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SYNTHESIS AND SPECTROSCOPIC CHARACTERIZATION OF POLYETHYLENEPOLYAMINE-MODIFIED POLYACRYLONITRILE (PPA) FIBER FOR FUTURE METAL-ION ANALYSIS

Avazyazov Mukhammad [1], ***Ashirov Mansur*** [1]

[1] Khorezm Mamun Academy, Uzbekistan

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Abstract

Polyacrylonitrile (PAN) fiber was sequentially modified through alkaline hydrolysis, amination/amidoximation with hydroxylamine hydrochloride and hydrazinium chloride, grafting with polyethylenepolyamine (PEPA), and acid activation to produce a chemically functionalized PPA fiber suitable as a solid support for reagent immobilization. Ferron (7-iodo-8-hydroxyquinoline-5-sulfonic acid), a well-established chromogenic reagent for Fe(III), Fe(II), and Al(III) ions, was subsequently immobilized onto the PPA fiber by equilibrium sorption from a 0.0057 M aqueous solution at 25 °C for 24 h. The parent PAN, synthesized PPA, and Ferron-immobilized PPA fibers were characterized by Fourier-transform infrared (FTIR) spectroscopy and diffuse reflectance spectroscopy (DRS) using an EFI ES-2000 color calibrator spectrophotometer. FTIR analysis confirmed the partial conversion of nitrile groups (2241 cm^{-1}) into amide, amidoxime, and amine-related functionalities, with progressive spectral changes upon each modification step. Immobilization of Ferron introduced additional absorption bands consistent with aromatic and sulfonate functional groups. DRS measurements revealed a pronounced optical response in the 380–520 nm region for the Ferron-immobilized fiber, distinct from the unmodified PPA spectrum. These results establish that PPA fiber effectively retains Ferron on its surface and that the immobilized reagent retains its chromogenic functionality, providing a spectroscopic basis for the future development of a solid-phase colorimetric sensor for the qualitative and quantitative determination of Fe(III), Fe(II), and Al(III) ions in wastewater samples.

Keywords: *polyacrylonitrile fiber; PEPA grafting; Ferron immobilization; FTIR spectroscopy; diffuse reflectance spectroscopy; 7-iodo-8-hydroxyquinoline-5-sulfonic acid; metal-ion detection; solid-phase sensor; wastewater analysis*

1. Introduction

Heavy metal pollution of water resources is a pressing environmental concern worldwide. Iron and aluminum ions, while naturally abundant, can reach elevated concentrations in industrial effluents, mining drainage, and municipal wastewater, posing risks to aquatic ecosystems and human

health (Deng S., Bai R., and Chen J. P., 2003; Burakov A. E., Galunin E. V., Burakova I. V., et al., 2018). The development of simple, rapid, and cost-effective analytical methods for monitoring these ions is therefore of considerable practical importance.

Spectrophotometric methods based on chromogenic reagents offer a well-established approach for metal-ion detection. Among the available reagents, 7-iodo-8-hydroxyquinoline-5-sulfonic acid, commonly known as Ferron, has been widely used for the colorimetric determination of Fe(III), Fe(II), and Al(III) owing to its ability to form stable, intensely colored complexes with these ions (Cheng K. L., Ueno K., and Imamura T., 1992; Moldovan Z. and Neagu E. A., 2002). The selectivity and sensitivity of Ferron-based methods have led to their application in diverse matrices, including natural waters, soils, and biological fluids (Parker D. R. and Bertsch P. M., 1992; Goto K., Tamura H., Onodera M., and Nagayama M., 1974).

In solution-phase colorimetry, the reagent is consumed in a single measurement, generating chemical waste and requiring frequent reagent preparation. Immobilization of chromogenic reagents on solid supports addresses these limitations by enabling reagent reuse, simplifying separation from the sample matrix, and facilitating integration into solid-phase sensing platforms (Ruvubu S. B. and Roy I., 2026; Rahman I. M. M., Begum Z. A., and Hasegawa H., 2013). The choice of support material is critical: it must provide abundant surface functional groups for reagent attachment, exhibit chemical and mechanical stability, and permit optical interrogation of the immobilized reagent.

Polyacrylonitrile (PAN) fiber is an attractive candidate for this purpose. PAN is produced on an industrial scale, possesses good mechanical properties, and contains nitrile groups that can be chemically transformed into a variety of functional moieties (Bashir Z., 1991; Shin D. H., Ko Y. G., Choi U. S., and Kim W. N., 2004). Alkaline hydrolysis converts a portion of the nitrile groups to carboxylate and amide functionalities, increasing hydrophilicity and providing anchoring points for further modification. Subsequent amination with hydroxylamine hydrochloride introduces amidoxime groups, which are known for their metal-chelating ability (Saeed K., Haider S., Oh T.-J., and Park S.-Y., 2008; Huang F., Xu Y., Liao S., Yang D., Hsieh Y.-L., and Wei Q., 2013). Grafting with polyethylenepolyamine (PEPA) further increases the density of amine-containing binding sites, creating a highly functionalized surface (Shahidova D. and Gafurova D., 2024).

In this study, we report the sequential synthesis of a PEPA-modified PAN (PPA) fiber and its use as a support for the immobilization of Ferron. The chemical transformations at each stage were monitored by FTIR spectroscopy, and the optical properties of the Ferron-immobilized fiber were characterized by diffuse reflectance spectroscopy (DRS). The overall objective is to establish the spectroscopic basis for a subsequent application in which the Ferron-PPA fiber is employed for the qualitative and quantitative determination of Fe(III), Fe(II), and Al(III) ions in wastewater samples using color change analysis with a calibrated DRS instrument.

2. Materials and Methods

2.1 Materials

Polyacrylonitrile (PAN) fiber was used as the base polymer. Sodium hydroxide (NaOH), hydroxylamine hydrochloride (CAS 5470–11–1), hydrazinium chloride (CAS 2644–70–4), dimethylformamide (DMF), polyethylenepolyamine (PEPA, 50% w/w aqueous solution), hydrochloric acid (HCl), and Ferron (7-iodo-8-hydroxyquinoline-5-sulfonic acid, CAS 547–91–1) were used as received. All solutions were prepared with bidistilled water.

2.2 Synthesis of PPA Fiber

The synthesis followed a four-step route: alkaline hydrolysis of PAN, amination/amidoximation, PEPA grafting, and acid activation. A 2.0 g portion of PAN fiber was first treated with 80 mL of 1 M NaOH at a bath ratio of 1:40 for 60 min at 90 °C. This treatment was intended to increase hydrophilicity and generate carboxylate and amide sites from a portion of the nitrile groups. The hydrolyzed fiber was washed five times with bidistilled water and dried at ambient temperature.

The hydrolyzed fiber was then aminated in 100 mL of reaction solution containing 6.0 g hydroxylamine hydrochloride, 0.7 g hydrazinium chloride, and 5.0 mL DMF. The pH

was adjusted to 8.0–8.5 with 1 M NaOH to form the active nucleophilic species. The reaction was maintained at 90 °C for 1.5 h with continuous stirring. Hydrazinium chloride was used in place of hydrazine hydrate to improve handling safety and storage stability while preserving the intended cross-linking function. After reaction, the fiber was washed five times with bidistilled water and dried at ambient temperature.

For PEPA grafting, the aminated fiber was immersed in 80 mL of 50% (w/w) PEPA solution at a bath ratio of 1:40. The reaction was conducted at 100 °C for 5 h under continuous stirring. The elevated temperature and extended reaction time were selected to support diffusion of PEPA into the fiber matrix and to increase the density of amine-containing binding sites. The obtained PPA fiber was washed exhaustively five times with bidistilled water and dried at ambient temperature.

Before Ferron immobilization, the dried PPA fiber was activated in 0.1 M HCl at a bath ratio of 1:75 for 24 h at 25 °C. For a 2.0 g fiber sample, 150 mL of acid solution was used. This step protonates amine groups and removes weakly retained oligomeric residues. The activated PPA fiber was washed with bidistilled water and dried at ambient temperature.

Table 1. *Summary of experimental conditions for each stage of PPA fiber synthesis and Ferron immobilization*

Stage	Experimental Conditions
Alkaline hydrolysis	PAN fiber (2.0 g); 80 mL of 1 M NaOH; bath ratio 1:40; 90 °C; 60 min; washed 5×, dried
Amination	Hydrolyzed fiber in 100 mL medium; 6.0 g $NH_2OH·HCl$, 0.7 g $N_2H_4·2HCl$, 5.0 mL DMF; pH 8.0–8.5; 90 °C; 1.5 h
PEPA grafting	Aminated fiber in 80 mL of 50% (w/w) PEPA; bath ratio 1:40; 100 °C; 5 h; washed, dried
Acid activation	Dried PPA fiber in 0.1 M HCl; bath ratio 1:75; 24 h at 25 °C; washed, dried
Ferron immobilization	PPA fiber in 20 mL of 0.0057 M Ferron; 24 h at 25 °C; characterized spectroscopically

2.3 Ferron Immobilization

Ferron was immobilized by immersing the activated PPA fiber in 20 mL of 0.0057 M Ferron solution for 24 h at 25 °C. After immobilization, the fiber was removed from the solution, rinsed briefly with bidistilled water to remove non-adsorbed reagent, and dried at ambient temperature prior to spectroscopic characterization.

2.4 Instrumental Characterization

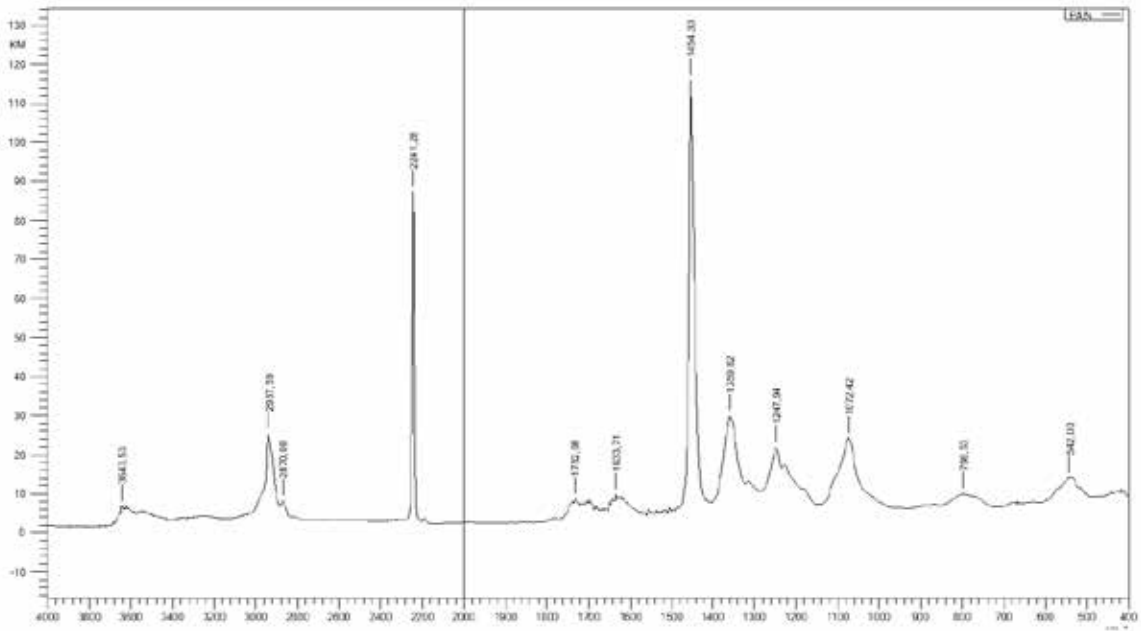
Fourier-transform infrared (FTIR) spectra of the parent PAN fiber, synthesized PPA fiber, and Ferron-immobilized PPA fiber were recorded using a Shimadzu IRTracer-100 FTIR spectrophotometer in the wavenumber range 400–4000 cm^{-1}. Diffuse reflectance spectra (DRS) of PPA and Ferron-immobilized PPA fibers were obtained with an EFI ES-2000 color calibrator spectrophotometer to monitor the optical change produced by Ferron immobilization.

3. Results and Discussion

3.1 FTIR Analysis of PAN Fiber

The FTIR spectrum of the initial PAN fiber is presented in (Figure 1). The characteristic nitrile (C≡N) stretching vibration was observed at 2241 cm^{-1}, together with aliphatic C–H stretching bands near 2938 and 2870 cm^{-1}. Additional bands appeared at 1454, 1359, 1248, 1072, 799, and 542 cm^{-1}, corresponding to the polymer backbone and various bending and vibrational modes of the unmodified PAN matrix (Bajaj P., Sreekumar T. V., and Sen K., 2002). These features are consistent with the well-documented FTIR profile of commercial PAN fiber (Bilba N., Bilba D., and Moroi G., 2004).

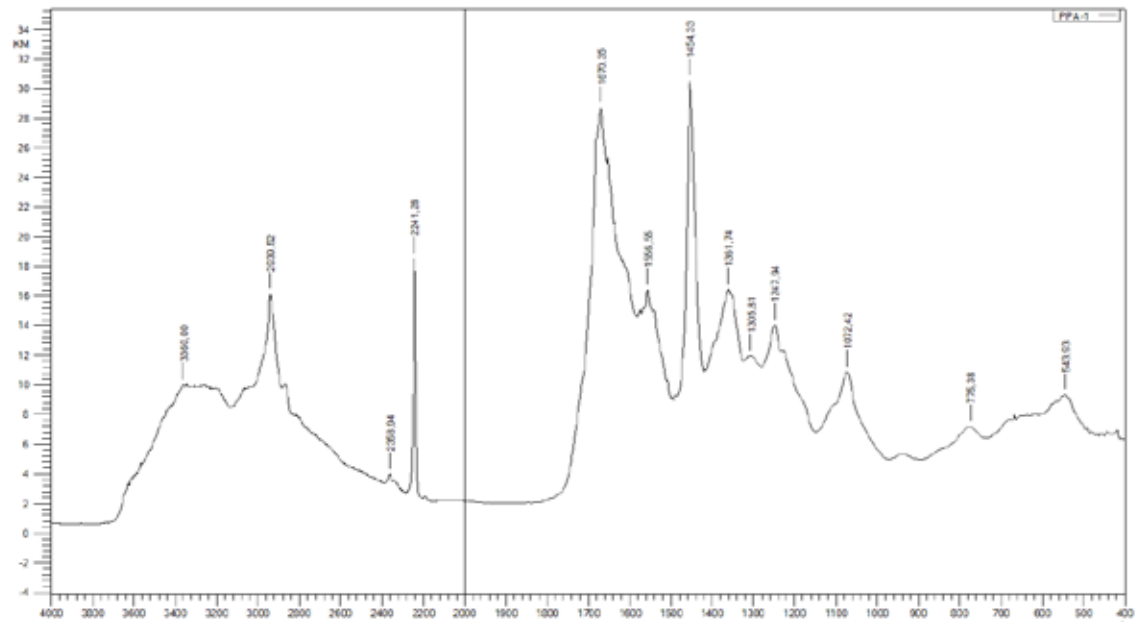
Figure 1. *FTIR spectrum of the initial PAN fiber before chemical modification, showing the characteristic nitrile absorption at 2241 cm^{-1}*



3.2 FTIR Analysis of Synthesized PPA Fiber

After conversion to PPA fiber through the sequential modification steps, the FTIR profile changed substantially (Figure 2). A broad band centered near 3360 cm-1 appeared, indicating the introduction of hydroxyl (O–H) and amine (N–H) functionalities. The nitrile band at 2241 cm-1 was still detectable but reduced in relative prominence, suggesting partial conversion rather than complete consumption of the PAN nitrile groups. This partial conversion is beneficial because the remaining nitrile segments preserve fiber mechanical integrity while the newly introduced polar groups provide chemical functionality (Saeed K., Haider S., Oh T.-J., and Park S.-Y., 2008).

Figure 2. *FTIR spectrum of synthesized PPA fiber after hydrolysis, amination, PEPA grafting, and acid activation, showing the appearance of new bands related to nitrogen- and oxygen-containing functional groups*

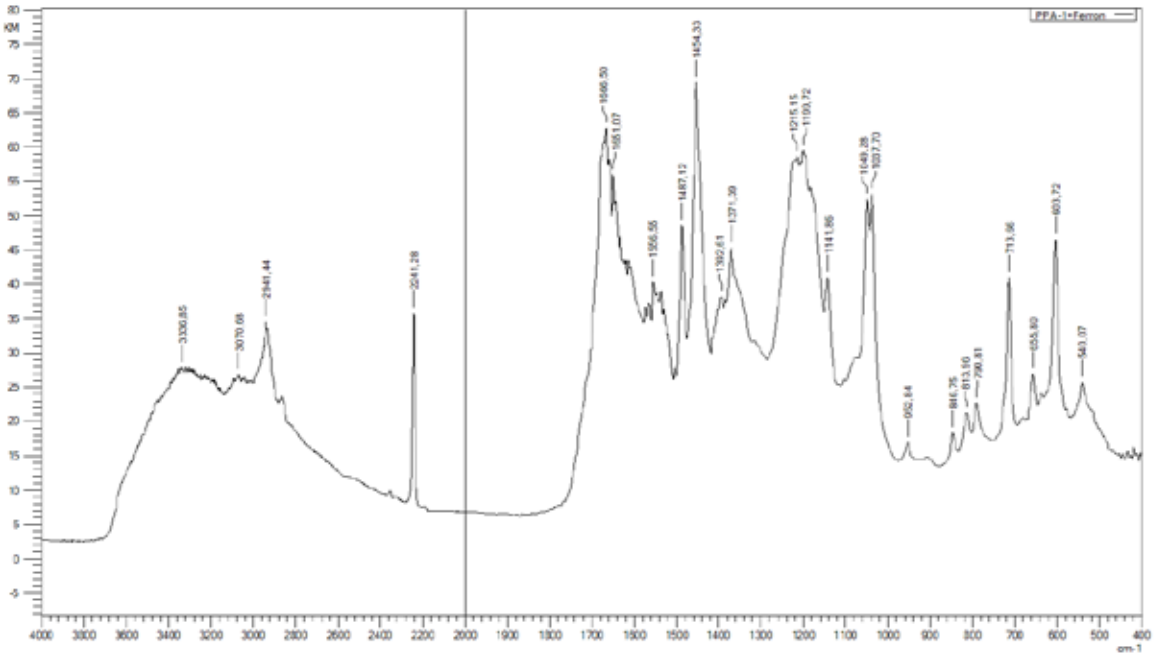


New and intensified bands were observed at 1670, 1557, 1454, 1362, 1306, 1248, 1072, 775, and 544 cm^{-1}. The bands at 1670 and 1557 cm^{-1} are assigned to C=O stretching (amide I) and N–H bending (amide II) of amide and amidoxime groups, respectively (Huang F., Xu Y., Liao S., Yang D., Hsieh Y.-L., and Wei Q., 2013; Zhao H., Liu X., Yu M. Wang Z., Zhang B., Ma H., Wang M., and Li J., 2015). The broad absorption in the 1300–1000 cm^{-1} region includes C–N stretching vibrations from amine and amidoxime functionalities. These spectral changes confirm the successful introduction of amide, amidoxime, imine/amine, and PEPA-associated functional groups on the fiber.

3.3 FTIR Analysis of Ferron-Immobilized PPA Fiber

Ferron immobilization produced further changes in the FTIR spectrum (Figure 3). The broad high-wavenumber region was retained, with features near 3337, 3071, and 2941 cm^{-1}. Strong bands appeared at 1667, 1651, 1557, 1487, 1454, 1393, 1371, 1215, 1191, 1142, 1049, 1038, 953, 846, 814, 790, 714, 656, 604, and 540 cm^{-1}. The bands in the 1200–1000 cm^{-1} region are particularly informative: the sulfonic acid group (–SO_3H) of Ferron typically exhibits strong S=O stretching vibrations in this range (NIST Chemistry WebBook.). Bands near 1487 and 1454 cm^{-1} are consistent with aromatic C=C stretching of the quinoline ring system. These spectral features indicate that Ferron was successfully retained on the PPA matrix, with its molecular structure intact after immobilization.

Figure 3. *FTIR spectrum of PPA fiber after immobilization with Ferron (7-iodo-8-hydroxyquinoline-5-sulfonic acid), showing additional bands attributable to aromatic and sulfonate groups*

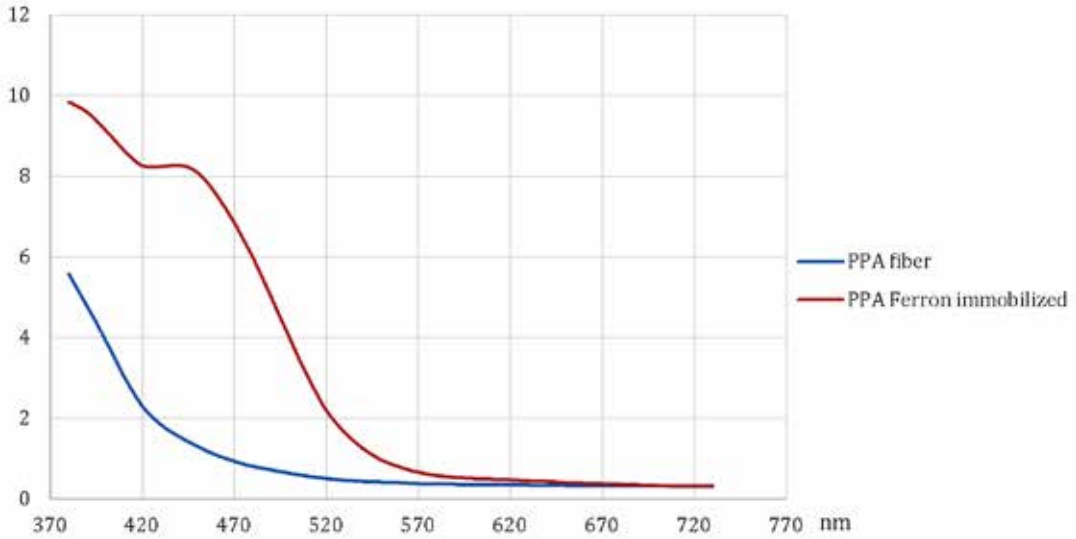


3.4 Diffuse Reflectance Spectroscopy

Diffuse reflectance spectroscopy (DRS) confirmed a pronounced visible optical change after Ferron immobilization (Figure 4). The unmodified PPA fiber showed relatively low reflectance across the visible range, with a gradual decrease from shorter to longer wavelengths. In contrast, the Ferron-immobilized PPA fiber displayed a significantly stronger reflectance response in the 380–520 nm region, followed by a pronounced decrease toward the baseline beyond approximately 520–570 nm. The two spectra converged at longer wavelengths, indicating that the major optical contribution of the immobilized Ferron occurs in the short-to-mid visible wavelength range.

The enhanced reflectance in the blue-green region is consistent with the known absorption properties of Ferron and its metal complexes (Cheng K. L., Ueno K., and Imamura T., 1992; Moldovan Z. and Neagu E. A., 2002). This optical response provides the basis for the intended use of the Ferron-PPA fiber as a solid-phase colorimetric sensor: when exposed to Fe(III), Fe(II), or Al(III) ions in aqueous solution, the formation of metal–Ferron complexes on the fiber surface is expected to alter the reflectance spectrum in a concentration-dependent manner, enabling quantitative analysis. The differential optical response between the immobilized Ferron and its metal complexes can be captured by the EFI ES-2000 color calibrator spectrophotometer and correlated with metal-ion concentration.

Figure 4. *Diffuse reflectance spectra of synthesized PPA fiber and Ferron-immobilized PPA fiber recorded with an EFI ES-2000 color calibrator spectrophotometer. The enhanced response at 380–520 nm is attributable to the immobilized Ferron*



4. Discussion

The sequential modification strategy described in this study successfully converted PAN fiber into a chemically activated PPA fiber suitable for reagent immobilization. The alkaline hydrolysis step likely generated a more hydrophilic surface and introduced carboxylate and amide sites, making the fiber more accessible to subsequent nucleophilic modification. The amination step introduced nitrogen-containing functional groups, while PEPA grafting increased the density of amine sites that can participate in electrostatic, hydrogen-bonding, and coordination-related interactions with the Ferron molecule (Shahidova D. and Gafurova D., 2024).

The FTIR results are consistent with this interpretation. The retention of the nitrile band at 2241 cm-1 in the PPA spectrum indicates that the reaction conditions produced a partially modified PAN backbone rather than complete degradation or exhaustive conversion. This is advantageous for fiber stability, because unconverted PAN segments can preserve mechanical integrity while the newly introduced amine, amidoxime, and amide-related groups provide chemical functionality. The broad 3360 cm-1 band and the intensified mid-infrared absorptions in the PPA spectrum are consistent with successful introduction of polar groups as reported in similar modification studies (Saeed K., Haider S., Oh T.-J., and Park S.-Y., 2008; Huang F., Xu Y., Liao S., Yang D., Hsieh Y.-L., and Wei Q., 2013; Zhao H., Liu X., Yu M. Wang Z., Zhang B., Ma H., Wang M., and Li J., 2015).

Ferron immobilization is the critical step that enables the future application of this material for metal-ion sensing. Ferron forms colored complexes with Fe(III), Fe(II), and Al(III) through coordination involving the 8-hydroxyquinoline moiety (Cheng K. L., Ueno K., and Imamura T., 1992; Moldovan Z. and Neagu E. A., 2002). The FTIR spectrum after immobilization showed additional bands attributable to aromatic and sulfonated Ferron

groups, and the DRS spectrum demonstrated a strong optical response in the visible region compared with unmodified PPA. Together, these results confirm that Ferron was retained on the PPA matrix and that immobilization changed the reflectance behavior of the fiber in a measurable and reproducible way.

The choice of PEPA as the grafting agent merits specific discussion. PEPA is a branched polyamine containing primary, secondary, and tertiary amine groups, providing multiple types of binding sites for the immobilization of anionic or zwitterionic species such as Ferron (which carries a sulfonate group at pH > 2–3). Shahidova and Gafurova (Shahidova D. and Gafurova D., 2024) reported that PEPA modification of hydroxylamine-activated PAN fiber yielded a sorbent with a static exchange capacity of up to 5.4 mg-eq/g, indicating a high density of functional groups. The acid activation step (0.1 M HCl, 24 h) employed in the present work serves to protonate these amine groups, generating positively charged ammonium sites that can interact electrostatically with the negatively charged sulfonate group of Ferron, thereby enhancing reagent retention.

The DRS results are particularly notable because they demonstrate that Ferron retains its chromogenic properties after immobilization. In solution-phase applications, Ferron exhibits strong absorption in the near-UV to blue region of the spectrum (Cheng K. L., Ueno K., and Imamura T., 1992). The enhanced reflectance observed in the 380–520 nm range for the immobilized Ferron corresponds to the same electronic transitions. This indicates that the immobilization process does not disrupt the conjugated π-electron system responsible for the chromogenic behavior. The convergence of the PPA and Ferron-PPA spectra at longer wavelengths confirms that the observed difference is specifically due to the immobilized reagent rather than any modification-induced changes in the PPA substrate itself.

It should be noted that the present study focused on the synthesis and preliminary spectroscopic characterization of the Ferron-PPA material. For practical application to wastewater analysis, additional studies are required to establish: (i) the optimal pH conditions for metal-ion binding and color development; (ii) calibration curves relating metal-ion concentration to the DRS response; (iii) detection limits and linear dynamic ranges for each target metal ion; (iv) selectivity and interference from coexisting ions; (v) repeatability and batch-to-batch reproducibility; and (vi) recovery from spiked real wastewater samples. These studies are the subject of ongoing work and will be reported in a subsequent publication.

5. Conclusion

A PEPA-modified polyacrylonitrile (PPA) fiber was successfully synthesized through a sequential four-step route involving alkaline hydrolysis, amination/amidoximation, PEPA grafting, and acid activation. Ferron (7-iodo-8-hydroxyquinoline-5-sulfonic acid) was subsequently immobilized onto the PPA fiber from aqueous solution.

FTIR spectroscopy confirmed the chemical transformations at each stage: (i) partial retention of the PAN nitrile groups with reduced intensity; (ii) appearance of a broad O–H/N–H band near 3360 cm-1; (iii) formation of amide (1670 cm-1) and amidoxime/amine (1557 cm-1) bands; and (iv) introduction of aromatic and sulfonate bands characteristic of Ferron after immobilization.

Diffuse reflectance spectroscopy revealed a distinct optical response from the Ferron-immobilized fiber in the 380–520 nm region, confirming that the immobilized reagent retains its chromogenic functionality. The macroscopic yellow coloration of the Ferron-PPA fiber provided qualitative visual confirmation of successful immobilization.

These results establish that PPA fiber is an effective solid support for Ferron immobilization and that the resulting material exhibits the spectroscopic properties necessary for the development of a solid-phase colorimetric sensor. Future work will focus on establishing the analytical performance characteristics of the Ferron-PPA fiber for the determination of Fe(III), Fe(II), and Al(III) ions in real and spiked wastewater samples.

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Contact: 1avazyazovmuhammad@gmail.com