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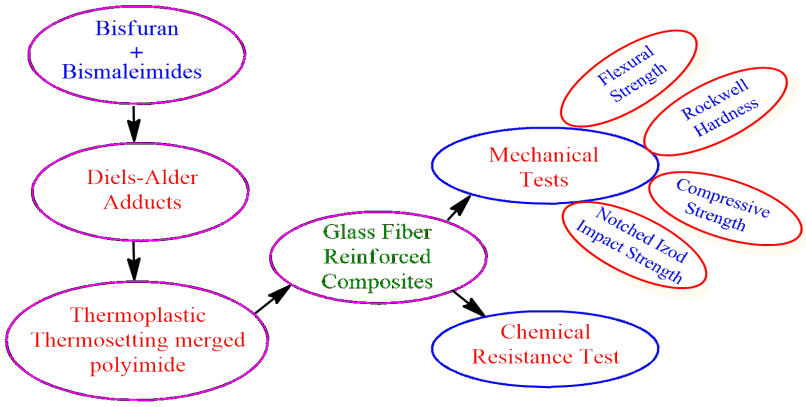
Polyimides Derived from Diglycidyl Ether of Bisphenol-A and Bisphenol-F via Diels–Alder Polymerization

Ami Y. Patel [a] and Yogesh S. Patel* [b]

Novel polyimides were synthesized by Diels–Alder polymerization. Bisfuran (BF) was reacted with several bismaleimides containing the diglycidyl ether of bisphenol-A and bisphenol-F (epoxy segments) to obtain Diels–Alder polyadducts. These polyadducts were subsequently aromatized and imidized (i.e., cyclized) through carboxylic acid and amide group formation to yield polyimides. The synthesized polyadducts and polyimides were characterized by elemental analysis, spectral techniques, number average molecular weight (Mn), and thermal analysis. The 'in situ' glass fiber-reinforced composites were prepared and characterized for their mechanical, electrical, and chemical properties. These properties were compared with those of other reported polyimides. All the composites exhibited good mechanical and electrical properties, as well as excellent resistance to organic solvents and mineral acids.

Graphical abstract



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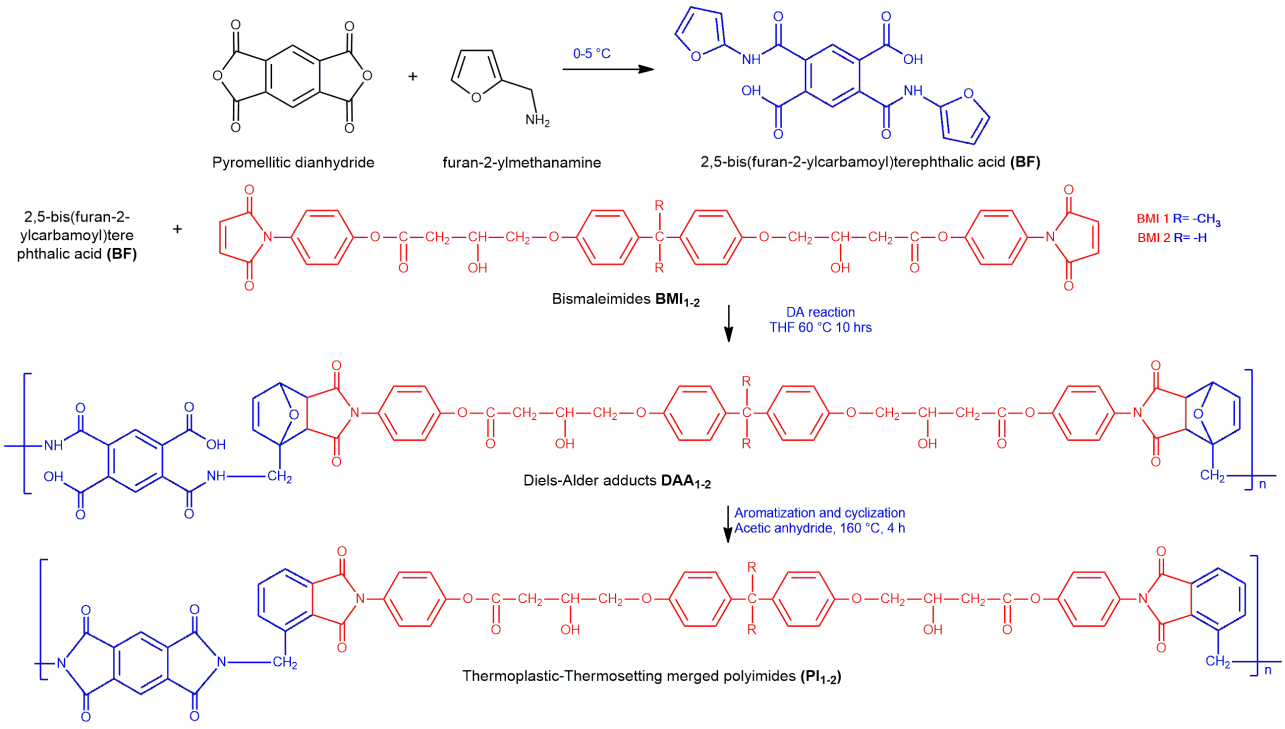
1. Introduction

Polyimides synthesized through Diels-Alder polymerization from bisfuran and bismaleimide monomers offer a promising approach to develop materials with both thermoplastic and thermosetting properties within a single macromolecular structure. The integration of bisfuran and bismaleimide moieties in the polymer backbone successfully allows the formation of robust and versatile polyimides, which exhibit desirable thermal, mechanical, and chemical properties. The Diels-Alder reaction, followed by aromatization and imidization, proves to be an effective method for the synthesis of these polyimides, leading to materials that may find applications in high-performance coatings, adhesives, and advanced composites. Future work will focus on optimizing the reaction conditions, exploring additional functionalization strategies, and evaluating the long-term stability and processability of these novel polyimides in various industrial applications. The pioneering work related to DA polymerization was carried out by Stille and Plummer [1]. PIs involving a bisfuran and bismaleimide monomer combination were reported by Tesoro and Sastri [2].

[a] Chemistry Department, S.P.T. Arts & Science College, Shri Govind Guru University, Godhra (389001) Gujarat INDIA. [b] Government Science College, Limkheda (389140) Gujarat INDIA. *Corresponding author. E-mail: **dryspatel@gmail.com**

Various strategies have been proposed to synthesize or modify polymer structures by exploiting the reactivity of furan derivatives with different dienophiles. Notable contributions in this area have come from the research groups [3-10]. A systematic study of DA polymerization involving furan derivatives and various bismaleimides has also been carried out by Gandini [11, 12] and other research groups [13-19]. Patel and his co-workers have reported studies on polyimides such as poly(epoxy-imide) [20], poly(s-triazine-imide) [21], poly(ether-imide) [22], poly(ester-amido-imide) [23], poly(amido-imide) [24], poly(ester-oxysilane-imide) [25], and poly(urethane-imide-ester) [26], through DA polymerization of bisfuran and bismaleimide with various structural variations.

Building on this concept and our prior work [27-30], primary objective was to prepare polyimides that possess both thermoplastic and thermosetting segments by introducing bisfuran and bismaleimide moieties in a single macromolecular framework. This study focuses on the synthesis of polyimides via DA reaction, followed by aromatization and imidization. Scheme 1 summarizes the synthetic approach adopted in this work, which involves the following phases: (i) Synthesis of bisfuran (BF) from pyromellitic dianhydride and furan-2-ylmethanamine, (ii) Synthesis of DA polyadducts DAA1-2 between the bisfuran (BF) and bismaleimides (BMI1-2), and (iii) Aromatization and imidization (i.e., cyclization) reactions to afford thermoplastic–thermoset merged polyimides (PI1-2). The *in situ* glass fiber-reinforced composites (GFRC1-2) were prepared from the synthesized polyimides and characterized by chemical, mechanical, and electrical analysis. The details of the synthesis procedures and the results obtained are discussed below.



Scheme 1. Synthesis of Thermoplast-thermoset merged polyimides.

2. Results and Discussion

Monomer synthesis

Synthesis of Bisfuran (BF)

Bisfuran (BF) was synthesized following the method reported by us [27-30]. The ^{1}H NMR spectrum showed a doublet peak at 4.32 δ ppm, corresponding to the methylene group. A doublet at 7.50 δ ppm, triplet at 7.75 δ ppm, doublet at 7.93 δ ppm, and singlet at 8.11 δ ppm were attributed to the six protons of the furan ring. The singlet at 10.93 δ ppm was assigned to the protons of the carboxylic acid group, while a triplet at 8.90 δ ppm corresponded to the –NH proton of the amide group, which was further confirmed by ^{13}C NMR data. In the DEPT-135 spectrum of bisfuran, the inverted peak at 42.24 δ ppm indicated the presence of a methylene bridge between the amide and furan rings. Peaks corresponding to substituted carbons of aromatic rings and carbonyl carbons were absent, while peaks for unsubstituted aromatic carbons were observed at 117.78, 119.24, 123.46, and 129.33 δ ppm in the DEPT-135 spectrum, supporting the proposed structure [27-30].

Synthesis of Bismaleimides (BMI1-2)

Bismaleimides were prepared by the method reported [31-32].

Polymer synthesis

The thermoplast-thermoset merged polyimide system was synthesized through a Diels-Alder (DA) intermolecular reaction, followed by simultaneous aromatization and imidization of the DA adducts via cyclization [27-30]. Elemental analyses of the DA adducts indicated that the cycloaddition of furan and bismaleimide rings proceeded with good yields, consistent with the predicted structures (Scheme 1). The degree of polymerization (DP) for the polymers DAA1-2, as estimated by non-aqueous titration, was found 7.

The IR spectra of DAA1-2 exhibit prominent characteristic bands for furan, amide, imide, ether, and carboxylic acid groups. The bands near 3120 cm^{-1} and 1580 cm^{-1} are

attributed to the furan ring. The bands around 3220 cm^{-1} and 1670 cm^{-1} correspond to the amide groups, while the bands around 3530 cm^{-1} and 1710 cm^{-1} are assigned to the -OH and -C=O stretching vibrations of the carboxylic acid group, respectively.

A comparison of the IR spectra of the non-aromatized and non-cyclized DAA1-2 with those of the aromatized and imidized PI1-2 revealed distinct differences. The IR spectra of the polyimides PI1-2 exhibited significant changes compared to those of the non-aromatized and non-cyclized DAA1-2, clearly confirming that aromatization and simultaneous imidization reactions had occurred with good yields for all the DA adducts. The IR spectra of polyimides displayed prominent characteristic bands corresponding to imide, aromatic carbon. The bands near 3030 cm^{-1} and 1510 cm^{-1} were attributed to the aromatic ring, while the bands around 2950 cm^{-1} and 2830 cm^{-1} were associated with aliphatic carbon. Additionally, the bands around 1785 cm^{-1} and 1730 cm^{-1} corresponded to asymmetric and symmetric imide groups, respectively. When comparing the IR spectra of DA adducts and polyimides, significant differences were observed. The IR spectra of DA adduct exhibited prominent characteristic bands for furan, amide, and carboxylic acid groups. A comparison of the IR spectra of non-aromatized and non-cyclized DA adducts with those of aromatized and imidized (cyclized) polyimides revealed clear distinctions. The disappearance of bands corresponding to carboxylic acid and amide groups in the spectra of DA adduct indicated that the imidization reaction had proceeded smoothly. Moreover, the disappearance of bands associated with the furan ring confirmed the completion of the aromatization reaction [27-30].

The thermal stability of the DAAs and PIs was evaluated through thermogravimetric (TG) analysis. DAA1-2 undergoes two stages of mass loss. The first stage, observed in the temperature range of 180°C to 300°C, corresponds to the decarboxylation of the polymer. The experimental weight loss values for the DA polyadducts were: DAA1: 7.42% and DAA2: 7.59%, which are in close agreement with the theoretical values: DAA1: 7.3% and DAA2: 7.4%. This consistency confirms that the Diels–Alder reaction was completed as expected. The second stage, above 300°C, corresponds to the pyrolysis of the polymer. The char residue left at 700°C was in the range of 4-6% for DAA1-2. A rapid rate of weight loss was observed between 400°C and 600°C.

For PI1-2, decomposition started above 400 °C (Figure 1), depending on the nature of the polyimides. All polymer samples showed approximately 50% weight loss in the temperature range of 500°C to 600°C, with complete weight loss (around 95–96%) occurring at about 700°C. The aromatized and imidized PI1-2 samples exhibited greater thermal stability than their pre-polymers (DAA1-2), which showed the decarboxylation behavior. Consequently, PI1-2 began their degradation at slightly higher temperatures compared to the non-aromatized DAA1-2.

A comparison of the thermal stability of all the polyimides revealed the following order of stability: B2 > B1. While most reported thermal data for commercial polyimides have been determined in nitrogen (N_2), the present study was conducted in air. This difference is noteworthy for comparison. Nevertheless, it can be concluded that the produced polyimides (PI1-2) exhibit good thermal stability and are comparable to commercial polyimides.

Composite Characterization

Chemical Resistance Test

The chemical resistance of all composite samples was evaluated according to the ASTM D543 method. This test assesses the resistance of composite materials to various chemical reagents, including acids, alkalis, and organic solvents, that the material may encounter in practical applications. The test involves measuring changes in weight, dimensions, and appearance of the samples after exposure to these reagents. The composite samples were immersed for 7 days at room temperature in the following chemical reagents: 25% v/v H_2SO_4, 25% v/v HCl, 25% w/v NaOH, ethanol, acetone, DMF (dimethylformamide), and THF (tetrahydrofuran). After 7 days, the samples were removed from the reagents and analyzed for changes in thickness and weight. The percentage weight loss in the composite samples is summarized in Table 1. All samples exhibited excellent resistance to acids, alkalis, and organic solvents, with no significant changes in physical appearance. However, concentrated alkali (NaOH) caused a slight change in thickness, with a reduction of approximately 1.6-2.0 %, while DMF caused a slightly higher reduction of 1.8-2.0 % in thickness.

Mechanical tests were then conducted on the specimens to further evaluate the material properties after chemical exposure.

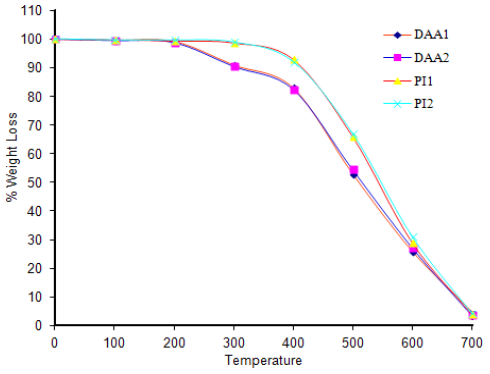


Fig. 1. Thermogram of DA adducts DAA1-2 and Polyimides PI1-2.

Mechanical and Electrical Tests

All the mechanical tests were performed using their specimens.

The compressive strength was measured at room temperature using a Universal Instron testing machine, in accordance with ASTM D695. Compressive properties describe the behavior of a material when subjected to compressive loads. Compressive strength is defined as the maximum force per unit area that a material can withstand in compression. The compressive strength of the composites was found to be in the range of 215–225 MPa.

The notched Izod impact strength was measured at room temperature using a Zwick Impact machine (Model No. 8900) in accordance with ASTM D256. The notched Izod impact test is a single-point test that measures a material's resistance to impact from a swinging pendulum. Izod impact is defined as the kinetic energy required initiating and propagating a fracture until the specimen breaks. Specimens are notched to prevent local deformation upon impact. This test is commonly used as a quality control measure to assess whether a material meets specific impact properties or to compare materials for general toughness. The results for the notched Izod impact strength of the composites were found to be in the range of 235–250 J/m.

Rockwell hardness was measured at room temperature using a Rockwell Hardness Tester (RAS/Saro Engg. Pvt. Ltd., India) according to ASTM D785. Rockwell hardness is a measure of a material's resistance to indentation. The test is conducted by first applying a minor load to a steel ball indenter, followed by an increase to a specified major load. The load is then decreased back to the minor load, and the Rockwell hardness is determined based on the net increase in the depth of the indentation. The Rockwell hardness of the composites was found to be in the range of 101–107 HR.

Flexural strength was measured at room temperature using a Universal Instron testing machine according to ASTM D790. Flexural strength is defined as a material's ability to resist deformation under a load. For materials that undergo significant deformation but do not break, the load at yield (typically measured at 5% strain of the outer surface) is reported as the flexural strength. The flexural strength of the composites was found to be in the range of 260–270 MPa.

The mechanical test results (Table 1) indicate that all the composites possess good mechanical properties, particularly due to the presence of bismaleimides. The overall trend in the mechanical properties of the prepared PI/glass fiber-reinforced composites is as follows: GFRC2 > GFRC1. This trend can be attributed to the increased rigidity imparted by the bismaleimide component.

The dielectric strength of an insulating material is defined as the maximum voltage required causing dielectric breakdown. The samples were placed between two electrodes, and the voltage was applied at a uniform rate of 500 V/s until breakdown occurred, typically between 10 and 20 seconds. Five breakdown voltage measurements were taken, and the electrical strength was calculated in kV/mm. The dielectric strength was measured using a high-voltage tester were found 18.9 to 20.0 kV/mm.

Table 1. Chemical, Mechanical and Electrical Properties of Glass Fiber Reinforced Composites GFRC1-2.

Composite*	GFRC1		GFRC2	
Mechanical properties				
Flexural strength	260 MPa		265 MPa	
Compressive Strength	215 MPa		220 MPa	
Notched Impact strength	235 MPa		240 MPa	
Rockwell hardness	101 MPa		104 MPa	
Electrical Properties				
Electrical strength	20.0 kv/mm		18.9 kv/mm	
Chemical Properties				
% Change in composites	*Thickness*	*Weight*	*Thickness*	*Weight*
25% H_2SO_4	1.2	1.5	1.2	1.5
25% HCl	1.0	1.2	1.1	1.2
25% NaOH	1.2	1.6	1.3	1.9
Ethanol	0.9	1.0	0.9	1.1
Acetone	1.0	1. 2	1.1	1.2
DMF	1.7	1.9	1.7	1.8
THF	1.3	1.6	1.4	1.5

* Reinforcement - E type glass cloth, Plain weave, 10 mm, 10 layers, Resin Content: 40±2%; Weight % molar ratio: - BF: BMI1-2 = 1:1(mol/mol); Curing temperature=150±10 °C; curing time=10h; curing pressure=60-70psi; Composite size: 25mm × 25mm × 3mm.

Glass fiber-reinforced composites of polyimides derived from bisfuran and bismaleimides with epoxy resins (DGEBA/DGEBF) exhibit superior mechanical and thermal properties compared to traditional bisfuran-bismaleimide systems [27-30]. The addition of epoxy enhances crosslinking density, strengthening the polymer network through epoxy–amine, epoxy–maleimide, and epoxy–polyimide interactions. Improved fiber-matrix adhesion increases load transfer efficiency, while ether linkages from epoxy resins enhance toughness and fracture resistance, reducing brittleness and microcracking. Furthermore, thermal and chemical stability are significantly improved, offering higher oxidation resistance, increased glass transition temperature (Tg), and superior solvent/moisture resistance. This hybrid composite system provides enhanced strength, durability, and performance, making it ideal for high-end applications in aerospace, automotive, and advanced engineering fields.

3. Material and Methods

All chemicals e.g Pyromellitic dianhydride, Furan-2-ylmethanamine, Commercial epoxy resin, diglycidyl ether of bisphenol-A (DGEBA), diglycidyl ether of bisphenol-F (DGEBF) and solvents used were of laboratory grade and purchased from the local market. Epoxy equivalent weight (EEW) specification of DGEBA and DGEBF are 190 and 160.

Elemental analysis was performed using a Thermo Finnigan Flash 1101EA (Italy). Infrared spectra were recorded on a Nicolet-760 FTIR spectrophotometer. 1H NMR and 13C NMR spectra were recorded on a Bruker spectrometer (400 MHz). The number average molecular weight (Mn) of the polyimides was determined by non-aqueous conductometric titration, following the method reported by [33-37]. Pyridine was used as the suspending solvent, and tetra-n-butyl ammonium hydroxide in 1,4-dioxane was used as the titrant. The thermal behavior of BF, DAA1-2 and PI1-2 were investigated by thermogravimetric analysis (TGA) using a Perkin Elmer TGA analyzer. The samples were heated at a rate of 10 K/min over the temperature range of 50–700°C in the presence of air. As the polyimides are insoluble in common organic solvents, their viscometric properties were not studied. E-type of glass woven fabric 0.25 mm thick was used for glass reinforcement.

Synthesis of Bisfuran (BF)

Bisfuran (BF) was synthesized following the method reported by us [27-30], Adding dropwise a solution of furan-2-ylmethanamine (0.2 mol) to a stirred solution of pyromellitic dianhydride (0.1 mol) while maintaining the temperature at 0-5°C for 1 hour. The resulting solution was then poured into ice water, leading to the precipitation of the reaction product. The white precipitate was filtered, washed, and purified by column chromatography.

Synthesis of Bismaleimides (BMI1-2)

Synthesis of Diglycidyl Ether of Bisphenol A (DGEBA) and Bisphenol F (DGEBF): Dissolve bisphenol A in excess epichlorohydrin at 60-70°C. Add aqueous NaOH to deprotonate hydroxyl groups, promoting etherification. Bisphenol A : Epichlorohydrin : NaOH ≈ 1 : 10 : 2. Nucleophilic substitution displaces chlorine in epichlorohydrin, forming glycidyl ether intermediates. Filter out NaCl, wash with water, and remove unreacted epichlorohydrin by vacuum distillation to obtain DGEBA resin. Similarly, bisphenol F reacts with

epichlorohydrin in NaOH to produce DGEBF, a lower-viscosity, more flexible epoxy resin.

Synthesis of Bismaleimides (BMI1-2)

Bismaleimides were prepared by the method reported [31-32]. In a three-neck round-bottom flask, N-4-carboxyphenyl maleimide (20 mmol) and tetrahydrofuran (THF) (20 mL) were added and stirred under nitrogen (N_2). The mixture was then refluxed until the solution became homogeneous. Epoxy resin (diglycidyl ether of bisphenol-A, DGEBA, or bisphenol-F, DGEBF) (20 mmol) and triethylamine (TEA) (4 mmol) were added to the solution via syringe. Upon the addition of these reagents, the color of the solution changed from yellow to light orange. After 6 hours, the solution turned red and was removed from heat. Upon cooling to room temperature, the viscous, dark red solution was stirred with 20 mL of 5% hydrochloric acid (HCl) solution and then extracted with 40 mL of dichloromethane (CH_2Cl_2). The organic layer (CH_2Cl_2) was separated and evaporated using a rotary evaporator (Büchi Rotovapor). The isolated red product, which was obtained as a pasty mass, was dried under reduced pressure at 65°C for 12 hours.

Synthesis of Diels–Alder Adducts (DAA1-2)

1:1 molar ratio of bisfuran to bismaleimide ensures complete reaction of both functional groups. Bisfuran (BF) and bismaleimide (BMI1-2) were dissolved in 50 mL of tetrahydrofuran (THF). The resulting mixture was refluxed for 10 hours at 60°C. After the reaction, the mixture was cooled and poured into a large volume of dry ether. The precipitates formed were filtered, washed, and air-dried. The unaromatized and uncyclized product obtained was designated as the Diels–Alder adducts (DAA1-2). These adducts were purified and characterized by elemental analysis, FTIR spectroscopy, and thermal analysis.

DAA1: Yield 70%; Color light brown; Empirical Wt. 1187.12g; IR (KBr, cm^{-1}): 3530(-OH st. acid), 3250(-NH st. amide), 3120(C-H st. furan ring), 3031(-CH st. aromatic ring), 2955, 2843(-CH st. aliphatic), 1781(C=O st. asym. imide), 1732(C=O st. sym. imide), 1717(C=O st. acid), 1673(C=O st. amide), 1622(C-C st. aromatic), 1584(C=C st. furan ring), 1464(COO^- st. sym.), 1245, 1160, 1065(furan in-plane CH deformation), 1043(C–O st.), 914, 875, 730(furan C–H out-of-plane deformation/C=O bending); Elemental analysis calculated for $C_{63}H_{54}N_4O_{20}$: C 63.74, H 4.58, N 4.72%; found: C 63.68, H 4.53, N 4.69%; Degree of Polymerization (Dp): 7; Number average molecular weight (Mn): 8300±60.

DAA2: Yield 75%; Color light brown; Empirical Wt. 1159.06g; IR (KBr, cm^{-1}): 3523(-OH st. acid), 3242(-NH st. amide), 3115(C-H st. furan ring), 3030(-CH st. aromatic ring), 2954, 2846(-CH st. aliphatic), 1780(C=O st. asym. imide), 1736(C=O st. sym. imide), 1713(C=O st. acid), 1670(C=O st. amide), 1623(C-C st. aromatic), 1589(C=C st. furan ring), 1460(COO^- st. sym.), 1240, 1163, 1062(furan in-plane CH deformation), 1045(C–O st.), 913, 870, 734(furan C–H out-of-plane deformation/C=O bending); Elemental analysis calculated for $C_{61}H_{50}N_4O_{20}$: C 63.21, H 4.35, N 4.83%; found: C 63.14, H 4.30, N 4.78%; Degree of Polymerization (Dp): 7; Number average molecular weight (Mn): 8102±60.

Synthesis of polyimides (Aromatization and Imidization of DA Adducts) PI1-2

1 g dried sample of the Diels–Alder adducts (DAA1-2) was refluxed with 1 mL of acetic anhydride for 4 hours at 150–160°C. The resulting solution was then cooled and poured into a large volume of water. The precipitates formed were filtered, washed, and air-dried. The brown-colored precipitates obtained, which were aromatized and imidized, were designated as polyimides (PI1-2). These polyimides were characterized by elemental analysis, IR spectroscopy, and thermal analysis.

PI1: Yield 65%; Color brown; Empirical Wt. 1115.06g; IR (KBr, cm^{-1}): 3040(-CH st. aromatic ring), 2960, 2857(-CH st. aliphatic), 1784(C=O st. asym. imide), 1727 (C=O st. sym. imide), 1506(backbone st. aromatic ring), 1375(imide, imide ring vibration, axial); Elemental analysis calculated for $C_{63}H_{46}N_4O_{16}$: C 67.86, H 4.16, N 5.02%; found: C 67.81, H 4.12, N 4.97%.

PI2: Yield 68%; Color brown; Empirical Wt. 1087.00g; IR (KBr, cm^{-1}): 3045(-CH st. aromatic ring), 2958, 2853(-CH st. aliphatic), 1783(C=O st. asym. imide), 1725(C=O st. sym. imide), 1503(backbone st. aromatic ring), 1373(imide, imide ring vibration, axial); Elemental analysis calculated for $C_{61}H_{42}N_4O_{16}$: C 67.40, H 3.89, N 5.15%; found: C 67.36, H 3.85, N 5.10%.

Preparation of Glass Fiber Reinforced Composites (GFRC1-2)

Glass fiber reinforced composites (GFRC1-2) were prepared as follows: Suspensions of bisfuran (BF) and bismaleimides (BMI1-2) in THF were prepared on a weight basis and stirred well for 10 minutes. The suspensions were then applied using a brush to polyimide-compatible glass cloth, and the solvent was allowed to evaporate for 10–20 minutes at room temperature. The dried prepregs were stacked one over another and placed between stainless steel plates. The stack was then compressed under a pressure of 60–70 psi at 150°C for 10 hours in an air-circulating oven. After the curing process, the composites were cooled to room temperature before the pressure was released. The composites were subsequently machined to the desired final dimensions for various mechanical and chemical tests.

4. Conclusions

The synthesis of thermoplast-thermoset merged segment polyimides (PI1-2) through Diels-Alder polymerization presents a novel strategy for developing advanced polymer materials with tailored properties. The 'in situ' produced polyimides exhibit excellent adhesion to glass fibers, making them ideal for reinforcing composites. These glass fiber-reinforced composites (GFRCs) demonstrate outstanding mechanical properties, including high compressive strength, flexural strength, and impact resistance, as well as impressive electrical properties, such as high dielectric strength. Additionally, they show strong resistance to a wide range of organic solvents and mineral acids, ensuring their durability in harsh environments. The combination of superior mechanical, electrical, and chemical resistance makes these materials highly suitable for demanding applications in industries such as aerospace, automotive, electronics, and other high-performance sectors. The unique properties of the thermoplast-thermoset polyimide system allow for the design of composite materials that can withstand extreme conditions while maintaining structural integrity and functionality. Further research is needed to optimize the molecular structure and processing conditions of the polyimide-based composites. This could involve exploring variations in the polymer backbone structure, fiber reinforcement types, and the fine-

tuning of processing parameters such as curing temperature and time, to further enhance the performance characteristics. Such advancements could lead to even more robust and versatile materials with potential for broader applications in advanced engineering and industrial fields.

Author Contributions

Yogesh Patel conducted the research during his Ph.D. tenure. Ami Patel provided critical feedback, guided the structure of the review, and prepared the manuscript for publication. Both authors discussed the findings in detail, contributed to writing, and finalized the manuscript.

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