



Oxidative polycondensation of phenylhydrazine-based Schiff base: structural, thermal, optical and dielectric investigations

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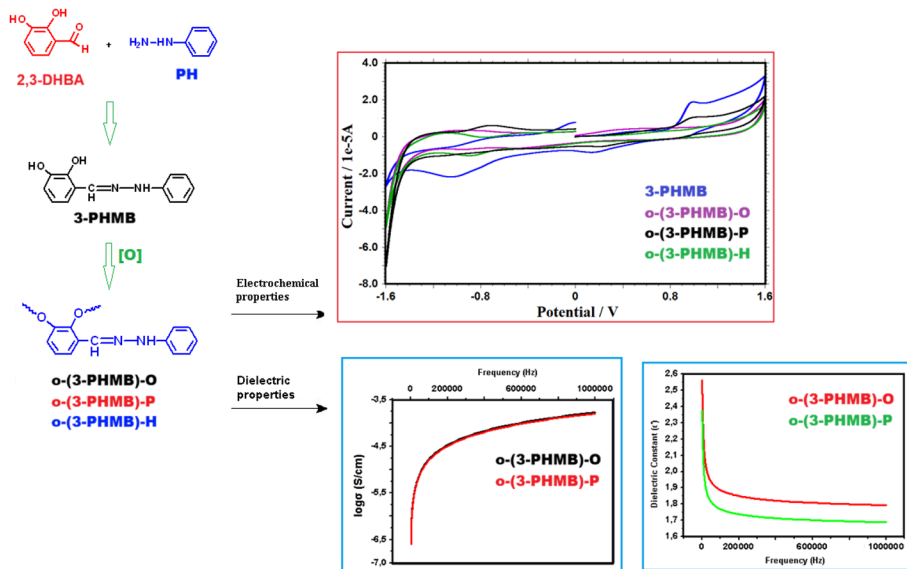
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Abstract

In this study, the Schiff base 3-((2-phenylhydrazono)methyl)benzene-1,2-diol (3-PHMB) was synthesized through a condensation reaction between 2,3-dihydroxybenzaldehyde (2,3-DHBA) and phenylhydrazine (PH). The obtained Schiff base was subsequently subjected to oxidative polymerization in a 10% KOH (w/v) aqueous medium using hydrogen peroxide (35% H₂O₂), sodium hypochlorite (6–14% NaOCl), and oxygen (O₂) as oxidants, yielding the oligomers o-(3-PHMB)-P, o-(3-PHMB)-H, and o-(3-PHMB)-O. The structural, morphological, thermal, optical, and electrochemical characteristics of the synthesized compounds were comprehensively investigated using FT-IR, NMR, SEM, TG-DTA, UV-Vis, CV, and GPC techniques. UV-Vis and CV analyses revealed that the optical band gaps (E_g) were 2.33 eV for 3-PHMB, 2.06 eV for o-(3-PHMB)-P, 2.16 eV for o-(3-PHMB)-H, and 2.02 eV for o-(3-PHMB)-O, while the electrochemical band gaps (E_g') were calculated as 2.03 eV, 1.65 eV, 1.63 eV, and 1.50 eV, respectively. Thermal analysis showed that the char yields at 1000 °C were 41.43% for o-(3-PHMB)-P, 38.72% for o-(3-PHMB)-H, and 44.35% for o-(3-PHMB)-O. Dielectric measurements indicated that o-(3-PHMB)-O exhibited higher polarization capacity and energy storage ability compared to the other oligomers. Moreover, the electrical conductivity values increased steadily with frequency; at 100 kHz, the conductivity was recorded as 1.74×10^{-5} S cm⁻¹ for o-(3-PHMB)-O and 1.64×10^{-5} S cm⁻¹ for o-(3-PHMB)-P. These findings demonstrate that the synthesized Schiff base oligomers are promising candidates for dielectric and optoelectronic applications due to their high thermal stability, controlled molecular weight distribution, and semiconducting properties.

Graphical Abstract



Keywords Phenylhydrazine-based oligomers · Oxidative polymerization · Dielectric properties · Optoelectronic applications

Introduction

Schiff bases are compounds formed by the condensation reactions of aldehydes or ketones with primary amines, and they possess a characteristic C=N bond in their structure. These compounds were first synthesized by the German chemist Hugo Schiff and are also known as imine or azomethine compounds due to their structural properties. Today, Schiff bases and their derivatives have become a significant research topic, particularly in biomedical applications and materials science, due to their catalytic properties [1]. In addition to their use in pigment and dye production, as intermediates in organic synthesis, and as polymer stabilizers, they also attract considerable scientific interest due to their broad biological activity profile [2–4]. Phenylhydrazine is an ammonia-soluble compound that can cause toxic effects by influencing metabolic processes in biological systems due to its oxidation sensitivity and xenobiotic structure [5]. Phenylhydrazine and its derivatives are important compounds that facilitate the synthesis of Schiff base structures by readily condensing with aldehydes and ketones thanks to their reactive –NH–NH– functional group. In the literature, these structures are mostly discussed at the monomeric level, focusing on detailed molecular characterizations [6]. In contrast, the conversion of phenylhydrazine-based Schiff bases into oligomeric systems and the investigation of their associated thermal and dielectric properties have been reported in a limited number of studies [7]. Furthermore, it has been shown that phenylhydrazine-containing

oligomeric and polymeric Schiff bases offer potential for functional applications such as adsorption, antibacterial, and antifungal properties [8, 9]. This study aims to synthesize phenylhydrazine-based Schiff base structures at the oligomeric level and to systematically determine the structure-property relationships.

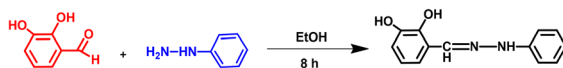
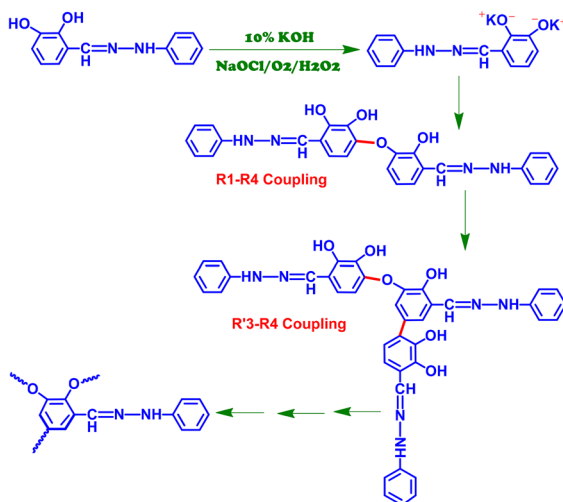
Chemical oxidative polymerization is a common polymerization technique performed in the presence of strong oxidants, and the literature indicates that fundamental properties of polymers, such as solubility, thermal stability, and conductivity, vary depending on the type of oxidant used [10–12]. Oxidative polymerization methods, generally performed with oxidants such as NaOCl, H₂O₂, and O₂, allow the use of inexpensive and readily available oxidants, their easy removal from the reaction medium, and the production of high molecular weight, thermally stable polymers [13]. Electrical conductivity, on the other hand, varies depending on parameters such as time and temperature and is also related to the water and moisture content during heating of the sample [14]. Polymers are used in numerous electronic applications thanks to their electrical conductivity properties [15–17].

3-PHMB Schiff base was synthesized as a result of the condensation reaction between 2,3-dihydroxybenzaldehyde and phenylhydrazine. Subsequently, this monomer was polymerized in aqueous basic medium by H₂O₂, NaOCl, and O₂ oxidants and o-(3-PHMB)-P, o-(3-PHMB)-H, and o-(3-PHMB)-O oligomers were obtained. Here, the suffixes “P”, “H”, and “O” represent the oxidants used during oxidative polymerization, namely hydrogen peroxide (H₂O₂), sodium hypochlorite (NaOCl), and oxygen (O₂), respectively. These abbreviations are used throughout the manuscript to clearly distinguish the oligomers synthesized with different oxidizing agents. The structural, thermal and optoelectronic properties of the synthesized monomer and oligomers were investigated in detail using advanced characterization methods such as FT-IR, ¹H-NMR, ¹³C-NMR, UV-Vis, CV and TG-DTA. The findings revealed that the type of oxidant used plays a decisive role in the properties of the products and that these oligomers have significant potential as functional materials. Also, the molecular weight, *T_g*, surface images and dielectric properties of oligomers were determined GPC, DSC, SEM and electrometer measurements, respectively.

Materials and methods

Materials

Potassium hydroxide (KOH), diethyl ether, ethanol, ethyl acetate, acetone, N, N'-dimethylformamide (DMF), methanol, dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), chloroform (CHCl₃), hexane, hydrogen peroxide (H₂O₂, 35% aqueous solution), hydrogen chloride (HCl) (37%), acetonitrile, 2,3-dihydroxybenzaldehyde (2,3-DHBA), phenylhydrazine (PH), sodium hypochlorite (NaOCl, 6–14% aqueous solution) and dichloromethane (CH₂Cl₂) were all utilized as supplied by Merck Chemical Co. (Germany).

Scheme 1 Synthesis of 3-PHMB**Scheme 2** Synthesis of 3-PHMB oligomers

Synthesis of Schiff base (3-PHMB)

The methods described in the literature were used to synthesize 3-((2-phenylhydrazono)methyl)benzene-1,2-diol [18–20]. 10 mmol (1.36 g) of 2,3-dihydroxybenzaldehyde was dissolved in 15 mL of ethanol to start the synthesis. After dissolving phenylhydrazine (10 mmol, 1.2 g) in 15 mL of ethanol, it was added to the main mixture and allowed to react for 8 h at room temperature. Thin layer chromatography (TLC) was used when the reaction was finished. After the reaction was finished, 15 mL of distilled water were added to precipitate the result, which was then vacuum-filtered. 10 mL of cold ethanol and 20 mL of distilled water were used to wash the crude product in order to purify it. The purifying process produced a light gray solid product. The finished product was dried vacuum oven at 50 °C 24 h. These synthesis procedures led to an 85% yield of the target chemical being extracted. Scheme 1 provides a summary of the synthesis's overall plan.

Synthesis of oligomers (o-(3-PHMB)-O, o-(3-PHMB)-P, o-(3-PHMB)-H)

The oxidative polycondensation of 3-PHMB with O_2 as an oxidant in a 10% KOH (w/v) aqueous solution medium produced the o-(3-PHMB)-O oligomer, and Scheme 2 shows the reaction schematically. 30 mL of a 10% (w/v) KOH aqueous solution and 3-PHMB (0.229 g, 1.0 mmol) were added to a 100 mL two-mouth round bottom flask that was equipped with a thermometer and condenser. Following the completion of dissolution, O_2 gas was added to the colorant-coated medium, and the system was covered with colorant. A magnetic stirrer was used to keep the reaction mixture at 50 °C for ten hours [21, 22]. When the reaction was finished, the solution's hue changed to black. After cooling to ambient temperature, 10% HCl was added to

neutralize the solution. Distilled water (5×50 mL) was used to filter and wash the reaction mixture. In a vacuum oven, the finished product - a black solid - was dried at 50°C 24 h. The yield of o-(3-PHMB)-O was calculated as 78%.

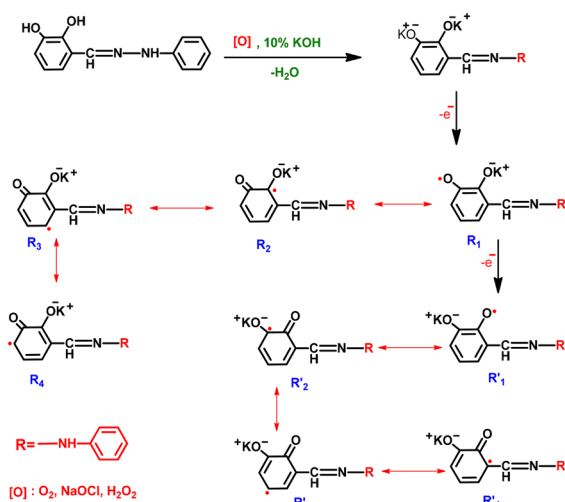
1 mmol (0.229 g) of 3-PHMB was placed in separate two-mouth round bottom flask. Then, 30 mL of 10% KOH (w/v) solution was added to each flask, and after the 3-PHMB dissolved, 6–14% NaOCl and 35% H_2O_2 aqueous solutions as oxidants were added separately to the reaction media. The mixtures were stirred under reflux at 60°C for ten hours. The purification process was carried out as described above. With yields of 70% and 83%, respectively, oligomers were created as o-(3-PHMB)-H and o-(3-PHMB)-P using NaOCl and H_2O_2 as oxidants. Scheme 2 illustrates how the oligomers synthesize when O_2 , NaOCl, and H_2O_2 are present in alkaline media.

The general resonance forms of radicals generated during the oxidative polymerization process and their possible coupling mechanisms are presented in Scheme 3. Although it is predicted that the different oxidizers used in the experiments (O_2 , NaOCl and H_2O_2) will exhibit similar behavior in basic radical production and coupling pathways, the preferred type of oxidizer has a decisive effect on the reaction kinetics and the final oligomeric structure.

Measurements

A Perkin Elmer FT-IR/FIR spectrometer Frontier (USA) equipped with PIKE technologies Gladi Attenuated Total Reflection (ATR) accessory was used to record the FT-IR spectra of the compounds in the wavelength range of $4000\text{--}400\text{ cm}^{-1}$ with a discrimination power of 4 cm^{-1} . The ^1H -NMR (600 MHz) and ^{13}C -NMR (150 MHz) spectra were obtained using an Agilent Technologies (USA) device with $\text{DMSO-}d_6$ as a solvent and TMS as an internal standard. Thermal evaluations of 3-PHMB, oligo-(3-PHMB)-H, oligo-(3-PHMB)-P and oligo-(3-PHMB)-O were performed using a PerkinElmer Diamond Thermal Analysis TG-DTA and PerkinElmer Sapphire differential scanning calorimetry (DSC). The TG-DTA analysis was carried out between

Scheme 3 Resonance formation mechanisms of 3-PHMB radical



25 and 1000 °C with a heating rate of 10 °C min⁻¹ (flow rate: 200 mL min⁻¹), and the DSC measurements were carried out between 25 and 300 °C with the same heating rate in a nitrogen environment (flow rate: 100 mL min⁻¹). The weight average molecular weight (M_w), number average molecular weight (M_n), peak average molecular weight (M_p), and Z-average molecular weight (M_z) and polydispersity (PDI) values of oligomers were determined with a Malvern Viscotek GPC Dual 270 max system for Gel Permeation Chromatography-Light Scattering (GPC-LS) analysis. A CHI 660 C electrochemical work station (CH Instrument, Austin, Texas, USA) with a three-electrode system—a glassy carbon electrode as the working electrode, a platinum wire as the counter electrode, and a silver wire as the reference electrode—was used to record the cyclic voltammetric curves of compounds. UV-Vis spectra were captured with an AnalytikJena Specord 210 Plus double-beam spectrophotometer in the 280–800 nm range. The Zeiss Gemini SEM 360 instrument (Germany) was used for imaging using Field Emission Scanning Electron Microscopy (FE-SEM). A QuadTech 7600 accuracy LCR meter impedance analyzer operating in the 50 Hz–1 MHz band was used to determine the dielectric characteristics of the oligomers. The monomer and oligomer samples were first sputter coated with gold using a Quadrum coating apparatus at 8×10^{-1} mbar Pa⁻¹ vacuum with a 10 mA applied current before imaging. Dielectric parameters of o-(3-PHMB)-P and o-(3-PHMB)-O were measured by Fytronix FY-FP-9000 model electrical conductivity system on Fytronix probe holder at room temperature.

Results and discussion

The structures of compounds

The Schiff base coded 3-PHMB's FT-IR spectrum shows the following functional groups, as shown in Fig. 1: 846 (olefinic -C=C- out-of-plane bending vibration), 1515 and 1443 (C=C stretching) 1596 (-C=N stretching vibration), 2973 and 2949 (aliphatic C-H stretching), 3048 and 3034 cm⁻¹ (aromatic C-H stretching vibration), 3326 (-NH stretching vibration) and 3427 (-OH stretching vibration). The distinctive amine peaks vanished once the Schiff base (3-PHMB) developed, and the azomethine (-N=CH-) group absorption band arose in the 1590–1600 cm⁻¹ region of the FT-IR spectrum [23]. Figure 1 displays the FT-IR spectra of the o-(3-PHMB)-P, o-(3-PHMB)-H, and o-(3-PHMB)-O samples. The FT-IR spectra of the oligomers were discovered to be broader than those of the monomers. Aliphatic C-H stretching vibrations for o-(3-PHMB)-P, o-(3-PHMB)-H, and o-(3-PHMB)-O are represented by the bands at 2910, 2908, and 2907 cm⁻¹, respectively [24]. The presence of C-N stretching vibrations is confirmed by the signals at 1597, 1597, and 1594 cm⁻¹. The C-C stretching vibrations are indicated by the bands at 1494, 1495, and 1496 cm⁻¹. Furthermore, it was determined that the bands at 1070, 1072, and 1071 cm⁻¹ were C-O-C vibrations [24]. For o-(3-PHMB)-P, o-(3-PHMB)-H, and o-(3-PHMB)-O, the bands seen at 3057, 3061, and 2961 cm⁻¹ correspond to aromatic C-H stretching vibrations, respectively. Accordingly, bands of NH stretching vibration were found at 3210, 3205, and 3207 cm⁻¹ [25].

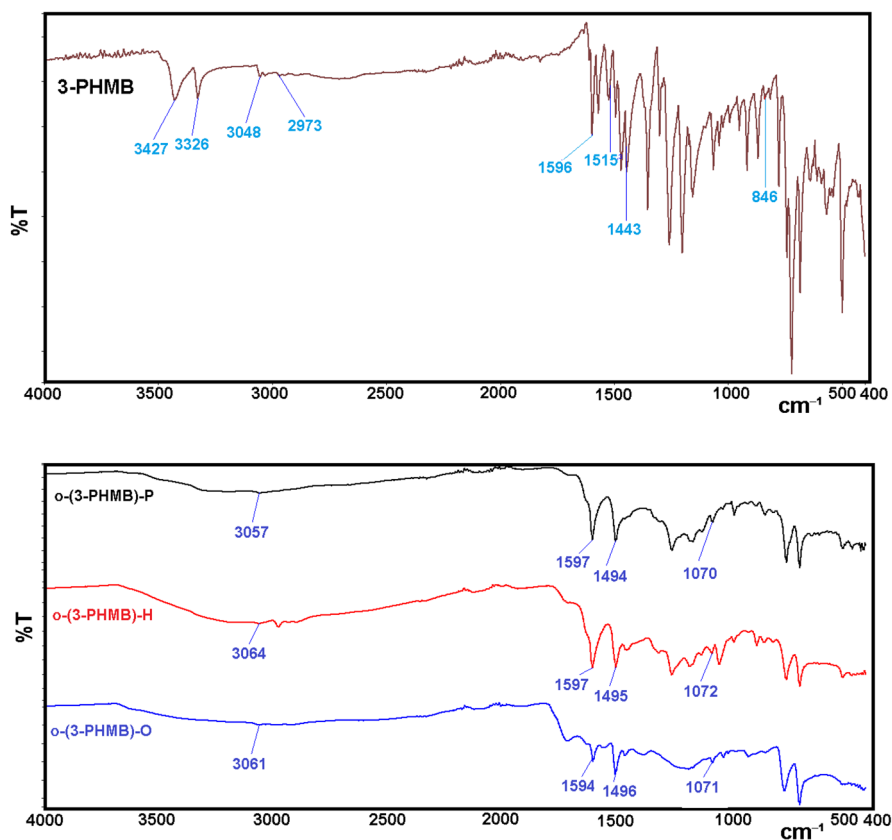


Fig. 1 FT-IR spectra of (3-PHMB) and o-(3-PHMB)-O, o-(3-PHMB)-P, and o-(3-PHMB)-H

By employing DMSO- d_6 solvent for ^1H and ^{13}C -NMR spectroscopic investigations, the molecular structure of 3-PHMB was clarified. Figure 2 displays the ^1H and ^{13}C -NMR spectra of 3-PHMB, and a comparison analysis was conducted. In the ^1H -NMR spectra of 3-PHMB (DMSO- d_6), the chemical shifts δ (ppm) values were ascertained as follows: 10.36 (s, 1H, -NH), 10.30 (s, 1H, -OH2), 9.22 (s, 1H, -OH1), 8.09 (s, 1H, -CH=N), 7.22 (d, 2H, Ar-Hd), 6.94 (t, 2H, Ar-He), 6.92 (t, 1H, Ar-Hf), 6.74 (d, 1H, Ar-Hc), 6.72 (d, 1H, Ar-Ha), and 6.67 (s, 1H, Ar-Hb). Schiff base production is demonstrated by the elimination of the peak unique to the aldehyde proton (H-C=O) and the detection of the signal associated with the imine proton (-CH=N-) at 8.09 ppm in the spectrum [26]. Furthermore, the value of δ (ppm) in the ^{13}C -NMR spectrum (DMSO- d_6) of 3-PHMB was found to be 145.72 (C7), 145.03 (C1), 144.67 (C2), 138.95 (C8), 129.77 (C9), 120.91 (C5), 119.68 (C4), 119.52 (C11), 118.72 (C6), 115.97 (C3), and 112.10 (C10). These results show the successful synthesis of the Schiff base structure in accordance with the values reported in the literature.

Figure 3 displays the o-(3-PHMB)-O compound's ^1H -NMR spectrum (DMSO- d_6). The following is a determination of the proton signals (δ , ppm) seen in the spectrum: 12.17 (s, 1H, -NH), 10.69 (s, 1H, -OH1), 10.27 (s, 1H, -OH2), 9.52 (s, 1H, -CH=N),

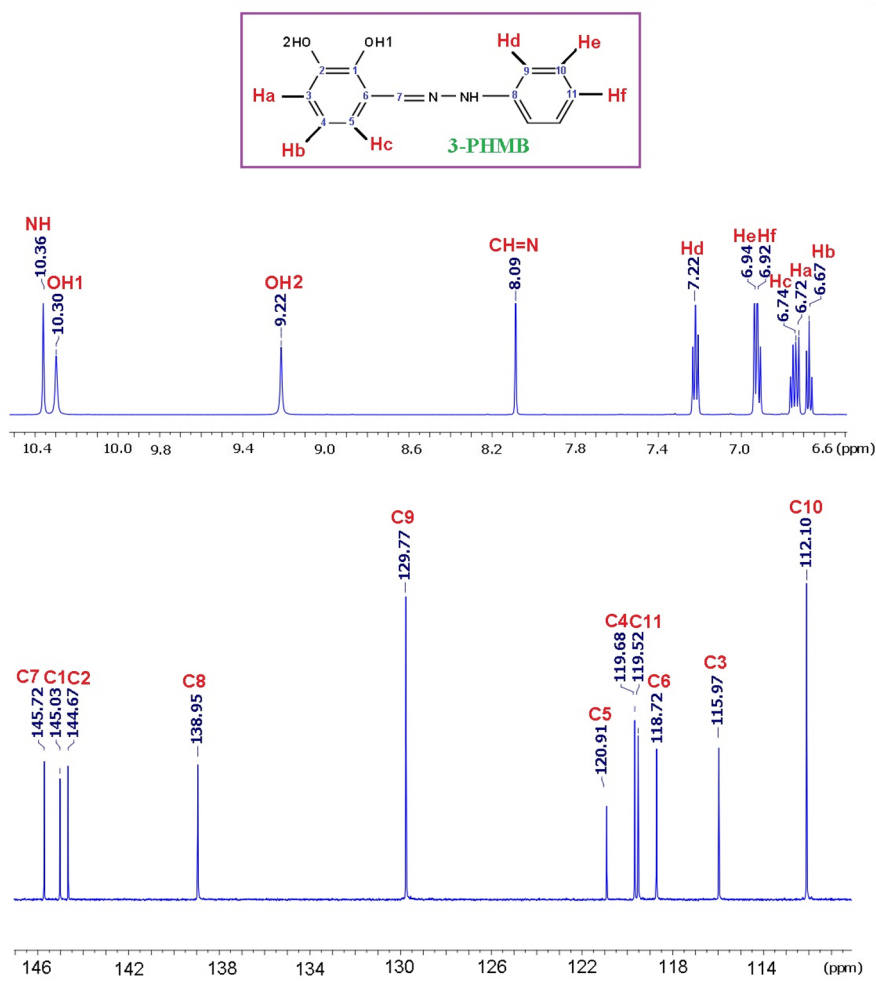


Fig. 2 ^1H -NMR and ^{13}C -NMR spectra of 3-PHMB

7.43 (d, 1H, Ar-Hc), 7.34 (t, 2 H, Ar-He), 7.19 (d, 2 H, Ar-Hd), 7.09 (d, 1H, Ar-Hb), 6.99 (t, 1H, Ar-Hf), and 6.30 (s, 1H, terminal proton). Specifically, the signals provided by the -OH1 and -OH2 protons at 10.69 and 10.27 ppm, respectively, show that the polymerization procedure was successful and the hydroxyl groups in the oligomer structure were protected. Furthermore, the following chemical shifts (δ , ppm) were assigned to the α -(3-PHMB)-O ^{13}C -NMR spectrum (DMSO- d_6): 171.20 (C7), 164.36 (Ar-C1-ipso), 150.47 (Ar-C2-ipso), 144.74 (Ar-C8-ipso), 134.29 (Ar-C5), 129.90 (Ar-C9), 128.02 (Ar-C11), 127.05 (Ar-C4), 124.74 (Ar-C6-ipso), 114.71 (C3), and 110.56 (Ar-C10).

As seen from the resonance structures in Scheme 3, C-O coupling occurs between R1-R3, R1-R4 or R'1-R'3 radicals. Also, C-C couplings were occurred between R3-R3, R3-R4, R4-R4 or R'3-R'3 radicals. Due to steric hindrance, bonding does not

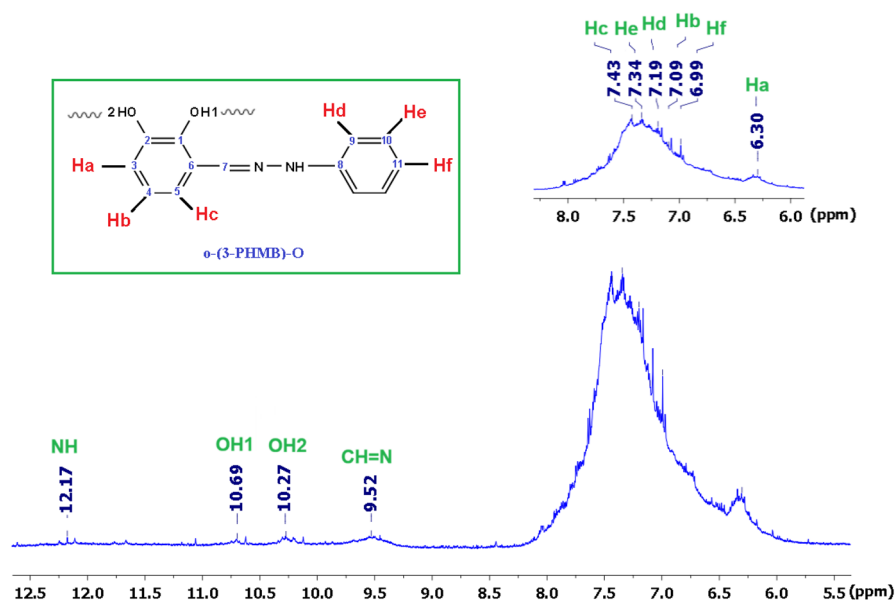


Fig. 3 ^1H -NMR spectra of *o*-(3-PHMB)-O

occur with the R₂, R'₂, and R'₄ resonance structures. Based on the proton integration of the -OH groups in ^1H NMR, the C-O coupling ratios for -OH1 and -OH2 were calculated as 40% and 60%, respectively. The lower initial decomposition temperatures of the oligomers than the monomer indicate that the chains are formed primarily by C-O bonding.

Thermal characterization of 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P, *o*-(3-PHMB)-H)

The TG and DTG curves of the synthesized 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P and *o*-(3-PHMB)-H are shown in Fig. 4. Thermal data (TG-DTG) are summarized in Table 1. Thermal analysis results reveal that the thermal stability of oligomers is directly related to the molecular structure, chain organization, and the chemical character of the oxidizing agent used during oligomerization. The initial mass loss observed in the thermograms up to 120 °C corresponds to the desorption of water and organic solvent from the samples, and these losses were found to be 2.3, 1.3 and 1.7% for *o*-(3-PHMB)-O, *o*-(3-PHMB)-P and *o*-(3-PHMB)-H, respectively. These low ratios indicate that the oligomers exhibit relatively compact and well-organized structures. The initial temperatures of 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P and *o*-(3-PHMB)-H were determined as 240, 165, 181 and 176 °C, respectively. The recorded temperatures for 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P, and *o*-(3-PHMB)-H were 228, 205, 203, and 217 °C, respectively, which corresponded to a 5% weight loss (T_5). The temperatures corresponding to 10% weight loss (T_{10}) for 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P and *o*-(3-PHMB)-H are shown as 247, 243, 236 and 258 °C, respectively. The temperatures corresponding to 20% weight loss (T_{20}) are shown as 266, 324, 285, 323 °C for 3-PHMB, *o*-(3-PHMB)-O,

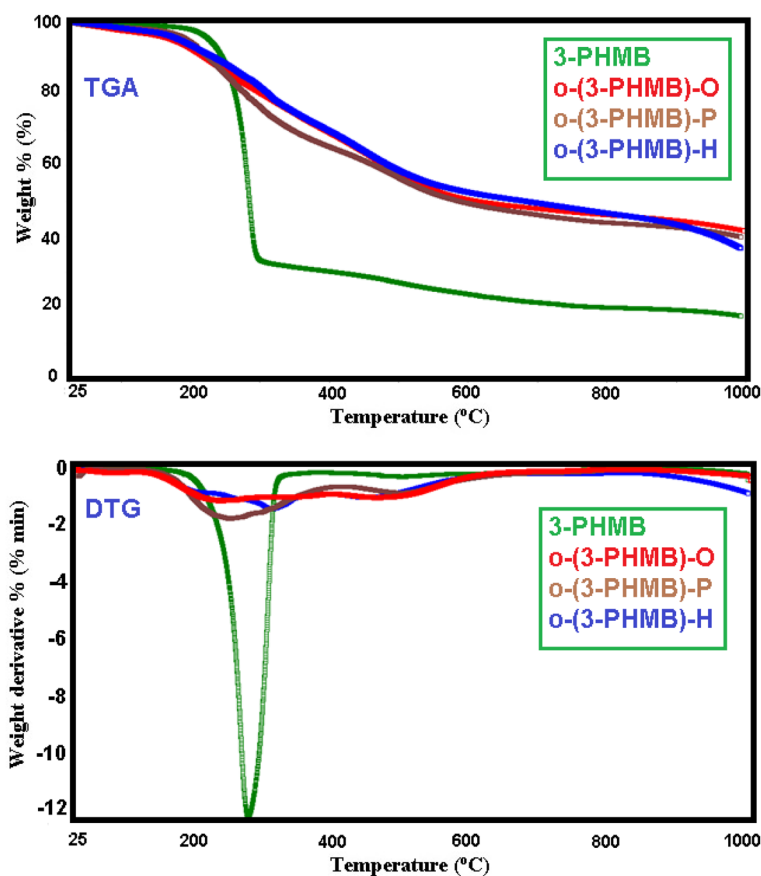


Fig. 4 The thermal curves of 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-P, o-(3-PHMB)-H

o-(3-PHMB)-P and o-(3-PHMB)-H, respectively. In particular, the high T_{20} values of o-(3-PHMB)-O and o-(3-PHMB)-H oligomers indicate that these structures exhibit a higher aromatic content and more limited chain mobility. The temperatures for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-P, and o-(3-PHMB)-H were determined to be 284, 732, 625, and 750 °C, respectively, which corresponded to 50% weight loss (T_{50}). This significant increase can be mechanistically explained by the increase in aromatic ring density with oligomerization, the strengthening of π - π interactions, and the formation of a stable carbon skeleton during thermal decomposition. The char values obtained at 1000 °C were 17.75%, 44.35%, 41.43% and 38.72% for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H, respectively. The observation of the highest char yield in the o-(3-PHMB)-O oligomer indicates that oxidative polymerization with oxygen forms more ordered chain structures and more stable aromatic carbon networks. Thermal decomposition of o-(3-PHMB)-O oligomer showed a three-stage: 27.56% mass loss was observed between 120 and 395 °C, 20.67% mass loss was observed between 395 and 645 °C and 7.41% mass loss was observed between 645 and 1000 °C. The o-(3-PHMB)-P oligomer showed a three-

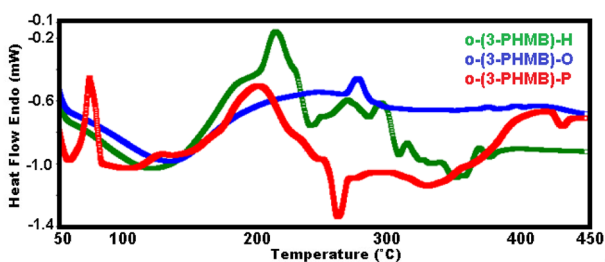
Table 1 Thermal analysis (°C) results of the monomer and oligomers

Compounds	3-PHMB	o-(3-PHMB)-O	o-(3-PHMB)-P	o-(3-PHMB)-H
^a T_{on}	240	165	181	176
^b T_{max}	273	242, 467, 730	249, 494, 739	310, 448, 732
^c T_5	228	205	203	217
^d T_{10}	247	243	236	258
^e T_{20}	266	324	285	323
^f T_{50}	284	737	625	750
char % at 1000 °C	17.75	44.35	41.43	38.72
LOI (%)	24.6	35.24	34.07	32.99
^g T_g	-	144	118	138
Endo	163	-	259, 329	348
Exo	288	-	200	213
^h ΔC_p	-	0.0513	0.145	0.072

^aThe temperature at which decomposition begins, ^bThe temperature at which the maximal rate of decomposition occurs, ^c, ^d, ^e, ^fTemperatures at 5%, 10%, 20% and 50% weight loss (T_5 , T_{10} , T_{20} , and T_{50}).

^gGlass transition temperature,

^hChange of specific heat during glass

**Fig. 5** DSC curves of o-(3-PHMB)-O, o-(3-PHMB)-P, and o-(3-PHMB)-H

stage decomposition: In the first stage, 24.09% mass loss occurred between 120 and 411 °C, in the second stage, 17.70% mass loss occurred between 411 and 677 °C and in the third stage, 16.78% mass loss occurred between 677 and 1000 °C. The o-(3-PHMB)-H oligomer showed a three-stage decomposition: 27.24% mass loss between 120 and 390 °C, 20.48% mass loss between 390 and 669 °C and 13.57% mass loss in the final stage between 659 and 1000 °C. The glass transition temperatures (T_g) of the produced oligomers were measured using the differential scanning calorimetry (DSC) method [27]. The thermal transition behaviors were assessed, and the resultant thermograms are shown in Fig. 5. The analyses revealed that the T_g values for o-(3-PHMB)-O, o-(3-PHMB)-P, and o-(3-PHMB)-H were 144, 118, and 138 °C, respectively. The results indicate that structural differences have significant effects on the thermal properties of the oligomers. Furthermore, it was revealed that a high T_g value is associated with low free volume and limited chain mobility, while a low T_g value is associated with larger free volume and increased chain flexibility. In this context, it can be said that the o-(3-PHMB)-O oligomer with a high T_g value has a more rigid and ordered chain architecture. In conclusion, the thermal behavior of 3-PHMB-based oligomers is directly related to the type of bonding, aromatic

structure density, chain regularity, and the oxidation character of the oxidizing agent used. The type of oxidizing agent not only initiates oligomerization but also plays a decisive role in determining the chain architecture, thermal stability, and degradation mechanism. This mechanistic approach allows for a holistic interpretation of the TG-DTG and DSC results within the framework of structure-property relationships.

The flammability characteristics of the polymers were assessed using the Limiting Oxygen Index (*LOI*), a flame retardancy test [28]. The *LOI* values of the synthesized oligomers were calculated using the following equation [29]:

$$LOI = 17.5 + 0.4 (s) \quad (1)$$

s represents the amount of char formed during the thermal decomposition of the polymer. The *LOI* values for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H were found to be 24.60, 35.24, 34.07, and 32.99, respectively. As seen in Table 1, the o-(3-PHMB)-O sample has a higher *LOI* value compared to o-(3-PHMB)-H and o-(3-PHMB)-P. In the literature, materials with an *LOI* value below 21 are defined as readily flammable in air; those in the range of 21–27 are defined as “slow-burning” and those above 27 are generally defined as “self-extinguishing” [30, 31]. According to this classification, o-(3-PHMB)-O, o-(3-PHMB)-P, and o-(3-PHMB)-H oligomers belong to the group of spontaneously extinguishable materials. The higher *LOI* value of the o-(3-PHMB)-O oligomer can be mechanistically related to the greater char formation during thermal decomposition and the resulting limitation of oxygen diffusion.

Molecular weight distributions of oligomers

An important technique for determining the molecular weight distribution and structural properties of oligomers is gel permeation chromatography (GPC). In this study, GPC analysis was used to examine the molecular weights of o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H oligomers. Peak molecular weight (M_p), Z-average molecular weight (M_z), weight-average molecular weight (M_w), and number-average molecular weight (M_n) data were obtained from the experiment. Table 2 provides detailed information about these data and Fig. 6 shows the GPC chromatogram of o-(3-PHMB)-O. The M_w values were determined as 3600, 4700 and 4100 Da for o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H, respectively, while the estimated M_n values were 3350, 3850 and 3600 Da, respectively. The variability in the molecular weight distribution is indicated by the M_w value, which is always greater than or equal to M_n . The M_w/M_n ratio is used to calculate the polydispersity index (PDI), which describes the distribution width of the molecules. As a result of the calculations, O-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P were found to have PDI values of 1.075, 1.221, and 1.139, respectively. These findings indicate

Table 2 M_n , M_w , M_z , M_p and PDI values of o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H

Oligomers	M_n (Da)	M_w (Da)	M_z (Da)	M_p (Da)	PDI
o-(3-PHMB)-O	3350	3600	3750	3300	1.075
o-(3-PHMB)-H	3850	4700	4850	3600	1.221
o-(3-PHMB)-P	3600	4100	4500	3400	1.139

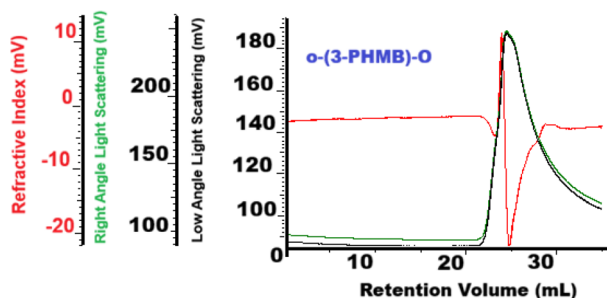


Fig. 6 GPC chromatogram of *o*-(3-PHMB)-O

that the molecular weight distribution of the produced oligomers was restricted. The homogeneity of the material and the regulated polymerization process are indicated by a PDI score close to 1.0 [32]. PDI values close to 1 indicate that the oligomers have a narrow molecular weight distribution and a controlled oligomerization process. Additionally, the degree of polymerization (DP), which represents the chain length of the oligomers, was calculated. The DP value is determined by dividing the average molecular weight (M) by the repeating unit weight (M_n). The molecular weight of 3-PHMB is $228.25 \text{ g mol}^{-1}$. DP values for *o*-(3-PHMB)-O, *o*-(3-PHMB)-H, and *o*-(3-PHMB)-P were determined as 15, 17, and 16, respectively, according to calculations. Chain length and therefore mechanical properties are affected by the increase in DP. This increase in DP values indicates an increase in chain length, and consequently, an improvement in the mechanical and thermal properties of the oligomers [33].

Electrochemical and optical properties of 3-PHMB and its Oligomers (*o*-(3-PHMB)-O, *o*-(3-PHMB)-P and *o*-(3-PHMB)-H)

One kind of potential dynamic electrochemical approach is the use of electrochemical CV measurements to ascertain the electrochemical characteristics of the substances. The CV measurements were used to evaluate one electron excitation from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO) of 3-PHMB, *o*-(3-PHMB)-O, *o*-(3-PHMB)-P, and *o*-(3-PHMB)-H. Figure 7 displayed the voltammograms that were collected. A crucial stability indicator that also shows a molecule's chemical activity is the HOMO-LUMO energy gap [34]. The following formulas were used to determine the E_{ox} and E_{red} potentials from the obtained voltammograms in order to determine the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) potentials [35, 36]:

$$E_{\text{HOMO}} = -(4.39 + E_{\text{ox}}) \quad (2)$$

$$E_{\text{LUMO}} = -(4.39 + E_{\text{red}}) \quad (3)$$

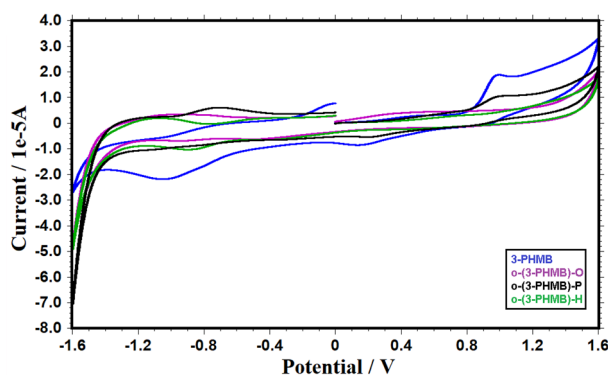


Fig. 7 Cyclic voltammograms of 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-P and o-(3-PHMB)-H

Table 3 Electronic and optical structure parameters of 3-PHMB and oligomers

Compounds	E_{red}	E_{ox}	$^a\text{HOMO (eV)}$	$^b\text{LUMO (eV)}$	$^cE_g' \text{ (eV)}$	$\lambda_{\text{onset (nm)}}$	$^dE_g \text{ (eV)}$
3-PHMB	-1.0355	+0.9907	-5.3807	-3.3545	2.03	532	2.33
o-(3-PHMB)-O	-0.9084	+0.5867	-4.9767	-3.4816	1.50	614	2.02
o-(3-PHMB)-H	-0.8905	+0.7364	-5.1264	-3.4995	1.63	574	2.16
o-(3-PHMB)-P	-1.0091	+0.6358	-5.0258	-3.3809	1.65	604	2.06

^{a} Highest occupied molecular orbital, ^{b} Lowest unoccupied molecular orbital, ^{c} Electrochemical band gap,

^{d} Optical band gap

The E_{ox} values for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P were recorded as 0.990, 0.586, 0.736, and 0.635 eV, respectively, while the E_{red} values were recorded as -1.035, -0.908, -0.890, and -1.009 eV, respectively. The HOMO energy levels for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P compounds were found to be -5.380; -4.976; -5.126, and -5.025 eV, respectively. The LUMO energy levels of the same compounds were found to be -3.354; -3.481; -3.499, and -3.380 eV, respectively. The higher (less negative) HOMO levels in oligomers compared to monomers indicate that increased conjugation along the chain strengthens the electron-donating character. The empirical formula [37] $E_g' = E_{\text{LUMO}} - E_{\text{HOMO}}$, which is displayed in Table 3, states the compounds' electrochemical band gap (E_g'). According to this equation, the electrochemical band gaps of 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P compounds were determined as 2.11, 1.52, 1.60, and 1.87 eV, respectively. It was discovered that the optical band gap (E_g) values determined from UV-Vis analysis and the electrochemical band gap (E_g') values derived from cyclic voltammetric measurements were compatible. This agreement shows that the methodologies are consistent. Additionally, because of their polyconjugated nature, polymers were discovered to have somewhat lower E_g values than their monomers [38]. The lower E_g' values in oligomers can be mechanistically explained by the narrowing energy difference between HOMO–LUMO levels due to the polyconjugated structure.

Figure 8 displays the UV-Vis spectra of the compounds 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P. Table 3 lists the λ_{onset} and E_g values derived

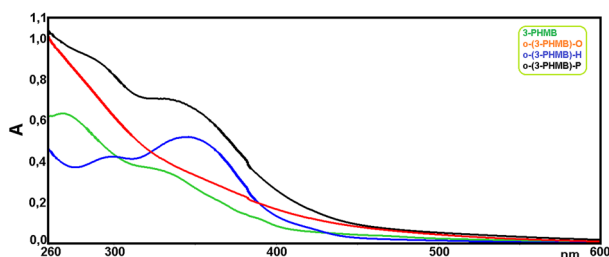


Fig. 8 The change UV spectra of 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H and o-(3-PHMB)-P

from these substances' UV-Vis spectra. The λ_{onset} values in the UV-Vis spectra of the pertinent compounds were used to compute the optical band gap (E_g) values, which came out to be 2.33, 2.02, 2.16, and 2.06 eV, for 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P, respectively. The methods employed are consistent with one another since the optical band gap values match the electrochemical band gaps. Increased conjugation length and the occurrence of polymerization are thought to be indicated by the UV-Vis spectra of oligomers showing absorption bands shifted to wider and longer wavelengths. Functional groups like -OH and -CH=N have comparable inductive and resonance effects, and the bands seen in the spectra of oligomers are quite close to one another, indicating that these compounds' molecular structures are similar [39]. Broad absorption bands are thought to be a sign of polymerization. According to published reports, polymers with E_g values below 3.0 eV are semiconducting, and those with values above 3.0 eV are insulating. In this regard 3-PHMB, o-(3-PHMB)-O, o-(3-PHMB)-H, and o-(3-PHMB)-P are classified as semiconducting [40].

Surface images of monomer (3-PHMB) and oligomer o-(3-PHMB)-H

The SEM images shown in Fig. 9 reflect the surface morphologies of the 3-PHMB (A) monomer and the o-(3-PHMB)-H (B) oligomer at different magnifications. It is noteworthy that the surface of 3-PHMB at 500X magnification is separated into irregular layers and exhibits a relatively compact structure. At 3kX magnification of the same monomer, the edges of these layers are seen to be brittle, with distinct gaps between them. The 500X magnification image of o-(3-PHMB)-H reveals the presence of sponge-like structures in addition to irregular layers on the surface. At higher magnification, the layers were observed to become more distinct, the edges exhibited a more brittle structure, and the particles adhering to the surface formed a porous structure. This morphological change can be mechanistically explained by the fact that chain growth and irregular packing during the oligomerization process increase surface roughness and porosity. In conclusion, it was determined that both samples exhibited a layered, heterogeneous, and porous surface morphology; however, oligomerization promoted the formation of a more irregular and porous structure on the surface.

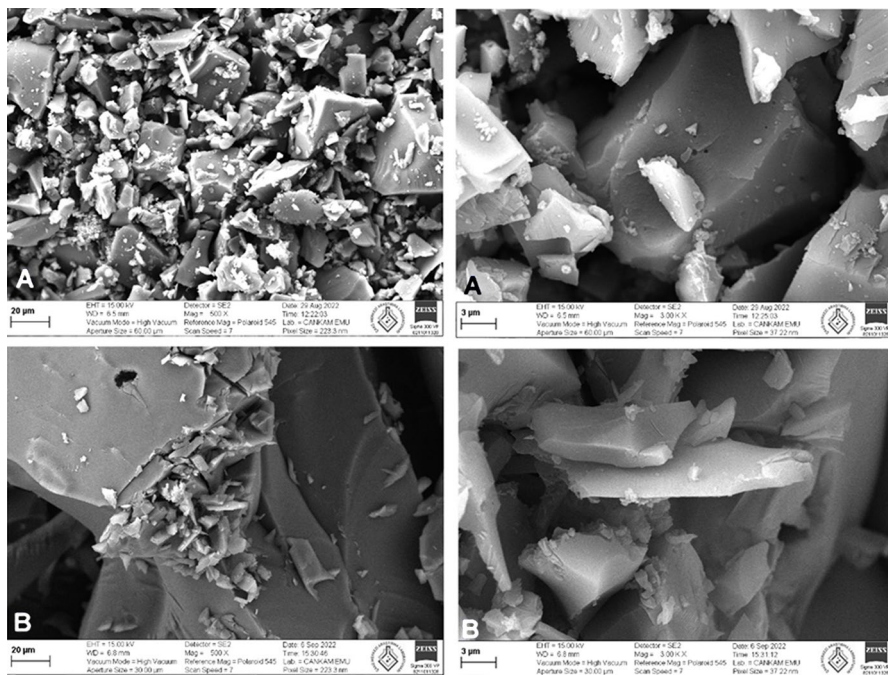


Fig. 9 SEM images of 3-PHMB (A) and o-(3-PHMB)-H (B)

Dielectric properties of o-(3-PHMB)-O and o-(3-PHMB)-P

Utilizing a room temperature and frequency range of 100 Hz to 1 MHz, the Fytronix FY-FP-9000 instrument was used to examine the dielectric characteristics of o-(3-PHMB)-O and o-(3-PHMB)-P compounds. Disc-shaped samples measuring around 50 mm in diameter and 5 mm in thickness were used for the measurements. The values of dielectric loss (ϵ''), and dielectric constant (ϵ') were ascertained. Equations 4 and 5 were used to calculate these parameters.

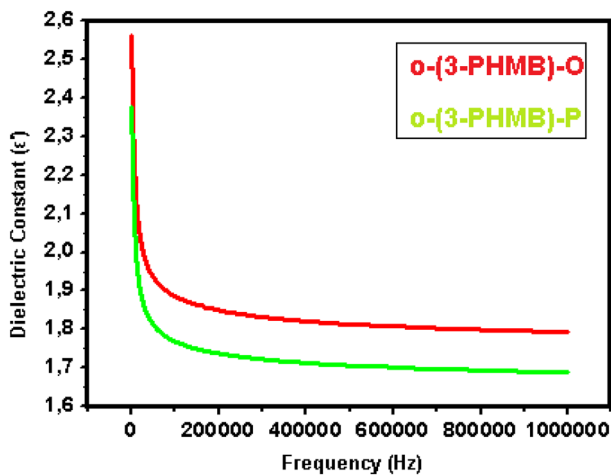
$$\epsilon' = C \frac{d}{\epsilon_0 A} \quad (4)$$

$$\epsilon'' = D_f \times \epsilon' \quad (5)$$

In this case, d is the sample thickness, A is the sample surface area, ϵ_0 is the free space permittivity ($8.854 \times 10^{-12} \text{ F m}^{-1}$), D_f is the dissipation factor, and C is the capacitance of the composites as determined in the frequency range of 100 Hz to 1 MHz. The dielectric constant, dielectric loss and conductivity values of o-(3-PHMB)-O and o-(3-PHMB)-P are given in Table 4 at 25 °C.

Table 4 Summary of dielectric constant, dielectric loss and conductivity values of o-(3-PHMB)-O and o-(3-PHMB)-P at 25 °C

Oligomers	Dielectric constant (ϵ')		Dielectric loss (ϵ'')		Conductivity (σ , S cm ⁻¹)	
	1 kHz	100 kHz	1 kHz	100 kHz	1 kHz	100 kHz
o-(3-PHMB)-O	2.57	1.88	0.298	0.254	2.72×10^{-7}	1.74×10^{-5}
o-(3-PHMB)-P	2.37	1.76	0.287	0.230	2.52×10^{-7}	1.65×10^{-5}

**Fig. 10** The change of the dielectric constant vs. frequency of synthesized o-(3-PHMB)-O and o-(3-PHMB)-P

Dielectric constant

A material is defined as a “dielectric” if it can store energy when an external electric field is applied. The dielectric constant (ϵ') indicates the amount of energy retained and lost by a material when an electric field is applied [41]. According to physical studies, the primary factor affecting the value of ϵ is the polarization properties of the material. The primary mechanisms affecting the value of ϵ in this environment are interface polarization, dipole polarization, electron polarization, and atomic polarization [42, 43]. Dipole polarization occurs at lower frequencies and lasts longer than other types of polarization. Additionally, an important factor affecting the value of ϵ' is the polarity of the material, and the dielectric constant increases with increasing polarity [44]. Figure 10 shows the findings of this research, which examined the ϵ' values of o-(3-PHMB)-O and o-(3-PHMB)-P with increasing frequency at room temperature. In the low-frequency range (1 kHz), the dielectric constant of o-(3-PHMB)-O oligomer is measured to be 2.57, while that of o-(3-PHMB)-P is 2.37. As the frequency increases, a significant decrease in the dielectric constant of both materials occurs, and in the high-frequency region (100 kHz), the value for o-(3-PHMB)-O decreases to 1.88, while o-(3-PHMB)-P decreases to 1.76. This decrease is due to the dipole polarization effective at low frequencies not being able to adapt to the electric field changes as the frequency increases. The higher dielectric constant of the

o-(3-PHMB)-O oligomer compared to o-(3-PHMB)-P across all frequency ranges is associated with higher dipole density and more effective chain polarization. This result indicates that the o-(3-PHMB)-O oligomer has a stronger polarization capacity.

Dielectric loss

Dielectric loss (ϵ'') is one of the fundamental parameters to be considered in material selection and expresses the rate at which the applied energy is converted into heat within the material [45, 46]. Figure 11 shows the frequency-dependent dielectric loss behavior of o-(3-PHMB)-O and o-(3-PHMB)-P oligomers at room temperature. The findings reveal that both oligomers exhibit high dielectric loss values at low frequencies. Specifically, at 1 kHz, the ϵ'' value is 0.298 for o-(3-PHMB)-O and 0.287 for o-(3-PHMB)-P. As the frequency increases, a significant decrease in ϵ'' values occurs, and these values drop to 0.254 and 0.230 at 100 kHz, respectively, reaching a more constant level in the high frequency regions. This is due to the dominance of free charge carriers and dipole polarization at low frequencies, while at high frequencies the dipoles cannot adapt to the electric field changes [47]. The higher ϵ'' values exhibited by the o-(3-PHMB)-O oligomer across all frequency ranges suggest a more efficient polarization relaxation mechanism and a higher dipole density caused by oxidation with oxygen. Consequently, although both oligomers exhibit decreasing energy losses with increasing frequency, the dielectric loss behavior of the o-(3-PHMB)-O oligomer reflects a mechanistically more dominant polarization process.

Conductivity

Figure 12 presents the frequency-dependent conductivity ($\log\sigma$) changes of o-(3-PHMB)-O and o-(3-PHMB)-P oligomers. It is observed that the conductivity values of both oligomers are low at low frequencies. The measured AC conductiv-

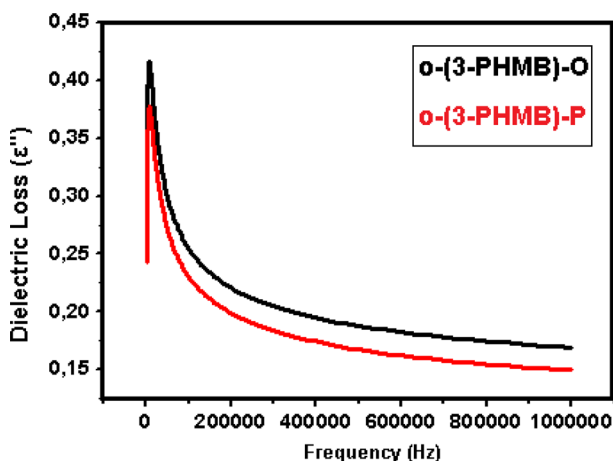


Fig. 11 The change of the dielectric loss vs. frequency of synthesized o-(3-PHMB)-O and o-(3-PHMB)-P

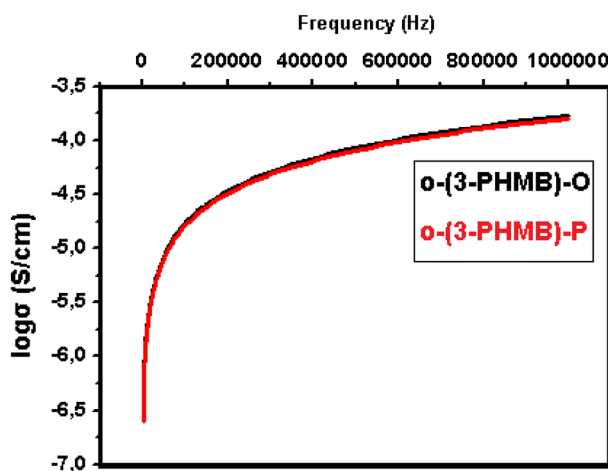


Fig. 12 The change of the conductivity vs. frequency of synthesized o-(3-PHMB)-O and o-(3-PHMB)-P

ity (σ) at 1 kHz is $2.72 \times 10^{-7} \text{ S cm}^{-1}$ for o-(3-PHMB)-O and $2.52 \times 10^{-7} \text{ S cm}^{-1}$ for o-(3-PHMB)-P. As the frequency increases, a regular and significant increase in conductivity values occurs. At 100 kHz, the conductivity increases by approximately two orders of magnitude to $1.74 \times 10^{-5} \text{ S cm}^{-1}$ and $1.65 \times 10^{-5} \text{ S cm}^{-1}$, respectively. This increase can be mechanistically explained by the fact that free charge carriers, which have limited mobility at low frequencies, adapt more quickly to changes in the applied electric field at high frequencies. The fact that the conductivity curves of both oligomers are quite close to each other indicates that the chain structure and charge transport mechanisms are similar. The increased mobility of charge carriers along the polymer chains with increasing frequency leads to an increase in AC conductivity. The fact that the conductivity values approach each other and follow a stable course in the high-frequency region indicates that conductivity is predominantly controlled by a field-assisted transport mechanism.

Conclusion

In this study, a phenylhydrazine-based Schiff base monomer (3-PHMB) was successfully synthesized and polymerized into three distinct oligomers using O_2 , NaOCl, and H_2O_2 as oxidants. Structural and GPC analyses confirmed efficient, controlled oligomerization, yielding molecular weights between 3600 and 4700 Da with narrow polydispersity ($\text{PDI} < 1.22$). The transition from monomer to oligomer significantly enhanced thermal stability, with 50% mass loss temperatures reaching up to 750 °C and high char yields (~44%) indicating excellent flame-retardancy (LOI: 33–35). Optoelectronic characterization revealed semiconducting behavior with narrowed band gaps (1.52–2.16 eV), while dielectric measurements highlighted the superior polarization capacity of the O_2 -oxidized variant. The higher dielectric constant of the o-(3-PHMB)-O oligomer can be explained not only by the increased dipole density but also by the increased relative contribution of the chain end groups due to its

low molecular weight. At shorter chain lengths, the higher proportion of polar end groups may enhance the overall dipole moment of the system and thus its dielectric response. Despite limitations regarding mechanical testing, the findings suggest these oligomers are promising candidates for flame-retardant coatings and organic electronics. Future work is recommended to focus on thin-film processability and composite integration.

Author contributions İsmet Kaya : Conceptualization, Data curation, Formal analysis, Funding acquisition, Methodology, Investigation, Resources, Validation, Visualization, Writing - review & editing. Erdal Bayır : Data curation, Investigation, Methodology, Software, Validation, Visualization. Adnan Solmaz: Data curation, Investigation, Methodology, Software, Validation, Writing - review & editing.

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Data availability Data will be made available on request.

Declarations

Competing interests The authors declare no competing interests.

Ethical approval Not applicable.

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