



Thymol-inspired benzoxazines: A bio-based route to multifunctional high-performance polymers

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ABSTRACT

In this work, bio-based bisphenol (TP) was synthesized from thymol and 4-phenoxybenzaldehyde. On utilizing TP, structurally different benzoxazines were synthesized using four different amines namely furfurylamine (ff), p-aminophenol (pa), tyramine (ty) and p-toluidine (pt) through Mannich condensation. The molecular structure of developed benzoxazines was confirmed by various spectroscopic analyzes. Among the benzoxazine monomers, TP-pa exhibited the lowest curing temperature of 213°C. TP-pt showed highest antimicrobial activity with maximum inhibition zones of 17 mm for *E. coli* and 25 mm for *S. aureus*. Thermal analysis revealed that poly(TP-ty) showed highest thermal stability with char yield of 46% at 850°C, limiting oxygen index value of 35 and heat resistance index up to 208°C. Among the polybenzoxazines, poly(TP-ty) exhibited the highest water contact angle (WCA) value of 146°. All the polybenzoxazines coated over MS specimen demonstrated corrosion inhibition efficiency of 99%, which further validated by density functional theory (DFT) studies. Flexural test revealed that a maximum flexural stress of 1.62 MPa and modulus of 451.69 MPa for thermally cured material.

1. Introduction

Polybenzoxazines have produced an abundance of attention recently as next-generation thermosetting resins considering their distinctive combination of low water absorption [1], thermal stability [2,3] and adaptable molecular structure. Bio-based monomer synthesis advancements have made them viable substitutes for conventional resins, especially in coatings and adhesive technologies [4]. Molecular engineering techniques have also made it possible to add self-healing [5], flame-retardant [6], corrosion resistance [7] and antibacterial qualities [8], increasing their significance in high-performance polybenzoxazines and smart materials. These advancements highlight the increasing significance of polybenzoxazines in advanced functional applications as well as sustainable material innovation.

Renewable feedstocks including lignin [9,10], cardanol [11], eugenol [12], magnolol [13], curcumin [14], guaiacol [15] and sesamol [16,17] are used to develop bio-based benzoxazines for diversified applications. This change lessens dependency on fossil fuels and supports international initiatives for environmentally friendly materials [18]. According to recent research, bio-based polybenzoxazines are great options for adhesive coatings as a consequence of their longevity,

chemical resistance [19] and good adherence [20]. They are appropriate for electronics [21,22] and aerospace applications [23] due to their significant temperature stability and minimal curing shrinkage. Eco-friendly synthesis methods, by adopting green chemistry principles to synthesize bio-based bisphenols from renewable raw materials to develop benzoxazines showed least toxicity and environmental impact are the focus of recent investigations [24]. As a result of their distinct photoluminescent activity, hydrophobicity and anticorrosion capabilities, thymol-derived benzoxazines are appropriate for coatings and protective applications in harsh conditions [25].

Utilizing bisphenol-type precursors to prepare benzoxazine monomers have been effectively carried out using thymol, a naturally occurring phenolic molecule produced from plants with an aldehyde. This supports sustainability objectives and lessens dependency on bisphenols manufactured from petroleum products. Emergence of low-toxicity, renewable thermosets by using thymol-based bisphenols, supporting the larger trend of green polymer chemistry and circular material design. 4-phenoxybenzaldehyde is chosen for its capacity to provide stability, aromatic reinforcement and functional diversity. In order to generate more sustainable bisphenols, thymol and 4-phenoxybenzaldehyde, a bio-based monoterpenoid were utilized. By systematically

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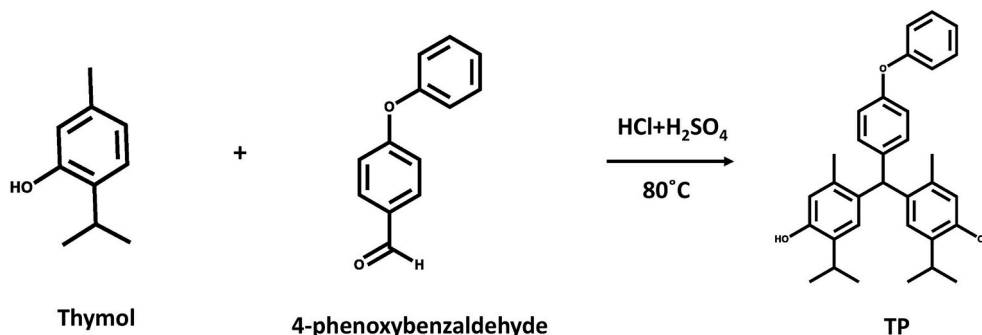
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Scheme 1. Synthesis of bio-based bisphenol TP.

varying the amine precursors, the work demonstrates how molecular design directly influences curing behavior, antimicrobial performance, thermal stability, flame retardancy, hydrophobicity, mechanical strength and combined with the corrosion inhibition efficiency of all coatings, highlight the multifunctional potential of sustainable polybenzoxazines. This integration of bio-based feedstocks with enhanced performance measures and DFT-validated corrosion protection has advanced significant for the development of next-generation, environmentally friendly polymer systems for high-performance applications.

In this direction, the current study utilized bio-based thymol to synthesis a new kind of bisphenol (TP). The benzoxazine was prepared by reacting bisphenol (TP) with different types of amine compounds such as furfurylamine (ff), p-aminophenol (pa), tyramine (ty) and p-toluidine (pt) through Mannich condensation. The molecular structure of developed benzoxazines was confirmed by HRMS, ATR-FTIR, ^1H NMR and ^{13}C NMR spectroscopic techniques. The polymerization of benzoxazines was assessed by DSC, while thermal stability was monitored by TGA analysis. Using the well diffusion technique, the antibacterial activity of benzoxazines was assessed against *S. aureus* and *E. coli*. The hydrophobic nature of the polybenzoxazines was tested by measurements of the water contact angle values. Corrosion resistant properties of polybenzoxazine was studied by using electrochemical impedance spectroscopic (EIS) and potentiodynamic polarization (PDP) techniques, which is additionally validated by DFT computational studies. Flexural test was carried out for the polybenzoxazines and the various characterization results were compared in relation to the different findings from the manifold investigations.

2. Experimental section

2.1. Materials

4-phenoxybenzaldehyde (97%) was sourced from BLD Pharma, India. Sulphuric acid (98%) and hydrochloric acid (98%) were purchased from Thermo-Fischer India. Paraformaldehyde (96%) was received from Sigma-Aldrich, India. P-aminophenol (98%) was purchased from ARRO Biochem, Mumbai. Tyramine (98%) was provided from Chemscone, India. Thymol (99%), furfurylamine (98%) and p-toluidine (99%) were purchased from SRL Pvt. Ltd, Mumbai, India. The solvents 1,4-dioxane (99%) and ethyl acetate (99%) were provided by Molychem, India.

2.2. Characterization techniques

High-resolution mass spectrometry (HRMS) was employed to determine the molecular mass of the synthesized compounds using a WATERS-XEVO G2-XS-QTOF instrument. Fourier-transform infrared (FTIR) spectra were recorded on a Shimadzu IR-Affinity-1S spectrophotometer equipped with a Miracle10 single-reflectance attenuated total reflection (ATR) accessory. The ^1H and ^{13}C NMR spectra of the synthesized bisphenol and monomers were obtained using a Bruker

500 MHz spectrometer, with deuterated chloroform (CDCl_3) as the solvent and tetramethylsilane (TMS) as the internal reference. Differential scanning calorimetry (DSC) analysis was conducted on a NETZSCH DSC 3500 under a nitrogen atmosphere (60 mL/min) at a heating rate of $10^{\circ}\text{C}/\text{min}$. Thermogravimetric analysis (TGA) was performed using a NETZSCH STA 2500, with 5 mg of sample heated under nitrogen flow (250 mL/min) at a heating rate of $20^{\circ}\text{C}/\text{min}$. The water contact angle was measured using a Kyowa goniometer, applying $5\ \mu\text{L}$ water droplets to each sample, with ten measurements averaged per sample. Surface morphology was examined via scanning electron microscopy (SEM) using a Carl Zeiss Sigma instrument equipped with a Gemini column, offering a resolution of 1.5 nm. Density functional theory studies were performed on Gaussian 16 software using semi empirical method where the preoptimized structures were generated using Gauss view. Antimicrobial activity was measured by well diffusion method and ISO 20743: 2021 absorption method. Corrosion resistance was assessed through open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) techniques. These tests were conducted in a 3.5% sodium chloride (NaCl) aqueous solution using a platinum wire as the counter electrode, Ag/AgCl electrode as the reference and the solution itself as the electrolyte. For PDP analysis, the potential was swept at a rate of $1\ \text{mV}/\text{s}$, ranging from 100 mV below to 100 mV above the corrosion potential (E_{corr}), which corresponds to the steady-state OCP. During EIS measurements, a 50 mV AC perturbation was applied across a frequency spectrum from 100 kHz down to 10 mHz.

2.3. Synthesis of bisphenol (TP)

Thymol (6 g/0.04 mol) was stirred with the acid mixture of HCl and H_2SO_4 of 2:1 ratio in a round-bottomed flask. The reaction mixture was agitated at 60°C for 30 min. Subsequently, (13.7 ml/0.02 mol) of 4-phenoxybenzaldehyde was added slowly to the reaction mixture and stirred at 80°C for 24 h. Using hexane and ethyl acetate of 9:1 ratio as the mobile phase, thin layer chromatography (TLC) was used to assess the progress and completion of the reaction. Upon completion the obtained product was neutralized with water and extracted using ethyl acetate, then dried in a vacuum oven and the desired product bisphenol (TP) was obtained with 82% yield (Scheme 1).

2.4. Synthesis of benzoxazine monomers (TP-bz)

By adding (2 g/0.004 mol) of bisphenol-TP and furfurylamine (0.79 ml/0.008 mol) in 1,4-dioxane and paraformaldehyde (0.4 g/0.016 mol) was added alongside in a 100 mL round-bottomed flask fitted with a magnetic stirrer and condenser. The reactant was subsequently heated up to 110°C for 24 h with constant stirring. The completion of the reaction was verified by TLC. The reaction mixture was diluted in ethyl acetate and then repeatedly washed with distilled water and saturated NaHCO_3 solution to get a transparent organic layer. The organic layer was evaporated using rotatory evaporation under low pressure. The recovered product, which is referred as TP-ff. By following same

small bending loads prior to irreversible deformation. The automated Young's modulus of 451.69 MPa emphasizes how cured TP-pa, aromatic backbone contributes stiffness and rigidity. However, the specimen responsive mechanical response is due to the comparatively modest maximum force (0.01 kN) and flexural strain at yield (0.65%). Although the material efficiently resists deformation due to its hard aromatic structure, it cracks at a lower strain than more flexible materials. Overall, the flexural study of polymerized TP-pa show that it provides a stiff but moderately strong matrix, appropriate for applications where rigidity and dimensional stability are more important than high-load structural conditions.

4. Conclusion

Bisphenol (TP) was successfully synthesized from thymol and 4-phenoxybenzaldehyde. Bisphenol was utilized to prepare bifunctional benzoxazines. The synthesized bisphenol and benzoxazine monomers were successfully confirmed by HRMS, ATR-FTIR, ^1H NMR and ^{13}C NMR. The lowest polymerization temperature was 213°C for TP-pa. TP-pt exhibited better antimicrobial activity with an inhibition zone of 17 and 25 mm against *E. coli* and *S. aureus*. Poly(TP-ty) was found to be the most thermally stable polymer with a highest char yield of 46%, LOI (35) and HRI (208°C). Poly(TP-ty) exhibited the maximum WCA value of 146° . All the polybenzoxazines exhibited superior resistance to the corrosion with a corrosion inhibition efficiency of 99%. According to mechanical studies, polymerized TP-pa exhibits the highest flexural stress (1.62 MPa). These results suggested the sustainable benzoxazines were developed to focused on demonstrating strong adhesion and resistance to aggressive environments. To advance next-generation sustainable technologies, this research will pioneer eco-friendly synthesis routes, polymerization kinetics and scalable processing.

CRediT authorship contribution statement

Mythili Settu: Investigation, Methodology, Writing – original draft. **Balaji Krishnasamy:** Conceptualization, Investigation, Resources, Supervision, Validation, Writing – review & editing. **Latha Govindraj:** Formal analysis. **Rathika Govindasamy:** Supervision.

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Declaration of competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jics.2026.103030>.

Data availability

Data will be made available on request.

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