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DEVELOPMENT OF A FUNCTIONAL MATERIAL BASED ON TIRON-IMMOBILIZED FILTER PAPER FOR THE DETERMINATION OF SILVER(I) IONS AND ITS SPECTROSCOPIC CHARACTERIZATION

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Abstract

This study reports the immobilization of the organic reagent Tiron onto filter paper to create a simple and low-cost optical platform for the determination of silver(I) ions. Immobilization was performed by soaking the paper in an aqueous Tiron solution (1.0×10^{-3} mol/L) followed by drying at room temperature. The resulting material was treated with Ag(I) ions and characterized by diffuse reflectance spectroscopy (DRS) and Fourier-transform infrared (FTIR) spectroscopy. The DRS spectra revealed a pronounced change in the optical response both after Tiron immobilization and after the binding of Ag(I) ions, confirming the formation of surface coordination complexes. FTIR analysis confirmed the successful immobilization of Tiron onto the cellulose matrix and the participation of the hydroxyl and sulfonate functional groups in coordination with Ag(I) ions. The developed Tiron-functionalized filter paper is inexpensive, environmentally safe, and easy to prepare, making it a promising material for the sorption of silver(I) ions and for the fabrication of paper-based optical sensors.

Keywords: Tiron; filter paper; immobilization; cellulose; silver(I); chelate complex; paper-based sensor; sorbent

Introduction

In recent years, interest in paper-based materials for the detection, separation, and on-site preconcentration of metal ions has increased steadily. This interest is driven by

the low cost and biodegradability of filter paper, its ability to transport solutions by capillary flow, the ease of cutting and zone patterning, and minimal reagent consumption. When organic ligands, nanomaterials, or

ion-selective reagents are immobilized onto a paper matrix, the paper can serve as a simple test platform suitable for use both in the laboratory and under field conditions.

Tiron is a catechol disulfonate ligand well known in analytical chemistry because it forms colored complexes with Fe, Ti, Al, and other metal ions. Its two phenolic –OH groups can coordinate to a metal center, while the sulfonate groups provide water solubility and an ion-exchange environment. When Tiron is immobilized onto filter paper, a metal-binding functional layer is formed on the surface of the cellulose fibers. The attachment of Ag to this layer may provide the basis for a paper-based sorbent, indicator material, antimicrobial material, or a platform for subsequent nanocomposite synthesis.

The main scientific question of this study is formulated as follows: is the binding of Ag on Tiron-modified filter paper due to simple physical adsorption, to the formation of an Ag+–Tiron complex, to the formation of Ag(0) nanoparticles, or to a combination of these processes? The literature review was aimed specifically at substantiating this question, establishing the criteria for the subsequent experiments, and shaping the theoretical part of the article.

Literature Review

Properties of Tiron as a Ligand

The principal structural advantage of Tiron is its catechol fragment: upon deprotonation, the two adjacent hydroxyl groups can provide bidentate coordination to metal ions. The disulfonate groups render the molecule hydrophilic, making Tiron a convenient analytical reagent that operates in aqueous media. References (National Center for Biotechnology Information.; Dojindo Molecular Technologies, Inc. Tiron (T021)) describe Tiron as the disodium salt of composition $C_6H_4Na_2O_8S_2$ and characterize it as a reagent that forms colored chelates with Fe, Al, Ti, and other metal ions.

Recent studies of the Fe(III)–Tiron system show that Tiron forms complexes with the metal at various stoichiometries and that the color of the complexes is pH-dependent (Nucera, A., Carniato, F., Baranyai, Z., Platas-Iglesias, C., Botta, M., 2023). This indicates that, when working with Ag, the effects of

pH, ligand-to-metal ratio, ionic strength, and competing cations must be investigated separately, because the complexation ability of Tiron is governed not only by the presence of the ligand but also by the protonation state of its phenolic groups and by the conformational freedom retained after immobilization onto a surface.

Tiron is not among the best-known selective reagents for Ag+; moreover, although its electron-donor catechol moiety is not fully matched to Ag+ according to hard–soft acid–base theory, it can create favorable conditions for coordination bonding, electrostatic interaction, and subsequent reduction processes. For this reason, characterization of the Ag-bound Tiron–paper material cannot rely on a mere color change or mass increase; methods that confirm the oxidation state of Ag are required.

Role of Filter Paper and the Cellulose Matrix

The main component of filter paper is cellulose, which is amenable to modification owing to its numerous hydroxyl groups, fibrous porous structure, and capillary properties. In paper microfluidic and test-strip devices, paper serves as a passive platform that transports the liquid, retains the reagent, and confines the reaction to a visible zone. Reviews on paper-based microfluidics show that paper is a low-cost, flexible material well suited to point-of-care analysis (Li, X., Ballerini, D. R., Shen, W., 2012; Yetisen, A. K., Akram, M. S., Lowe, C., 2013; Costa, M. N., Veigas, B., Jacob, J. M., Santos, D. S., Gomes, J., Baptista, P. V., Martins, R., Inácio, J., Fortunato, E., 2014).

Numerous studies on cellulose-based adsorbents report that the introduction of carboxyl, amino, thiol, phosphate, sulfonate, or chelating groups enhances the ability to capture metal ions (Shaghaleh, H., Xu, X., Wang, S., 2018; Abdelhamid, H. N., Mathew, A. P., 2021; Si, R., Pu, J., Luo, H., Wu, C., Duan, G., 2022). Fakhre and Ibrahim applied chemically modified cellulose to the adsorption of heavy metal ions; such an approach methodologically supports the idea of immobilizing a chelating ligand such as Tiron onto a cellulose surface (Fakhre, N. A., Ibrahim, B. M., 2018).

When Tiron is immobilized onto filter paper, two types of problems arise. The first is preventing the reagent from being washed out: because Tiron is highly water-soluble, a simple soaking procedure may not be sufficient for permanent immobilization. The second is avoiding blockage of the metal-binding active sites of the ligand: if covalent attachment or coating with a polymer layer blocks the catechol groups of Tiron, Ag binding will decrease. It is therefore essential to demonstrate by FTIR the efficiency of the immobilization procedure, the mass stability of the material upon washing, and the preservation of the functional groups of the ligand.

Ag⁺ Detection and Paper-Based Silver Platforms

Silver is widely used in industry, photochemistry, electronics, medicine, and antimicrobial materials. At the same time, monitoring Ag⁺ ions in water and food samples is important because excess silver poses environmental and toxicological risks. Fan et al. proposed a paper test-strip platform modified with black phosphorus quantum dots for the detection of Ag⁺ in drinking water, combining fluorometric and colorimetric signals (Fan, X., Lv, J., Li, R., Chen, Y., Zhang, S., Liu, T., Zhou, S., Shao, X., Wang, S., Hu, G., Yue, Q., 2023).

A paper-based colorimetric array test-strip approach has also been developed for the simultaneous detection of multiple metal ions (Liu, L., Lin, H., 2014). These studies demonstrate that metal ions can be detected visually or semi-quantitatively using reagents immobilized on paper. The Tiron–Ag–paper system could join this family of platforms; its advantages include the ready availability of Tiron as a simple organic ligand, the low cost of filter paper, and the possibility that additional optical or antimicrobial properties emerge on the surface after Ag binding.

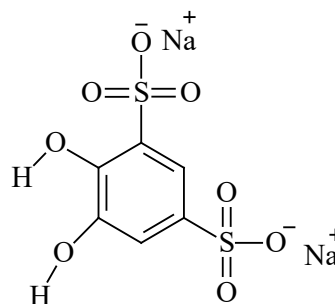
The retention of Ag⁺ on paper can occur through several mechanisms: weak coordination with the –OH groups of cellulose, complexation with the phenolate catechol groups of Tiron, electrostatic interaction near the sulfonate groups, or the formation of Ag(0) nanoparticles under reducing conditions. If the paper treated with an AgNO₃ solution changes color or darkens during the

experiment, this also points to the possible photochemical or chemical reduction of Ag⁺.

Experimental Section

Standard solutions of the analytical reagents. Tiron (disodium salt of 1,2-dihydroxybenzene-3,5-disulfonic acid, CAS 149–45–1) was selected as the immobilized analytical reagent. The substance was obtained in analytical grade from Shanghai Macklin Biochemical Co., Ltd. (China). A 1×10^{-3} M standard solution of Tiron was prepared in distilled water and used in the immobilization procedures. The molecular structure of the Tiron salt is shown in Figure 1.

Figure 1. Molecular structure of Tiron (disodium salt of 1,2-dihydroxybenzene-3,5-disulfonic acid)



Buffer solutions. An acetate buffer solution was used to maintain the required pH of the systems under study. In analytical chemistry and in sensor-related work, the acetate buffer system (pH range 3.7–5.6) is one of the most commonly used stable buffer systems. It is prepared from a mixture of a weak acid (acetic acid, CH₃COOH) and its salt (sodium acetate, CH₃COONa).

Preparation of the polymer matrix. Before immobilization of the Tiron reagent (disodium salt of 1,2-dihydroxybenzene-3,5-disulfonic acid), the filter paper was cut into circles with a diameter of 2 cm. The resulting filter paper samples were first washed with distilled water and then dried at room temperature.

Equipment. The following instruments were used to examine the samples during the experiments: an Eye-One Pro spectrophotometer (ES-2000), a UV–Vis spectrophotometer (Shimadzu 1900i), a pH meter (Mettler-Toledo GmbH), a magnetic stirrer (MS7-H550-Pro), and an infrared spectrometer (Shimadzu IRAffinity-1S). The deionized

water used in the experiments was obtained from an EASYpure RoDI water purification system (Thermo Scientific, USA).

Immobilization procedure. To modify the cellulose matrix and achieve the maximum adsorption equilibrium of the Tiron reagent, the prepared filter paper samples were soaked in an aqueous Tiron solution at a concentration of 1×10^{-3} M for 24 h at room temperature. This period ensures the deep diffusion of Tiron molecules into the micro- and macropores of the cellulose and the formation of stable hydrogen bonds with the hydroxyl groups of the matrix fibers. After removal from the Tiron solution, the immobilized filter papers were separated from the excess solvent using a filter cassette and dried to constant mass at room temperature in a dark place for 24 h. This regime ensured the complete evaporation of free and bound water molecules from the matrix and the maximum stabilization of the IR spectral characteristics.

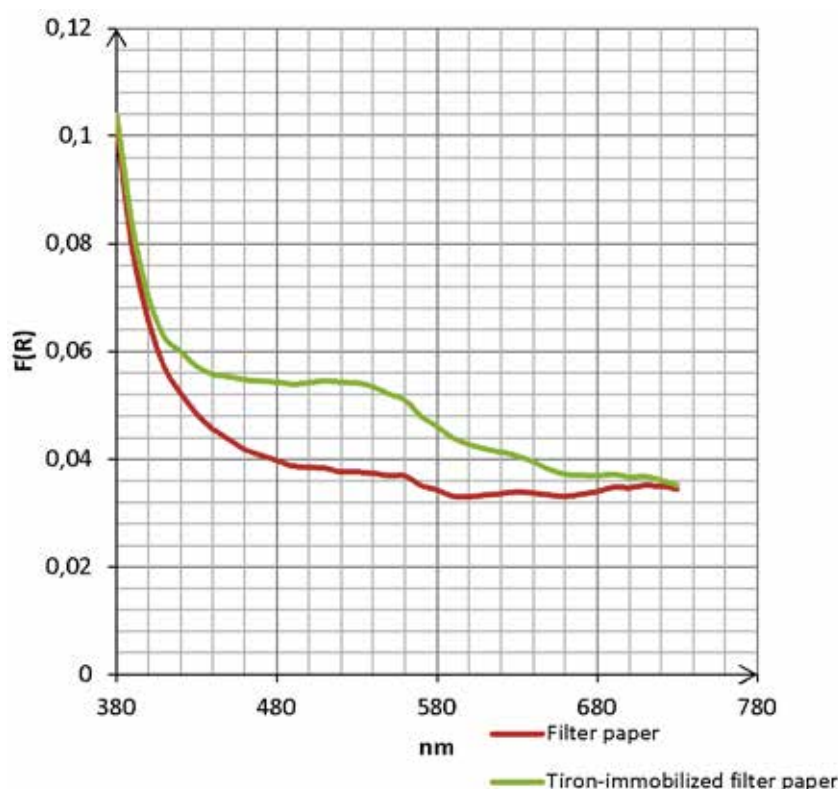
A 100 mL acetate buffer solution with pH 4.71 was prepared, and AgNO_3 was dissolved

in it to obtain a 0.001 M Ag^+ solution. The solution was prepared at room temperature on a magnetic stirrer under dark conditions. The Tiron-immobilized filter paper was immersed in the resulting solution for 1–3 min in a dark room at +4 to +8 °C. The filter paper was then removed from the solution and dried in a dark room at 20–25 °C for 24 h. If the drying step is not performed in a dark room, Ag^+ is reduced and converted to Ag^0 .

Optical Spectral Analysis

The optical response of the Tiron-immobilized filter paper and of the Filter paper+Tiron+Ag(I) complex links the theoretical considerations of the literature review to the experimental results. The graphs show the variation of the Kubelka–Munk function $F(R)$ over the 380–730 nm range. These spectra clearly demonstrate the differences between plain filter paper, Tiron-immobilized filter paper (Filter paper+Tiron), and Tiron-modified paper with sorbed Ag(I) ions (Filter paper+Tiron+Ag(I)).

Graph 1. $F(R)$ spectra of plain filter paper and Tiron-immobilized filter paper. After Tiron immobilization, the absorption/reflectance function in the 400–650 nm range is higher than that of plain paper



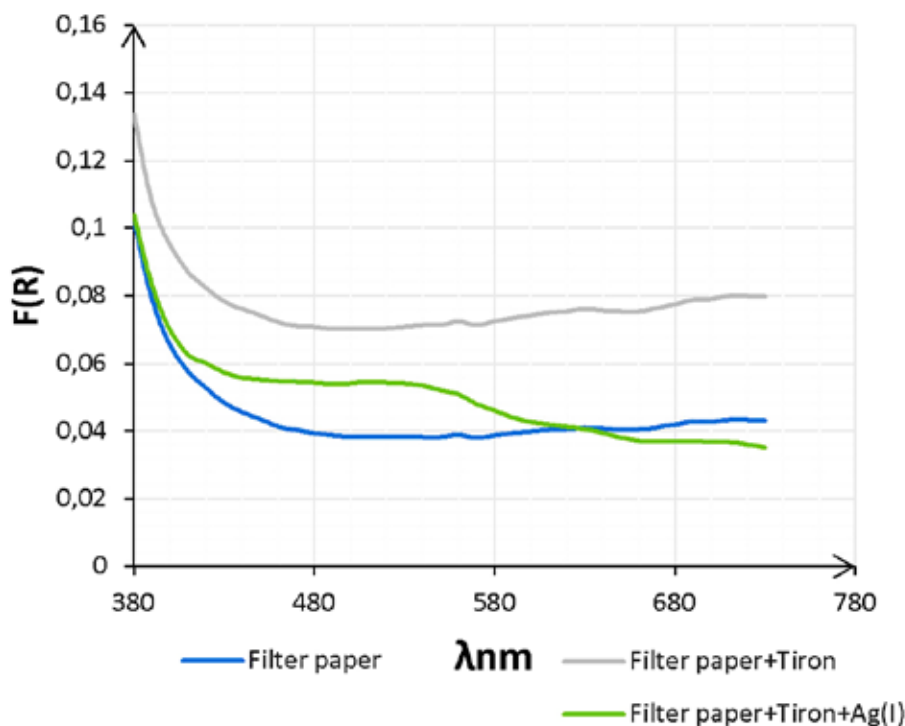
As can be seen from Graph 1, filter paper treated with Tiron exhibits higher $F(R)$ val-

ues than plain filter paper over almost the entire visible range. In particular, in the 420–

–560 nm range, the curve of the Tiron-immobilized paper is clearly distinct from that of plain paper. This indicates that Tiron molecules are retained on the surface of the cellulose fibers and that the optical properties of

the paper have changed. Although the Tiron solution and the Tiron-immobilized paper appear colorless to the naked eye, the spectral measurements reveal the modification with higher sensitivity.

Graph 2. *F(R)* spectra of filter paper, Filter paper+Tiron, and Filter paper+Tiron+Ag(I) samples. The change in the shape of the curve after the uptake of Ag(I) ions indicates the formation of a silver-containing surface complex/compound



In Graph 2, three samples are compared: plain filter paper, Filter paper+Tiron, and Filter paper+Tiron+Ag(I). The Filter paper+Tiron sample exhibits a higher optical response than plain filter paper, confirming that Tiron is immobilized on the carrier surface. After the uptake of Ag(I) ions, the curve of Filter paper+Tiron+Ag(I) differs sharply from that of Filter paper+Tiron: the signal is higher in the 380–550 nm range, with a decrease toward 560–730 nm. This change indicates that an interaction has occurred between the functional groups of Tiron and the Ag(I) ions.

Together, the color change and the spectral differences observed after the uptake of Ag(I) indicate that the Tiron-immobilized paper acts as a surface sensitive to silver ions. The color development may be associated with the formation of an Ag(I)–Tiron complex, the coordination binding of Ag(I) to the cellulose/Tiron surface, or, under certain conditions, the partial formation of Ag(0) particles.

The presented results show that the immobilization of Tiron on filter paper enhances the spectral response relative to plain paper and that the optical response acquires a new character after the uptake of Ag(I) ions. Thus, Tiron-immobilized filter paper can be regarded as a low-cost cellulosic platform for the sorption of Ag(I) ions with a visible color response. These results are consistent with the complexation behavior of Tiron with metal ions reported in the literature and with the practical advantages of paper-based sensors.

IR/FTIR Spectral Analysis

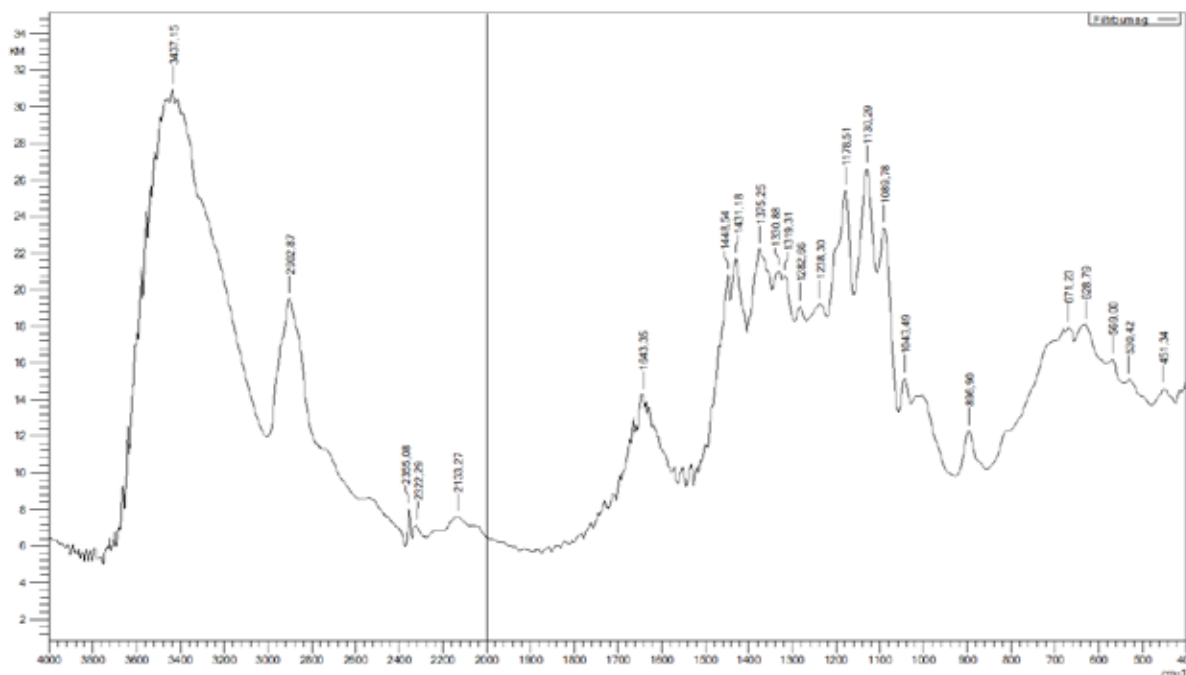
Infrared spectroscopy (IR or FTIR) was used to identify the functional groups in the samples and to assess changes in their chemical environment after the modification process. The spectra of filter paper, Tiron-immobilized filter paper, and the Filter paper+Tiron+Ag complex are interpreted in-

dividually below. The spectra were recorded in the 4000–400 cm^{-1} range, with particular attention to the O–H, C–H, aromatic ring,

C–O/C–O–C, S=O/S–O, and low-frequency metal–ligand regions.

1. IR Spectrum of Plain Filter Paper

Figure 3. IR/FTIR spectrum of plain filter paper. O–H, C–H, and C–O–C/C–O vibration regions characteristic of the cellulose matrix are observed in the spectrum



In the spectrum of plain filter paper, the broad and intense band around 3437.15 cm^{-1} is associated with the stretching vibrations of the hydroxyl (–OH) groups of cellulose and with strong hydrogen bonding (Maréchal, Y., Chanzy, H., 2000). The band around 2902.87 cm^{-1} corresponds to the stretching vibrations of aliphatic C–H bonds in the cellulose chain (Oh, S. Y., Yoo, D. I., Shin, Y., Seo, G., 2005). The signal around 1643.35 cm^{-1} is attributed to bound water molecules or to O–H deformation vibrations on the cellulose surface.

The absorptions in the 1448.54, 1431.18, 1375.25, 1330.88, 1319.31, 1282.66, and 1238.30 cm^{-1} regions may be related to the C–H deformation, C–O, and C–O–H vibrations of the cellulose skeleton. The strong signals around 1178.51, 1130.29, 1089.78, and 1043.49 cm^{-1} indicate the C–O–C glycosidic bond and the C–O stretching vibrations diagnostic of cellulose (Oh, S. Y., Yoo, D. I., Shin, Y., Seo, G., 2005). The band at 896.90 cm^{-1} is regarded as characteristic of β -glycosidic linkages. The 671.23, 628.79,

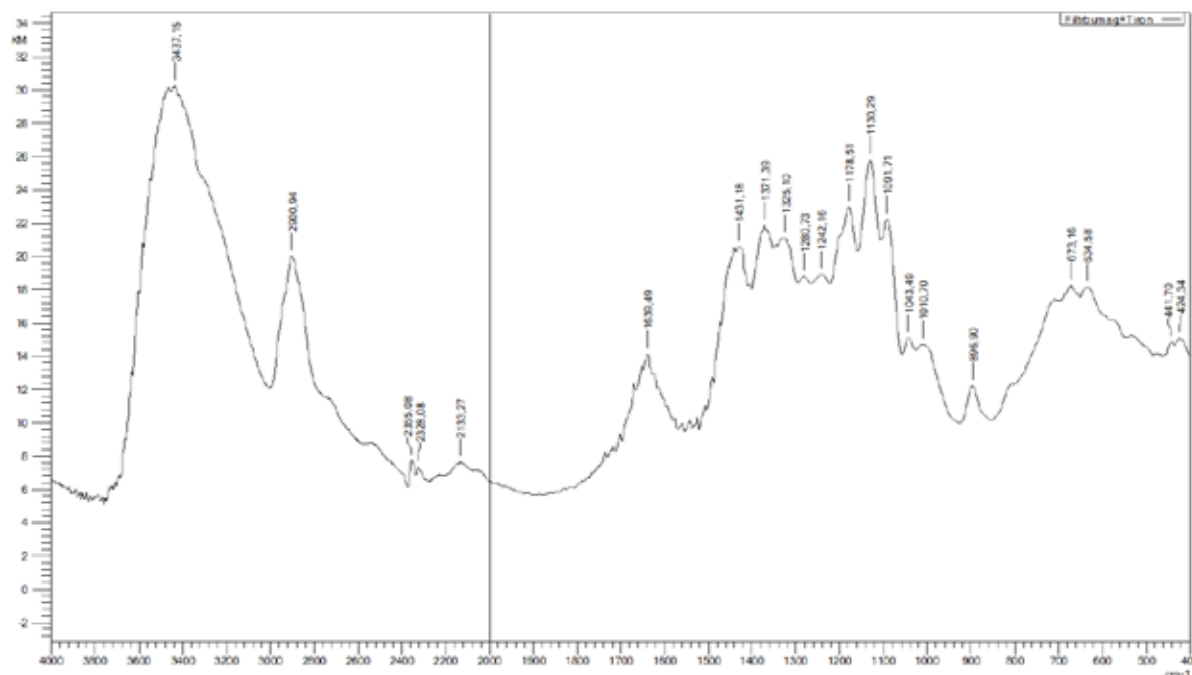
569.00, 530.42, and 451.34 cm^{-1} regions are associated with low-frequency deformation vibrations.

The spectrum of plain filter paper provides the typical IR signatures of a cellulose-based matrix and serves as a control spectrum for comparing the subsequent modifications.

2. IR Spectrum of Tiron-Immobilized Filter Paper

In the Filter paper+Tiron spectrum, the broad O–H band around 3437.15 cm^{-1} is retained, indicating the presence of hydrogen bonding between cellulose and the phenolic –OH groups of Tiron (Maréchal, Y., Chanzy, H., 2000). The C–H signal at 2900.94 cm^{-1} corresponds to the vibrations of the organic skeleton (Oh, S. Y., Yoo, D. I., Shin, Y., Seo, G., 2005). The band around 1639.49 cm^{-1} is close to the 1643.35 cm^{-1} signal of plain filter paper but is slightly shifted, which may indicate a change in the O–H bonding environment as a result of Tiron immobilization (Maréchal, Y., Chanzy, H., 2000; Oh, S. Y., Yoo, D. I., Shin, Y., Seo, G., 2005).

Figure 4. IR/FTIR spectrum of Tiron-immobilized filter paper. After the introduction of Tiron, the composition of the signals in the 1400–1000 cm^{-1} range changes.



The signals in the 1431.18, 1371.39, 1325.10, 1280.73, and 1242.16 cm^{-1} regions may be enhanced or reshaped owing to vibrations related to the aromatic ring of Tiron, the phenolic C–O vibrations, and the sulfonate groups. The bands around 1178.51, 1130.29, 1091.71, 1043.49, and 1010.70 cm^{-1} indicate that the S=O/S–O and C–O vibrations of Tiron are superimposed on the C–O–C/C–O vibrations of cellulose (Oh, S. Y., Yoo, D. I., Shin, Y., Seo, G., 2005; Pavia, D. L., Lampman, G. M., Kriz, G. S., Vyvyan, J. A., 2015). The signals at 673.16 and 634.58 cm^{-1} are interpreted as low-frequency vibrations related to the deformation of the aromatic ring or to the sulfonate groups.

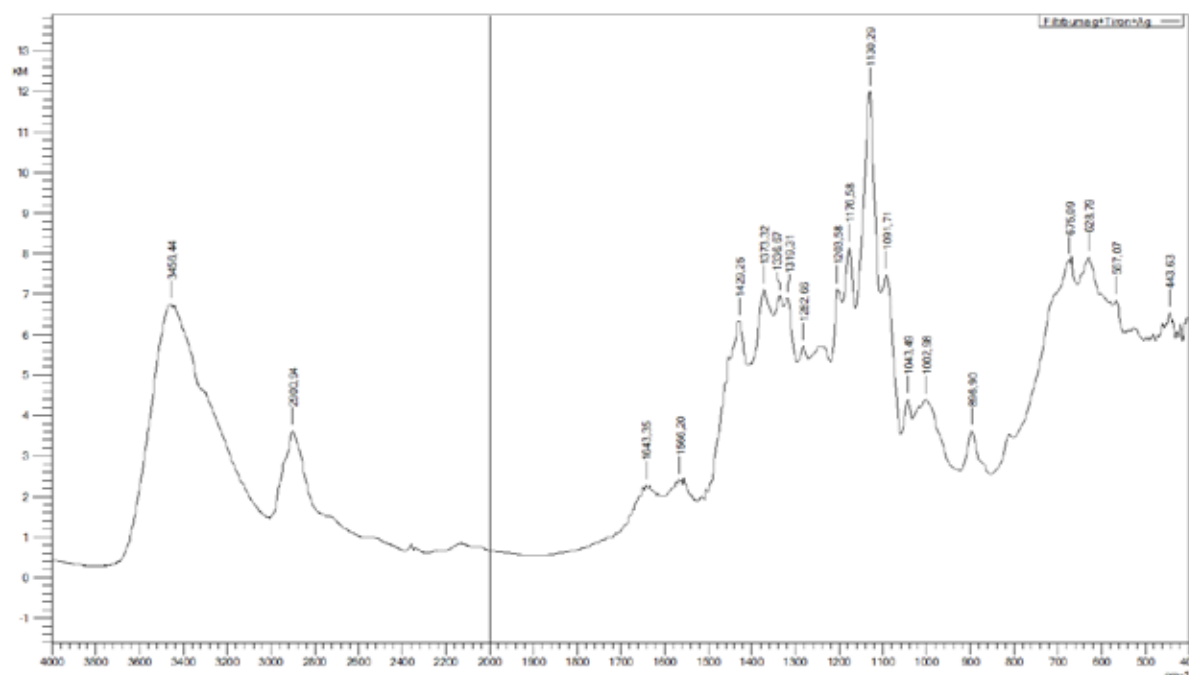
After Tiron immobilization, new or shifted absorptions appear in the 1400–1000 cm^{-1} range while the typical cellulose signals are preserved in the spectrum. This indicates that Tiron is retained on the surface of the filter paper.

3. IR Spectrum of the Filter paper+Tiron+Ag Complex

In the Filter paper+Tiron+Ag spectrum, the broad O–H band at 3456.44 cm^{-1} is shifted to a slightly higher frequency compared with the 3437.15 cm^{-1} region of the Tiron-immobilized sample. This indicates

that a change has occurred in the environment of the hydroxyl groups and in hydrogen bonding after the uptake of Ag ions (Pavia, D. L., Lampman, G. M., Kriz, G. S., Vyvyan, J. A., 2015). The signal at 2900.94 cm^{-1} indicates that the C–H vibrations of the organic skeleton are preserved. The bands around 1643.35 and 1566.20 cm^{-1} may be associated with the aromatic/catechol fragments of Tiron and with the electron environment modified as a result of the interaction with Ag.

The signals in the 1429.25, 1373.32, 1336.67, 1319.31, and 1282.66 cm^{-1} regions are slightly changed compared with the corresponding regions of the Tiron-immobilized paper. This indicates that the phenolic C–O and aromatic ring vibrations of Tiron have passed into a new coordination environment in the presence of Ag ions. The strong absorptions in the 1203.58, 1176.58, 1130.29, 1091.71, 1043.49, and 1002.98 cm^{-1} regions indicate that the sulfonate, C–O, and C–O–C groups may participate in the binding of Ag (Nakamoto, K., 2009). The changes in the low-frequency 675.09, 628.79, 567.07, and 443.63 cm^{-1} regions are likely related to near metal–ligand vibrations or to surface complexation (Nakamoto, K., 2009).

Figure 5. IR/FTIR spectrum of the Filter paper+Tiron+Ag complex. After treatment with Ag, shifts and intensity changes are observed in the regions of certain functional groups

General Analysis of the IR Spectra

A comparison of the three spectra shows that plain filter paper gives mainly the O–H, C–H, C–O–C, and C–O vibrations characteristic of cellulose. After Tiron immobilization, the composition of the signals in the 1400–1000 cm^{-1} range changes significantly, indicating that the aromatic ring, phenolic C–O, and sulfonate groups of Tiron have been introduced onto the paper surface. After the uptake of Ag, additional shifts and intensity changes are observed in the 3456, 1566, 1429–1282, and 1203–1002 cm^{-1} regions.

Thus, the IR analyses progressively confirm the immobilization of Tiron and the subsequent interaction with Ag: in the first stage, the filter paper is functionalized with Tiron; in the second stage, the Ag ions bind to the phenolic and sulfonate groups of Tiron, as well as to the oxygen-containing groups on the cellulose surface. However, the IR spectrum does not fully establish whether Ag is present as Ag(I) or Ag(0). Therefore, XRD, XPS, or SEM–EDS analyses are additionally required to unambiguously prove the oxidation state of Ag.

Table 1.

Sample	Main regions, cm^{-1}	Probable assignment	Scientific interpretation
Filter paper	3437; 2902; 1643; 1178–1043; 896	O–H, C–H, bound water/O–H deformation, C–O–C/C–O, β -glycosidic bond	Control spectrum of the cellulose matrix.
Filter paper+Tiron	3437; 1639; 1431–1242; 1178–1010; 673–634	O–H, aromatic/catechol fragment, C–O, S=O/S–O, ring deformation	Indicates that Tiron is immobilized on the paper surface.
Filter paper+Tiron+Ag	3456; 1643; 1566; 1429–1282; 1203–1002; 675–443	O–H shift, aromatic/catechol, C–O/S–O, low-frequency surface vibrations	Indicates that Ag ions interact with the Tiron/cellulose groups.

The generalized interpretation of the main IR bands is given in the following table 1:

Conclusion

In this study, the organic reagent Tiron was successfully immobilized onto the surface of filter paper, and a functional material capable of binding silver(I) ions was obtained. The diffuse reflectance spectroscopy (DRS) results showed that the optical properties of the filter paper changed after Tiron immobilization and that the spectral response changed even more significantly after treatment with Ag(I) ions. The FTIR analyses confirmed the successful immobilization of Tiron onto the cellulose matrix and the participation of the hydroxyl and sulfon-

ate functional groups in the interaction with Ag(I) ions. The obtained results demonstrate that Tiron-modified filter paper is an effective functional material for binding silver(I) ions. The material is simple to prepare, economically affordable, and environmentally safe, which allows its application in the development of paper-based optical sensors, sorption materials, and rapid metal-ion detection systems. Further studies are underway to enhance the analytical performance of the material, including a detailed assessment of its selectivity, sorption capacity, limit of detection, and applicability to real samples.

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