

Determination of Phenolic Hydroxyl Content in Poly(phenylene oxide) by Differential Ultraviolet Spectrophotometry

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Abstract Poly(phenylene oxide) (PPO) exhibits excellent dielectric properties, making it an ideal substrate for high-frequency, high-speed copper-clad laminates. The phenolic hydroxyl group at the end of PPO plays a key role in its reactivity. Accurately quantifying the phenolic hydroxyl content in PPO is essential but challenging. In this study, we proposed a method for measuring the phenolic hydroxyl content of PPO using differential UV absorption spectroscopy. In alkaline solutions, the phenolic hydroxyl in PPO completely ionizes to form phenoxide ions, leading to a significant increase in UV absorbance at approximately 250 and 300 nm. Notably, the differential UV absorbance at approximately 300 nm was directly proportional to the phenolic hydroxyl concentration. Using 2,6-dimethylphenol as a standard, a calibration curve was established to relate the phenolic hydroxyl concentration to differential UV absorbance at approximately 300 nm, providing a precise and straightforward method for phenolic hydroxyl quantification in PPO with distinct advantages over conventional techniques.

Keywords Poly(phenylene oxide); Phenolic hydroxyl; Redistribution; Differential UV spectrophotometry

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INTRODUCTION

The rapid advancement of high-frequency electronic communication technology has exposed the limitations of traditional copper-clad laminate substrates, particularly those based on epoxy resins, which struggle to meet dielectric requirements at elevated frequencies.^[1,2] Consequently, alternative materials are being actively explored, with poly(phenylene oxide) (PPO) emerging as a promising candidate owing to its excellent dielectric properties. PPO has a low dielectric constant ($D_k=2.45$) and minimal dielectric loss ($D_f=0.0007$), making it particularly suitable for high-frequency applications.^[3,4]

Despite its excellent performance, PPO faces challenges when integrated with other resins such as polyamide^[5] and polyester^[6], particularly the commonly used and cost-effective epoxy resin^[7]. This integration difficulty stems primarily from the limited number of reactive functional groups in PPO.^[8] To overcome this challenge, researchers have focused on modifying PPO by reducing its molecular weight to increase the number of terminal hydroxyl groups, although this adjustment entails a slight compromise in the dielectric performance.^[9,10] These hydroxyl groups can participate di-

rectly in the curing process with epoxy groups, thereby enhancing the co-curing process and improving material compatibility.^[11,12] Consequently, the hydroxyl content of the modified PPO plays a crucial role in determining the success of these integration strategies.

However, the accurate quantification of the hydroxyl content in modified PPO remains a significant challenge. Traditional methods, such as acid-base titration, have been used to quantify phenolic hydroxyl groups. Krijgsman *et al.*^[13] dissolved PPO in pyridine and titrated it with tetrabutylammonium hydroxide in a mixture of isopropanol and methanol. Another method involves reacting the hydroxyl groups with acyl chloride and titrating the resulting hydrochloric acid. Although titration methods that replace hydrogen in the phenolic hydroxyl groups are more commonly employed, they often suffer from reduced accuracy. This is because the reactivity of the terminal hydroxyl groups in PPO can vary depending on factors such as molecular weight and structural variations, leading to inconsistent results.

Instrumental techniques, such as nuclear magnetic resonance (NMR) spectroscopy, offer a more reliable and precise quantification of hydroxyl content using an internal standard to ensure stability and accuracy.^[14] However, NMR spectroscopy is complex and requires high-purity samples. As an alternative, infrared (IR) spectroscopy has been explored for

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direct measurement of hydroxyl content. Some studies have focused on the absorption peak at 3500 cm^{-1} in toluene solutions of PPO at varying concentrations.^[15] Unfortunately, because of the typically low hydroxyl content of PPO, the absorption peaks are weak and often masked by noise or overlapping peaks, making IR spectroscopy unsuitable for precise hydroxyl quantification. Gel permeation chromatography (GPC), which estimates hydroxyl content based on molecular weight measurements, has also been used in some studies.^[16] However, its accuracy is limited because it does not consider unreacted raw materials or side reactions during synthesis.

An alternative approach involves differential ultraviolet (UV) absorption spectroscopy. In alkaline media, the phenolic hydroxyl groups dissociate into phenoxide ions, causing characteristic shifts in their differential UV absorption peaks. Two distinct absorption peaks, typically near 250 and 300 nm, are associated with phenolic hydroxyl groups, and the intensity of these peaks can be used to quantify the hydroxyl content.^[17] Wu *et al.*^[18] used water as the solvent and focused on a stronger absorption peak near 250 nm, achieving more reliable results for lignin phenolic hydroxyl quantification. Similarly, Chen *et al.*^[19] used a solvent system of 2-methoxyethanol/water/acetic acid=8/2/0.2 (V/V/V) or 2-methoxyethanol/0.2 mol/L NaOH=1/9 (V/V) to measure the phenolic hydroxyl content in lignin; however, solvent interference remains a potential issue, complicating the interpretation of UV absorption data.

In this study, various phenolic hydroxyl-terminated PPOs were synthesized through redistribution methods, as outlined by White.^[20] Differential UV spectroscopy was used to analyze and quantify the phenolic hydroxyl content in the PPO samples. A phenolic hydroxyl standard reference was chosen to calibrate the measurements, providing a more accurate and reliable method for quantifying the phenolic hydroxyl in PPO.

EXPERIMENTAL

Materials

The PPO used in this study was RX040 sourced from Sinbo Co., China. Another PPO variant, SA90, was purchased from Sabic Co. (USA). The chemicals 2, 6-dimethylphenol (DMP), bisphenol A (BPA), and tetramethylbisphenol A (TMBPA), each with a purity of 98%, were purchased from J&K Co. Methanol, dichloromethane (spectrum grade), and tetraethylammonium hydroxide were obtained from Aladdin Co., Ltd. All other reagents used were of analytical grade and were supplied by SCR Co., Shanghai, China.

Preparation of PPO with Varying Phenolic Hydroxyl Content

To prepare PPO samples with varying phenolic hydroxyl contents, a redistribution method was employed. This technique involves the reduction of the molecular weight of PPO. Initially, 20 g of PPO and 0.8 g of TMBPA were dissolved in 150 mL of the solvent and heated to $85\text{ }^{\circ}\text{C}$. A 35 mL solution of BPO in toluene, with a concentration of 0.04 g/mL, was gradually added to the reaction mixture over a 3 h period. After the addition was complete, the reaction was allowed to continue for 3 h. After cooling to room temperature, methanol (20 wt% of the total reac-

tion mixture) was slowly introduced under vigorous stirring to precipitate and filter out high-molecular-weight PPO that did not participate in the reaction. The filtrate was then transferred to an excess of methanol (five times the volume of the filtrate), resulting in the precipitation of a white solid. The solid was filtered, washed, and dried under vacuum at $100\text{ }^{\circ}\text{C}$ to obtain the Modified PPO product designated as MPPO-1. The mass of TMBPA was adjusted to 1.6, 2.4, and 3.2 g, yielding MPPO products with different phenolic hydroxyl contents, labeled MPPO-2, MPPO-3, and MPPO-4, respectively.

Measurement of Phenolic Hydroxyl Groups by UV Spectrophotometry

Phenolic hydroxyl groups were quantified using Shanghai Meipuda P7 UV spectrophotometry. A 0.1 mol/L methanol solution of tetraethylammonium hydroxide (TEAH) was used as the alkaline ionization reagent. The solution was mixed separately with BPA, TMBPA, and DMP in fixed proportions under vigorous stirring. The UV absorbance spectra were recorded before and after ionization and subsequently exported from the instrument. Differential spectra were obtained by manually subtracting the pre- and post-ionization absorbance curves. Based on the results obtained with varying concentrations of BPA, TMBPA, and DMP, three calibration curves of the differential absorbance versus phenolic hydroxyl concentration were constructed. PPO samples of known masses were tested under identical conditions and the absorbance values were measured. The phenolic hydroxyl content of the PPO samples was determined using the calibration curves.

Characterization of PPO Samples

The molecular weights and molecular weight distributions of the PPO samples were determined by GPC using a Waters 1525/2414 system. PPO samples were dissolved in tetrahydrofuran (THF) at a concentration of 3 mg/mL. Polystyrene (PS) was used as the standard and THF was employed as the mobile phase at a flow rate of 1 mL/min.

Fourier-transform infrared spectroscopy (FTIR) was used to analyze the PPO samples. FTIR spectra were recorded using the KBr pellet method on an Agilent Nicolet 5700 infrared spectrometer, with a scan rate of 32 scans per second and a resolution of 4 cm^{-1} .

RESULTS AND DISCUSSION

Characteristics of Different MPPO Samples

The redistribution method, a well-established technique for reducing the molecular weight of PPO, was employed to obtain PPO samples with varying phenolic hydroxyl contents. As shown in Fig. 1, free radicals initiated the reaction by triggering the functionalized phenol to attack the benzyl ether phenoxy terminal radical. The process followed a quinone-retraction mechanism, in which the functionalized phenol was incorporated into the PPO backbone. The reaction proceeded as an equilibrium process, progressively reducing the molecular weight of PPO as more functionalized phenol units were integrated.

Four PPO samples with reduced molecular weights were obtained *via* redistribution; their corresponding GPC curves are presented in Fig. 2. The number-average molecular weights of the modified PPO samples (MPPO-1 to MPPO-4) decreased sequentially from initial values of 25781 to 3489,

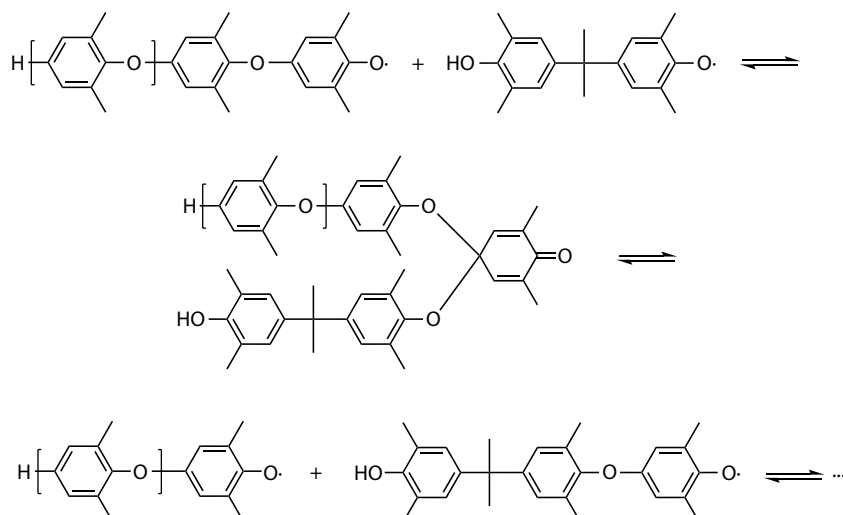


Fig. 1 Redistribution process of PPO in the presence of TMBPA and BPO.

3069, 2591, and 1909, respectively. Although the redistribution reaction theoretically results in PPO products with terminal hydroxyl groups at both ends, the exact number of terminal hydroxyl groups cannot be directly determined from GPC results alone.

The FTIR spectra of these PPO samples are shown in Fig. 3, where a prominent band near 3610 cm^{-1} corresponds to the phenolic hydroxyl group. It is evident that as the molecular weight of PPO decreased, the intensity of this peak increased, indicating an increase in the content of phenolic hydroxyl groups. However, quantifying this peak precisely remains challenging because of the complexities involved in calculating its shape characteristics.

Measurement of Phenolic Hydroxyl Content Using Differential UV Spectrophotometry

UV absorption of phenolic hydroxyl groups

Phenolic hydroxyl groups are typically nonionizable in nonpolar or acidic solutions. However, under alkaline conditions, these groups ionize to form phenoxide ions, as shown in Fig. 4. The formation of phenoxide ions significantly enhanced the UV absorbance at approximately 250 and 300 nm. For example, when TMBPA is exposed to a strong organic base, such as TEAH, in a methanol solution, its UV absorption at these wavelengths increases noticeably, as illustrated in Fig. 5(a). Subtracting the UV spectra before and after the addition of TEAH yielded a differential UV spectrum (Fig. 5b), which highlighted the changes induced by ionization. This ionization process resulted in two distinct differential UV peaks, confirming complete ionization of the phenolic hydroxyl group in TMBPA in the TEAH environment.

As PPO is insoluble in methanol, dichloromethane, a non-polar solvent, was selected because of its high solubility in PPO. This ensured that PPO remained dissolved and did not

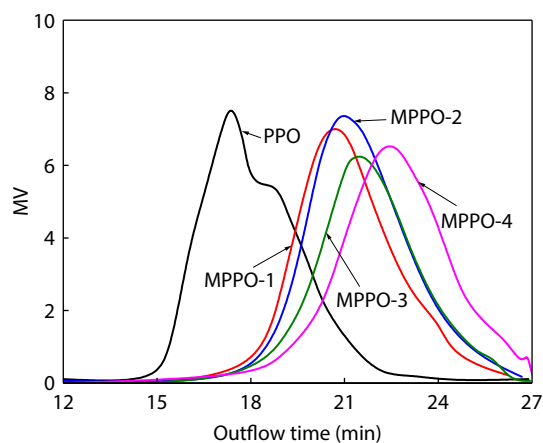


Fig. 2 GPC curves of PPO and redistribution products.

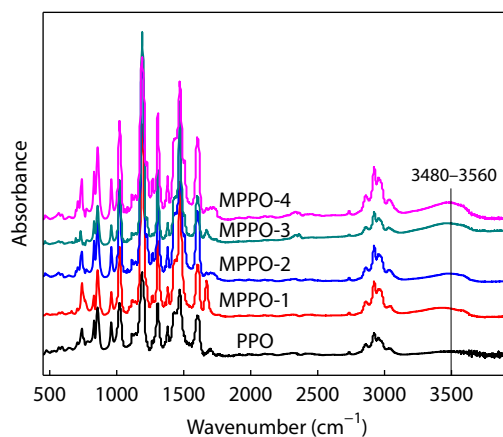


Fig. 3 FTIR spectra of PPO and redistribution products.

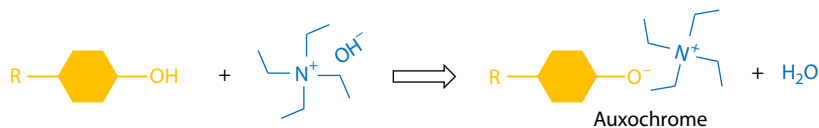


Fig. 4 Ionization of phenolic hydroxyl groups in alkaline solution.

precipitate, even after the addition of an alkaline solution. A TEAH/methanol mixture was chosen as the alkaline organic solution because it effectively promotes the complete ionization of the phenolic hydroxyl group and provides better solubility in the organic solvent system than conventional inorganic bases such as potassium hydroxide. However, the limited solubility of PPO in methanol necessitates careful control of the methanol content to maintain the solution stability during testing. When a TEAH methanol solution with a concentration exceeding 12% was added to the system, slight precipitation of PPO occurred. This effect is expected to be even more pronounced for higher-molecular-weight PPO, potentially leading to test failure. In contrast, the addition of 10% TEAH methanol solution did not cause precipitation of any PPO sample used in this study. Therefore, the final solution mixture was prepared at a dichloromethane-to-methanol volume ratio of 9:1.

As shown in the UV spectra in Fig. 6(a), the addition of the TEAH/methanol mixture to the PPO-dichloromethane solution resulted in enhanced UV absorption peaks around 250 and 300 nm. This enhancement was clearly visible in the differential UV absorption curve (Fig. 6b), which was derived by subtracting the spectra before and after TEAH addition of TEAH. These observations suggest that TEAH increases the alkalinity of the PPO-dichloromethane solution, promoting the

complete ionization of the terminal phenolic hydroxyl group of PPO. While PPO exhibits ionization behavior similar to that of small phenolic molecules, it is important to note that the UV absorption peaks of different phenolic compounds may shift slightly owing to structural differences. For instance, UV absorption peaks for the phenolic hydroxyl groups in BPA, TMBPA, and PPO were observed at approximately 284, 289, and 300–310 nm, respectively.

Establishment of measurement standard curve

Figs. 7(a), 7(c), and 7(e) show the differential UV spectra of DMP, BPA, and TMBPA in dichloromethane at various concentrations before and after the addition of the TEAH/methanol mixture. Around 250 nm, these spectra exhibited a weaker linear relationship between absorbance and concentration, likely due to the absorption effect of the solvent. In contrast, a more pronounced linear relationship is observed at approximately 300 nm. At this wavelength, the maximum differential absorbance was recorded for each compound, and the corresponding concentration of the phenolic hydroxyl groups was determined to establish their relationship. It should be noted that DMP is a phenolic monomer with a hydroxyl group at one end, and BPA and TMBPA, each of which has hydroxyl groups at both ends, follow the following relationships: $[\text{OH}]_{\text{DMP}} = [\text{DMP}]$, $[\text{OH}]_{\text{BPA}} = 2[\text{BPA}]$, and $[\text{OH}]_{\text{TMBPA}} = 2[\text{TMBPA}]$. As shown in Figs. 7(b), 7(d) and 7(f), the correlation coefficients for the curves of DMP, BPA,

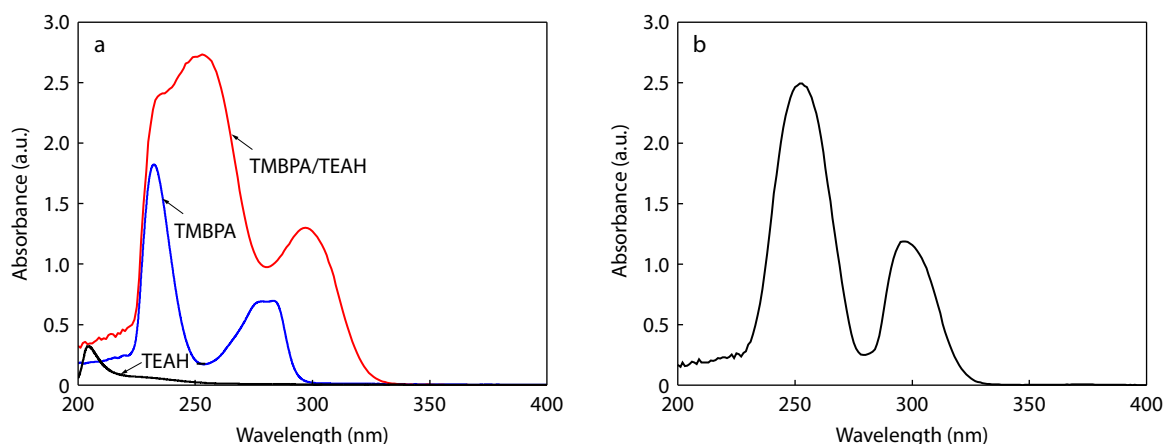


Fig. 5 (a) UV spectra of the TMBPA solution before and after adding TEAH and (b) their differential UV spectrum.

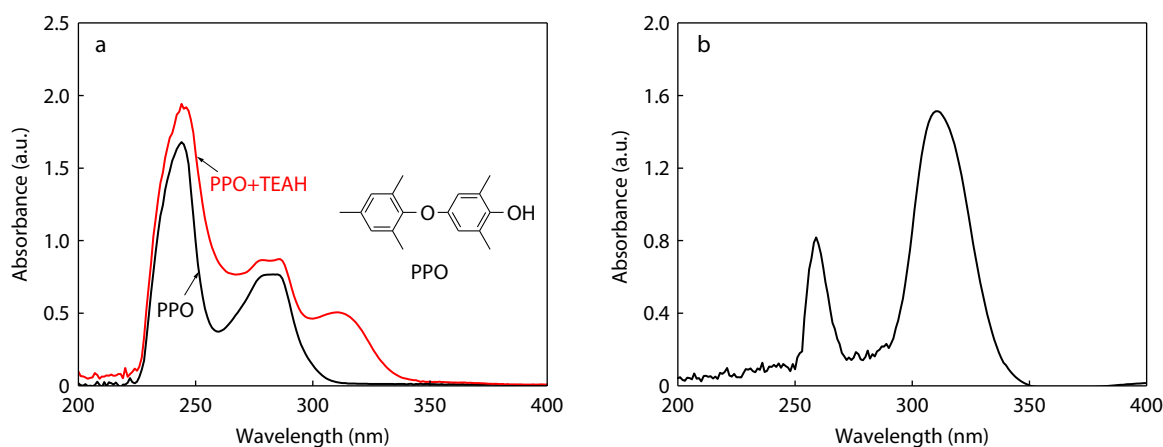


Fig. 6 (a) UV spectra of the PPO-dichloromethane solution before and after adding TEAH and (b) their differential UV spectrum.

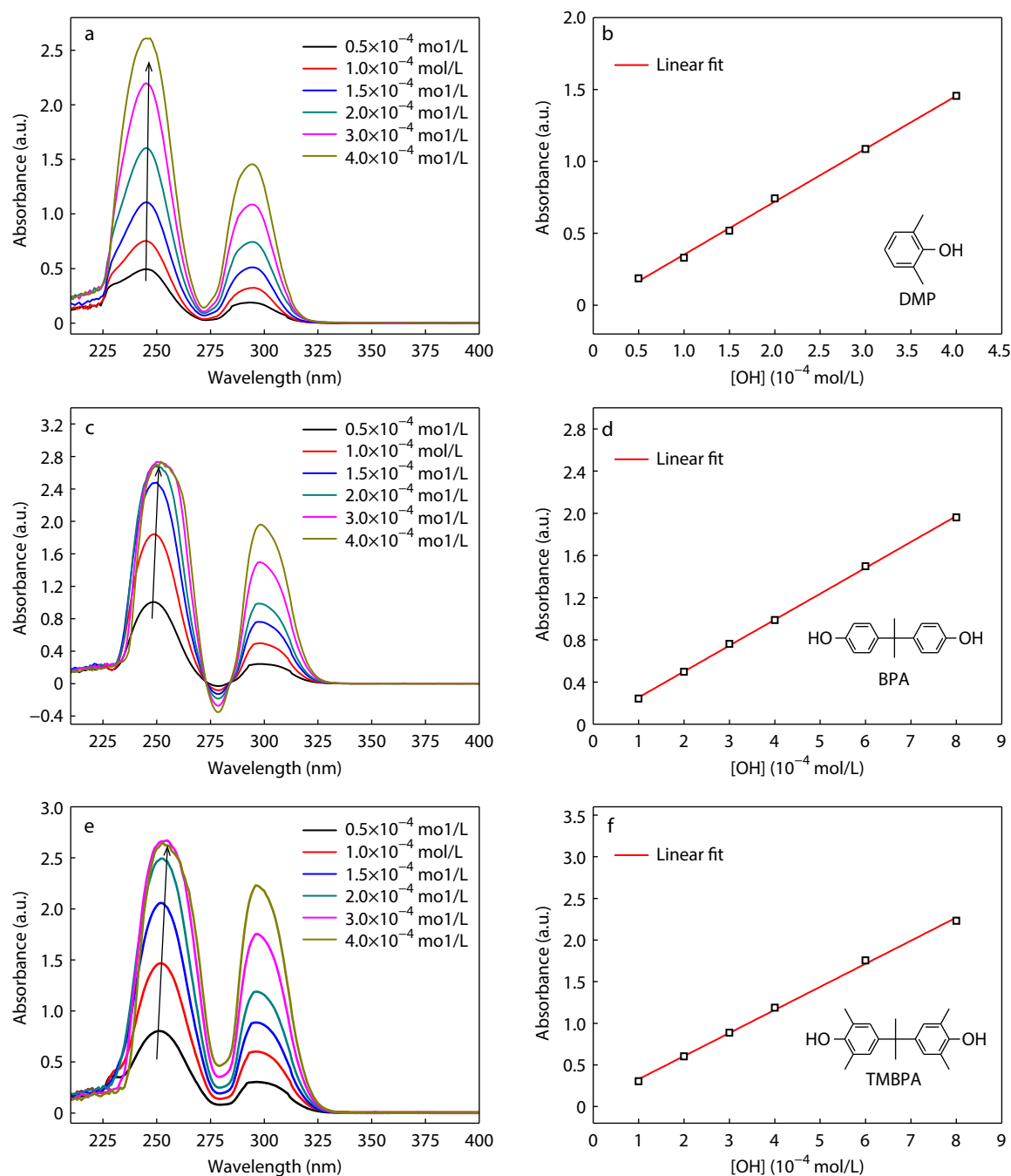


Fig. 7 Differential UV spectrograms of ionization of three reference materials with different concentrations in alkaline solution: (a) DMP, (c) BPA, and (e) TMBPA. The corresponding standard curves for absorbance at 300 nm versus the phenolic hydroxyl content: (b) DMP, (d) BPA, and (f) TMBPA.

and TMBPA are all greater than 0.99, indicating excellent linearity. This suggests that the phenolic hydroxyl groups in these compounds were completely ionized. More importantly, the differential UV absorption of the terminal phenoxy groups before and after ionization was strictly proportional to their respective concentrations. This enables reliable quantification of phenolic hydroxyl groups in these compounds within the concentration range of 5×10^{-5} – 4×10^{-4} mol/L.

Unlike these standard compounds, obtaining well-defined or oligomeric PPO standards with specific molecular weights

is considerably more challenging. Consequently, constructing a standard curve for directly determining the terminal phenolic hydroxyl group content in PPO is not feasible. DMP, a monomer used for synthesizing PPO, and both BPA and TMBPA, which are standard compounds with double-terminal hydroxyl groups, were selected as standards for measuring the hydroxyl content of PPO.

The standard curves obtained for DMP, BPA, and TMBPA were used to measure the phenolic hydroxyl content in the PPO samples, as shown in Table 1. As the molecular weights

Table 1 Phenolic hydroxyl content in PPO samples obtained based on standard curves of DMP, BPA and TMBPA.

Sample	Phenolic hydroxyl content (mmol/g)		
	DMP standard curve	BPA standard curve	TMBPA standard curve
PPO	0.11	0.15	0.20
MPPO-1	0.28	0.41	0.40
MPPO-2	0.43	0.60	0.57
MPPO-3	0.55	0.77	0.72
MPPO-4	0.73	1.17	1.08
SA90	1.05	1.54	1.40

of MPPO-1 to MPPO-4 decreased sequentially, the phenolic hydroxyl content increased. The phenolic hydroxyl contents of the PPO samples, calculated from the standard curves of DMP, BPA, and TMBPA, followed the trend predicted by their molecular weights. However, discrepancies were observed in the phenolic hydroxyl content derived from the three standard curves.

To further assess the reliability of these results, the phenolic hydroxyl content of commercially available end-hydroxy PPO (SA90) was measured using three standard curves. As shown in Table 1, the phenolic hydroxyl content calculated using the DMP-based standard curve closely matched the manufacturer's specified value (1.08 mmol/g), while the values calculated using the BPA-based and TMBPA-based standard curves were higher than the manufacturer's value. Furthermore, three repeated tests of the SA90 sample's end-phenolic hydroxyl group content were performed *via* differential UV spectroscopy using a DMP-based standard curve, yielding an average relative deviation of only 0.79%. This further supports the reliability of the DMP-based standard curve for measuring the phenolic hydroxyl content in PPOs.

The ultraviolet (UV) absorption peak of a substance is closely related to its molecular structure. In contrast to PPO, the phenyl rings in BPA and TMBPA were linked by methylene rather than ether bonds. Upon ionization in an alkaline solution, the conjugative effect of the phenoxide terminal group was weaker and less pronounced than that of PPO, which contains aromatic ether bonds. Consequently, BPA and TMBPA exhibit lower differential UV absorbance values. Therefore, using these compounds as standards for measuring the hydroxyl content of PPO would lead to an overestimation of the hydroxyl content. Furthermore, BPA lacks electron-donating methyl groups at the ortho position of its phenolic hydroxyl group, which further diminishes its differential UV absorbance. Therefore, when BPA was used as a standard, the overestimation of PPO's hydroxyl content of PPO became more pronounced, which is consistent with previous experimental findings.

In contrast, DMP, a monomer used to synthesize PPO, lacks an ether bond connecting the phenyl rings present in the repeating unit of PPO. The delocalization effect of the benzene rings in the PPO molecular chain, spanning from the ether bond to the phenoxide terminal group, resulted in minimal influence of the ether bond on UV absorption. Consequently, the structural differences between DMP and PPO had a negligible impact on their UV absorption in solution. Therefore, DMP is more suitable as a standard material for measuring the phenolic hydroxyl content in PPO.

Compared to the titration method, the differential UV spectroscopy approach presented in this study avoids errors arising from incomplete end-capping reactions and misjudgment of the titration endpoint. By subtracting the UV absorbance curves before and after ionization, the method effectively minimizes the influence of factors such as solvent interference. Compared to NMR spectroscopy, it offers additional advantages in terms of lower cost and higher efficiency. Furthermore, this methodology can be extended to determine the phenolic hydroxyl content of a variety of polymer materials, demonstrating its broad applicability.

CONCLUSIONS

A reliable and precise method for quantifying the phenolic hydroxyl content of PPO based on differential UV spectroscopy was successfully developed. A mixture of dichloromethane and methanol (9:1) was used as the solvent to prevent PPO precipitation and minimize interference with the UV absorption of phenolic hydroxyl groups, whereas TEAH served as the alkaline ionization agent to fully ionize the phenolic hydroxyl groups in PPO. By subtracting the UV spectra before and after TEAH addition of TEAH, a differential UV spectrum was obtained, showing two absorption peaks at approximately 250 and 300 nm. The differential UV absorption near 300 nm was directly proportional to the phenolic hydroxyl concentration. software, enabling the establishment of a standard curve. A comparison of the curves constructed using DMP, BPA, and TMBPA as standards indicates that the DMP-based curve provided the most accurate results. This is attributed to the delocalization effect of the benzene rings in the PPO molecular chain from the ether bond to the phenoxide terminal group, which minimally influences the UV absorption. Using the spectral difference before and after ionization largely eliminated the effects of solvent interference and baseline drift. This methodology can be extended to determine the phenolic hydroxyl content in various polymer materials, offering valuable insights for material characterization and quality control.

Conflict of Interests

The authors declare no interest conflict.

Data Availability Statement

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

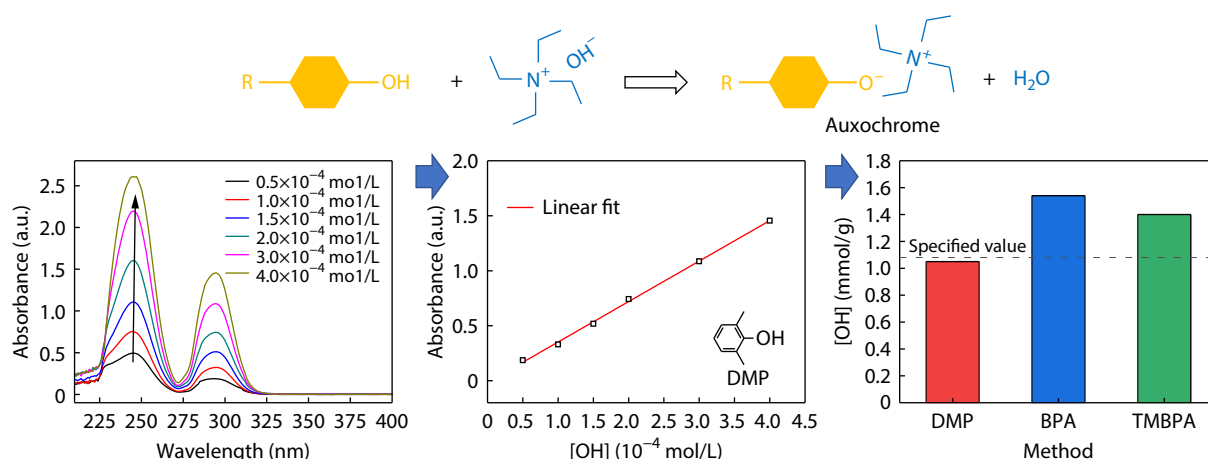
Graphical Abstract

Determination of Phenolic Hydroxyl Content in Poly(phenylene oxide) by Differential Ultraviolet Spectrophotometry

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This study presents a novel method for quantifying the phenolic hydroxyl content in poly(phenylene oxide) using differential UV absorption spectroscopy. The approach exploits the substantial increase in absorbance at 300 nm in alkaline solutions, providing a precise and straightforward technique with distinct advantages over conventional methods.



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REFERENCES

- Ouyang, Y. F.; Zhang, X. R.; Shi, J. H.; Yu, Y.; Liu, L. W.; Jia, Z. Q.; Wang, Q. Y.; Xu, H.; Wang, X. M. Diamine-modified bismaleimide for toughening of UV-cured epoxy acrylate coating. *Mater. Chem. Phys.* **2024**, 313, 128703.
- Li, A. Q.; He, H.; Shen, Y.; Li, Q. Y. High peel strength and excellent solder heat-resistance epoxy adhesive for flexible copper clad laminate. *J. Appl. Polym. Sci.* **2023**, 140, e54596.
- Liao, L. Y.; Ruan, W. H.; Zhang, M. Q.; Lin, M. S. Recent progress in modification of polyphenylene oxide for application in high-frequency communication. *Materials* **2024**, 17, 1086.
- Yu, J. B.; Chen, Y. C.; Zhong, W. Y.; Hu, D. D.; Jia, X. Y.; Sun, J. Y.; Jiang, Y.; Jiang, X. L.; Wang, H. F.; Zhao, L. Microcellular long-chain branched polyphenylene oxide (PPO) with excellent comprehensive properties and comprehensive analysis of its dielectric properties. *Eur. Polym. J.* **2024**, 214, 113157.
- Zhang, Z. Y.; Cai, K. P.; Liu, S. B.; Guo, W. H.; Zhang, B. S.; Yang, M. C.; Liu, W. T. The effect of HIPS-g-MAH on the mechanical properties of PA66/PPO alloy. *Polym. Bull.* **2022**, 79, 7939–7951.
- van Aert, H.A.M.; van Steenpaal, G.J.M.; Nelissen, L.; Lemstra, P.J.; Liska, J.; Bailly, C. Reactive compatibilization of blends of poly(2,6-dimethyl-1,4-phenylene ether) and poly(butylene terephthalate). *Polymer* **2001**, 42, 2803–2813.
- Chen, Y. H.; Xu, P. P.; Yu, S. H.; Luo, S. B. Active ester-functionalized polyphenylene oxide for low-dielectric and high-thermal-stability epoxy composites in advanced packaging. *ACS Appl. Polym. Mater.* **2025**, 7, 8821–8830.
- Hay, A. S. Poly(phenylene oxide)s and poly(arylene ether)s derived from 2,6-diarylphenols. *Prog. Polym. Sci.* **1999**, 24, 45–80.
- Zhou, Y. B.; Peng, Z. Q.; Ji, J. Q.; Wu, Y. C.; Chen, Z. G.; Huang, H. H.; Liu, S. M.; Zhao, J. Q. Fluorinated low molecular weight poly(phenylene oxide): synthesis, characterization, and application in epoxy resin toward improved thermal and dielectric properties. *Eur. Polym. J.* **2021**, 157, 110674.
- Wu, X. K.; Xu, C. A.; Lu, M. G.; Wang, K. X.; Li, Z.; Yang, H. Preparation and characterization of high temperature resistant thermosetting polyphenylene ether resin. *J. Appl. Polym. Sci.* **2022**, 139, e52858.
- Chen, C. H.; Gu, Z. C.; Tsai, Y. L.; Jeng, R. J.; Lin, C. H. Identification of the reaction mechanism between phenyl methacrylate and epoxy and its application in preparing low-dielectric epoxy thermosets with flexibility. *Polymer* **2018**, 140, 225–232.
- Shan, X. Y.; Huang, Q. X.; Wang, L. Z.; Diao, Y. X.; Li, J. C. Preparation and properties of low dielectric and flame-retardant epoxy resin/low molecular weight polyphenylene oxide composites. *Mater. Today Commun.* **2025**, 47, 113017.
- Krijgsman, J.; Feijen, J.; Gaymans, R. J. Synthesis and characterisation of telechelic poly(2,6-dimethyl-1,4-phenylene

<https://doi.org/10.1007/s10118-025-3463-y>

- ether) for copolymerisation. *Polymer* **2003**, *44*, 7055–7065.
- 14 Chan, K. P.; Argyropoulos, D. S.; White, D. M.; Yeager, G. W.; Hay, A. S. Facile quantitative analysis of hydroxyl end groups of poly(2,6-dimethyl-1,4-phenylene oxide)s by ³¹P NMR spectroscopy. *Macromolecules* **1994**, *27*, 6371–6375.
- 15 Wang, X. B.; Chen, G. B.; Zeng, X. P.; Zhang, Y. H. A method for determining the content of phenolic hydroxyl groups in polyphenylene ether. CN107356549A, China, 2017.
- 16 Pan, W. H. Determination of OH group content in polyphenylene oxide. *Anal Test Technol Instrum.* **2017**, *23*, 184–188.
- 17 Goldsch, O. Determination of phenolic hydroxyl content of lignin preparations by ultraviolet spectrophotometry. *Anal. Chem.* **1954**, *26*, 1421–1423.
- 18 Wu, B. G.; Zhao, J. L.; Du, J. B. Determination of phenolic hydroxyl group of lignin by ultraviolet spectrophotometry. *J. Northeast For. Univ.* **1993**, *21*, 58–65.
- 19 Chen, L.; Wei, X. X.; Wang, H.; Yao, M.; Zhang, L. M.; Gellerstedt, G.; Lindström, M. E.; EK, M.; Wang, S. F.; Min, D. Y. A modified ionization difference UV-Vis method for fast quantitation of guaiacyl-type phenolic hydroxyl groups in lignin. *Int. J. Biol. Macromol.* **2022**, *201*, 330–337.
- 20 White, D. M. The synthesis of 4-hydroxyarylene ethers by the equilibration of phenols with poly(2,6-dimethyl-1,4-phenylene ether). *J. Org. Chem.* **1969**, *34*, 297–303.