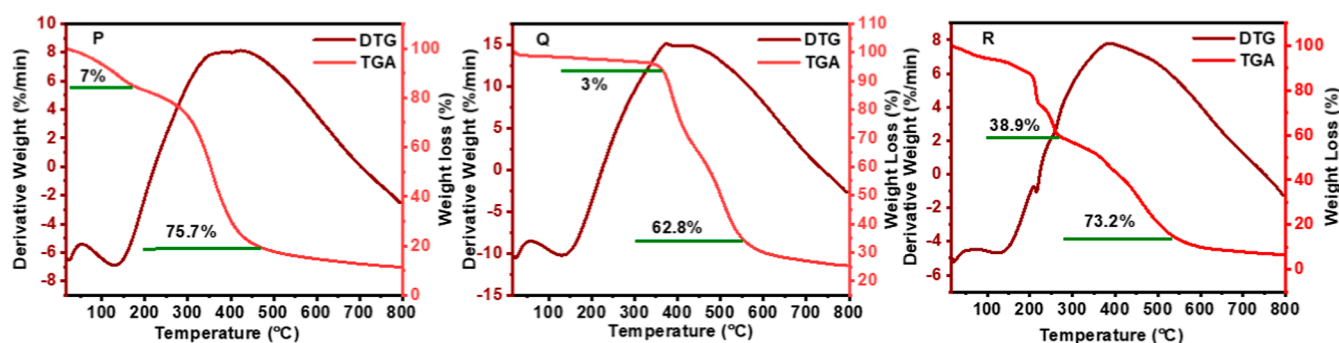


Figure 13. TGA and DTA analyses of vitrimers P, Q, R.

type LUMO, dissimilar to the π -type conductance band of imine-based vitrimers. The π -type LUMO of aromatic amines has greater overlap probability than the σ -type LUMO of aliphatic amines with the π -type CB of the vitrimer. The furan ring plays a major role in determining the HOMOs of the polymers, while the LUMOs are attributed mainly to --HC=N bonds and adjacent aromatic rings⁴⁵

2.8. Quantum Yield. Quantum yield for the bio-vitrimer was calculated relatively by taking a known fluorescein as a reference material.⁴⁶ The graphical comparison of quenching (%) with respect to the concentration in μM is displayed in Figure 7.

2.9. Palladium Nanoparticles. In this report, a Pd nanoparticle was prepared stabilized by biobased polyimine vitrimer “P”. In Figure 8, the TEM image of palladium-incorporated “P” materials with two different scales 200 nm and 1 μm is represented. The average particle size (D_0) and standard deviation (σ_{sd}) of Pd NPs were calculated as 40 nm and 1.59, respectively. These NPs were prepared by mixing palladium acetate (20.0 mg) with polyimine “P” (200.0 mg) in ethanol solvent (10.0 mL) at 70 °C overnight. Later, a mixture of sodium borohydride (1.0 mmol) dissolved in distilled water (5.0 mL) and 1.0 mL of PVP were added slowly for 10.0 min. Figure 8 (insight) shows an indexed SAED pattern of single and polycrystalline Pd-vitrimer. The “ d -spacing” for a single crystalline Pd from the SAED pattern is calculated to be 1.8 2.7, 2.7, and 2.19 Å, having (hkl) values of (103), (141), (212), and (231), respectively. For a polycrystalline Pd, the “ d -spacing” value is in the range of 3.9–1.26 Å (Table S4). These data are consistent with reported literature data as per JCPDS card number 78–0639, having a monoclinic primitive structure, with “ a ” value of 6.825 Å, “ b ” value of 12.73 Å, and “ c ” value of 4.779 Å.⁴⁷

The stability of polyimine vitrimers “P” can be explained by the nondissolution of materials in various solvents such as toluene, acetone, and ethyl acetate even after 3 days. However, in ethanol, water “P” is undergoing dissociation with opening of imine bonds and simultaneous release of amines and aldehydes into a water/ethanol solution. This dissociation (discoloration) happens only partially after 72 h. This is due to the absence of either acid or higher temperatures. The solubility of polyimine “Q” in acetonitrile, methanol, ethyl acetate, THF, 1.0 M NaOH, and 1.0 M HCl (35%) was tested. It was found that in THF, polyimine “Q” was completely soluble and converted into a monomer (recyclable), whereas it was quite stable in acidic and basic conditions (HCl and NaOH). It remains intact in acidic and basic solutions for more than 3 days. They show slight discoloration in ethyl acetate and acetonitrile. The cross-linking density and presence

of furan rings were likely responsible for the stability of network in high acidic and basic solutions (Figures S3 and S4).

2.10. Applications of “Polyimine Vitrimer P”.

2.10.1. Anti-Counterfeiting Labels. Counterfeiting has been a severe problem over the past few decades due to the adverse impact of illicit activities.^{48,49} It also poses threats to the welfare of consumers, along with disrupting society on various levels. Therefore, there is an urgent need to develop a novel anticounterfeiting material, which can be stable under acidic, basic, and oxidant conditions. In this direction, fluorescent materials have been developed for anticounterfeiting due to their robustness and stability. Various fluorescent materials such as organic dyes, inorganic semiconductors, quantum dots, and luminescent materials have been widely used. Among these materials, carbon-based materials have been widely used for next generation, owing to their high abundance and hence these are used for security labels.⁵⁰ In this work, polyimine vitrimer P has been used for fluorescent security labels. It is also highly important that the fluorescent property should be intact even under harsh environmental conditions. As shown in Figure 9, the polyimine P exhibits fluorescence by irradiating UV light with a wavelength of 365 nm. Letters “BHU” were written on butter paper, and the letters can easily be seen by the naked eye under UV light excitation as they show blue color, unlike in the presence of natural light (Figures S5 and S6). In our experiment, “polyimine vitrimer P” (2.0 mg) was added to ethanol (5.0 mL) at 25 °C and was allowed to stay for 15.0 min. Later, the thumb of the right-hand is dipped in that solution and placed on a TLC plate (Figure 9A,B). The TLC plate with the fingerprint is exposed to visible light, and UV-light (365 nm) is displayed in Figure 9A,B. This result indicates that the polyimine vitrimer can be utilized for forensic purposes as anticounterfeiting materials. This may be due to transition of $n \rightarrow \pi^*$ of the vitrimer when it interacts with UV light radiations.

These prints should not disappear during practical application. In the stability test explored, “vitrimer P” prints in various chemical environments such as alkaline, acidic, reducing, and oxidizing environments. The photographs of the plates before and after the exposure to HCl (0.1 M), NaOH (0.1 M), NaBH_4 (0.1 M), and H_2O_2 (0.1 M) and also in toluene for 48 h are presented (Table 1). Yet, for all conditions, the shapes of the pattern remain the same and can be clearly seen by eyes under UV light excitation (365 nm) and are invisible in the range of 400–800 nm. In aq. NaBH_4 , NaOH and H_2O_2 solutions, was completely soluble after 25.0 min. However, this polyimine P is highly stable under acidic conditions even after 48 h, illustrating that dynamic network cross-linking helps to stabilize under low pH conditions.

2.10.2. Plastic Degradation. Imines, which contain a double bond between nitrogen and a carbon atom ($-\text{CH}=\text{N}$), can undergo several types of reactions that can generate free radicals, especially when exposed to specific external conditions such as UV light, heat, or in the presence of chemical initiators.^{51,52} We assumed that the vitrimer is having dynamic associative cross-linking, which is temperature-dependent and exchange reactions take place. We added 5.0 μL of vitrimer R to ethanol (5.0 mL) and heated it at 80 °C for 1 h. Later a polyethylene sheet was immersed in that solution for 2 days at room temperature. After 2 days, the polyethylene sheet was dried under vacuum and submitted for SEM analysis. It has been reported in the literature that an imine-based vitrimer can generate free radicals at elevated temperatures. Therefore, we expected that it can degrade polyethylene plastic (low-density polyethylene), and SEM images of plastic are provided below after treatment with vitrimer P (Figure 10).

At elevated temperatures, the imine linkages can break through homolytic cleavage (splitting of the bond into two radicals).⁵³ This could initiate a free radical process, especially in the presence of oxygen (leading to oxidation). The interaction of imine-based polymers with oxygen, especially under conditions of UV exposure, may lead to the formation of reactive oxygen species (ROS), such as peroxy radicals. These ROS can further generate free radicals, contributing to the material's degradation or reactivity.⁵⁴ In the context of polyimine vitrimers, the presence of free radicals could be used to facilitate the dynamic exchange of imine linkages, allowing the polymer to be reprocessed or repaired by recross-linking the chains.⁵⁵ Since the oxygen in the furan ring is reactive and can interact with molecular oxygen (O_2) to form reactive oxygen species (ROS), such as peroxy radicals ($\text{ROO}^{\bullet}$),⁵⁶ degradation starts with the generation of a free radical.⁵⁷ Free radicals play a central role in initiating chemical reactions that cause the polymer chains to break down, leading to degradation of the plastic. A photostable material in PLE (photoluminescent emission) refers to a material that can withstand prolonged exposure to light without significant degradation of its photoluminescent properties.⁵⁸ This has a lot of significance especially in determination of sensing, anticounterfeiting property. The PLE plot of vitrimer "P" emission intensity versus time plot shows it can be stable even after exposure to light for 1800 s (Figure S9).

2.11. Rheological Property. The Bingham pseudoplastic model includes yield stress and plastic viscosity of vitrimer P. It initially resists flowing until the shear stress exceeds a certain value (yield stress) after that it increased with diminishing rate (Non-Newtonian Fluids) (Figure 11a), with a linear relationship between shear stress and shear rate.⁵⁹ The Herschel Bulky model and power law equation confirmed the Non-Newtonian fluid property of vitrimer P.⁶⁰ Frequency-sweep measurements of "vitrimer P" in the low region show a wide rubbery plateau, indicating storage moduli (G') are significantly higher than the loss moduli (G'') (Figures 11b and S7). This is because networks are inherently cross-linked in the vitrimer⁶¹ and exhibit a solid-like property at high frequencies and liquid-like character at low frequencies.⁶² The viscosity near zero at a high shear rate (Figure 11c) shows a Bingham characteristic.⁶³ Young's modulus has been calculated from amplitude sweep (Figure 10d), the slope value (54,610 Pa) which in the elastic region of stress versus strain.⁶⁴ Before a certain shear strain (Figure 11e), it was a viscoelastic solid (ves) material and after that, it was a viscoelastic liquid (Vel).⁶⁵ The plastic

deformation phase occurred at high shear strain, as shown in Figure 11f. Further mechanical properties from amplitude sweep and frequency sweep are mentioned in Tables S10 and S11.

2.12. Adhesive Property. Adhesive bonding is widely used in electronics, aerospace, biomedical, and other fields.^{66,67} In our work, the prepared imine-based vitrimer (P) had strong detachable, reworkable adhesive properties.^{68–70} Because of its excellent vitrimeric behavior, it can be recycled through dissolution and remodeling.⁷¹ It is noted that the vitrimer was capable of hanging a weight of 75.0 kg of water (Figure S8). Similarly, two butter papers were allowed to stick to each other using vitrimer P, which lifted water (750.0 mL) without curing at a high temperature (Figure 12).

2.13. TGA and DTA Analyses. The thermal stability of different vitrimer materials was studied by TGA. In vitrimer P, only a 7% decomposition at 171.2 °C and 76.7% decomposition at 466 °C (Figure 13) could be deduced from the nonisothermal degradation curves. From DTG analysis of vitrimer P, an exothermic upward reaction was observed due to reversible exchange reaction of the imine bond. However, in the case of vitrimer R, 38.9% weight loss at 274 °C and 73.2% weight loss at 538 °C were observed. Polyimine undergoes thermal decomposition; an exothermic peak might appear on the DTG curve as the imine bonds break down and release heat. The exothermic peak in the DTG curve of polyimine vitrimers is most likely related to the dynamic exchange of imine bonds at elevated temperatures, which can lead to network reorganization, cross-linking, or self-healing behavior (Figure 13).

Finally, powdered X-ray diffraction (XRD) shows that all polyimine materials are highly amorphous. The PXRD data obtained were similar to that of the reported biodegradable polymer and 2θ values range from 20° and 40°, indicating that these were polystyrene thermoplastic (Figure S10). ^1H NMR spectra of polyimines⁷² can be found in the Supporting Information (Figures S11–S14).

3. CONCLUSION

In summary, this work demonstrates a facile and convenient preparation of vitrimers from biomass-derived DFF. The vitrimer derived from polyethylenimine exhibits a fluorescence property, and it can be quenched in the presence of various organic molecules. Among all, aromatic diamines will be able to quench the fluorescence property maximum. In addition, this biobased vitrimer is used as an *anti-counterfeiting material*, with properties of plastic degradation and stabilization of nanoparticles. Further fluorescent vitrimer material behaves like pseudo plastic and has adhesive property. The application of the vitrimer highlights the potential of imine work, which is a recyclable thermoset.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.5c01639>.

Reactant preparation, rheological analysis, FTIR, TGA, UV, and band gap analysis (PDF)

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Author Contributions

K.V.S.R. conceived the main idea. A.R. executed most of the experimental work. Some experimental setup and experimental procedures were performed by M.P. S.T., A.K.P. has performed rheological studies, characterization of vitrimers. The initial manuscript was written by A.R. and corrected by all authors. All authors have given approval to the final version of the manuscript.

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Notes

The authors declare no competing financial interest.

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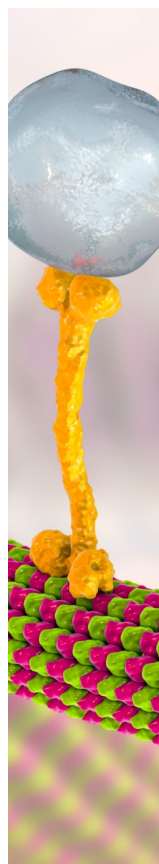
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