



Synthesis, Solvatochromic Properties and pH Sensory of Novel Symmetrical Bis(Tricyanofuran)hydrazone Chromophore



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NOVEL symmetrical bis(tricyanofuran) chromophore comprising a hydrazone group was prepared and characterized in order to study its solvatochromism and pH-sensory properties. The preparation of the bis(tricyanofuran)hydrazone chromophore was achieved via a diazo-coupling reaction of benzidine bis(diazonium) salt with tricyanofuran. The chemical structure of the prepared chromophore was confirmed via nuclear magnetic resonance spectroscopy (^1H and ^{13}C NMR), Fourier-transform infrared spectroscopy (FTIR), elemental analysis (C, H, N). The UV-visible absorption spectra displayed an interesting solvatochromism in different solvents depending on the polarity of the applied solvent. The pH molecular switching was stimulated under a reversible deprotonation-protonation structural change in acetone solution demonstrating color change from red to violet depending on the pH of the medium from acidic to alkaline, respectively.

Keywords: Bis-tricyanofuran, Hydrazone, Solvatochromism, pH sensory

Introduction

A stimuli-responsive material has been designed to possess one or more features that can be altered in a controlled manner by an external stimulus, such as heat (thermochromism), pH (halochromism), solvent polarity (solvatochromism), electricity (electrochromism), and light (photochromism) [1-7]. Substances with solvatochromic effect, in particular, have been used in a variety of applications, such as detection of explosives, toxic chemicals and solvent polarity, photo and/or electroluminescent agents for laser, optical light emitting diodes, and dye sensitized solar cells [8-13]. Solvatochromism is defined as the ability of a certain substance to change its color in response to solvent polarity as a result of variations in its absorption spectra according to solvent polarity [14-22].

Hydrazone-based chromophores are generally defined as materials with a Schiff-base functional

group comprising a secondary amine proton. Hydrazones are a significant group of compounds that have been applied in a variety of research fields, such as sensors, antimicrobial and antifungal agents, and nonlinear optics [23-31]. As a consequence, numerous researchers have been preparing hydrazone-based chromophores as major target molecular structures and study their medical and biological activity [32-41]. The extensive utilization of antimicrobial agents leads to the growth of strongly resistant pathogens. Thus, there has been a considerable concern in the development of diverse creative compilation of pharmacological and biological agents [42-46]. These remarks highlight the need for new hydrazones that gain diverse pharmacological and biological activities. Hydrazones are known by their easy production and are attractive materials in the design of new molecular switching chromophores with solvatochromism and pH-sensing properties [47, 48]. A hydrazone moiety

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can function as an electron bridge in a push-pull molecular system, or it can operate itself as an electron-donor fragment, once in conjugation with an electron-deficient group [49-51]. Herein, we report on the preparation, characterization and photophysical assessment of a novel symmetrical Bis (TriCyanoFuran) Hydrazone (BTCFH) chromophore (Fig. 1).

Experimental details

Apparatus and materials

Melting point was recorded uncorrected in (°C) on Stuart SMP30. Fourier-transform infrared (FTIR) spectrum was reported on Nexus 670 (Nicolet, United States). Elemental analysis (C, H, N) was investigated by Perkin-Elmer 2400 (Norwalk, United States). Mass spectrum was recorded at 70 eV using Shimadzu GCMS-QP 1000-EX mass spectrometer. UV-Vis absorption spectra were recorded on UNICAM UV-Vis 300. Nuclear magnetic resonance (NMR) spectrum was studied on a BRUKER AVANCE 400 spectrometer at 400 MHz at ambient conditions; chemical shifts were given in ppm. The pH values were recorded by BECKMAN COULTER PHI340 meter with a glass-calomel electrode.

Spectroscopic grade solvents were obtained commercially from Aldrich and Fluka. Both tricyanofuran moiety and the bis(tricyanofuran) hydrazone (BTCFH) chromophore was prepared according to previously reported literature procedures [14].

Synthesis of bis(tricyanofuran)hydrazone chromophore

A mixture of benzidine (184 mg, 1 mmol) and hydrochloric acid (10 mL) in 50 mL conical flask was stirred on a magnetic stirrer and cooled to 0-5°C in an ice-bath. A solution of sodium nitrite (145 mg, 2.1 mmol) in water (5 mL) was then added dropwisely. The produced benzidine bis(diazonium) chloride salt was stirred for an additional 30 minutes at 0-5°C. In a separate

conical flask, a mixture of tricyanofuran (418 mg, 2.1 mmol) and sodium acetate (2 g) was vigorously stirred at 0-5°C in acetone (10 mL). The cold aqueous solution of benzidine bis(diazonium) chloride salt was added dropwisely. After stirring for an additional two hours, the crude product was filtered off under vacuum, washed with distilled water (3x10 mL), re-crystallized from ethanol and finally air-dried to provide a red solid (287 mg; yield 47%). mp 215-217°C; ¹HNMR (400 MHz, DMSO-d₆) δ (ppm): 12.78 (s-broad, 2H, NH), 7.79 (s, 2H *aliphatic*, =C-H), 7.77 (d, 4H *aromatic*), 7.40 (d, 4H *aromatic*), 1.81 (s, 12H *aliphatic*). ¹³CNMR (400 MHz, DMSO-d₆) δ (ppm): 177.61, 173.82, 157.23, 137.07, 125.32, 118.36, 114.75, 113.21, 113.18, 112.57, 98.83, 98.06, 55.65, 26.78, 23.15. IR (neat, v/cm⁻¹): 3385 (NH), 2207 (CN), 1584 (C=N). MS *m/z* (%): 603 [M-H]⁺. Elemental analysis calculated for C₃₄H₂₄N₁₀O₂ (604.62): C 67.54, H 4.00, N 23.17; Found: C 67.68, H 3.91, N 23.09

Results and Discussion

Synthesis of bis(tricyanofuran)hydrazone chromophore

The strong electron-deficient tricyanofuran (TCF) was obtained according to a previously reported literature procedure employing Knoevenagel reaction among 3-methylacetoin and malononitrile in absolute ethyl alcohol and sodium ethoxide as a strong base [14]. Knoevenagel reaction is an attractive reaction for the formation of electron-withdrawing materials, such as the highly electron-withdrawing TCF moiety. The strong electron-withdrawing cyano substituents on the oxygen-containing TCF heterocycle were helpful for the stabilization of the negatively-charged TCF carbanion produced by proton abstraction from the active-methyl group using sodium acetate as a weak base. The prepared negatively-charged TCF carbanion was then subjected to diazo-coupling by the benzidine

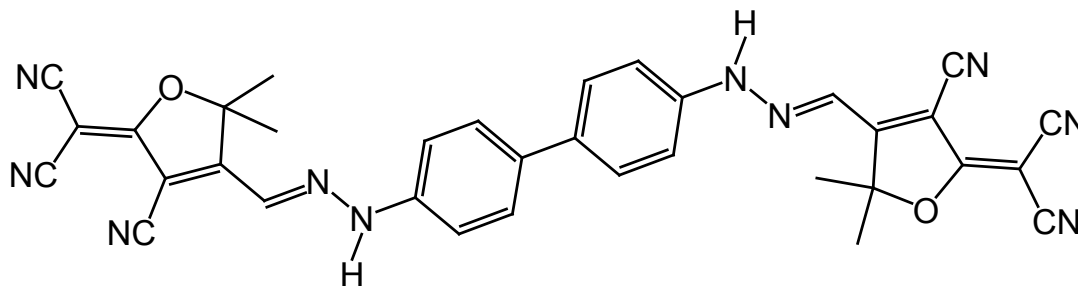


Fig. 1. Molecular structure of symmetrical Bis (TriCyanoFuran) Hydrazone (BTCFH) chromophore.

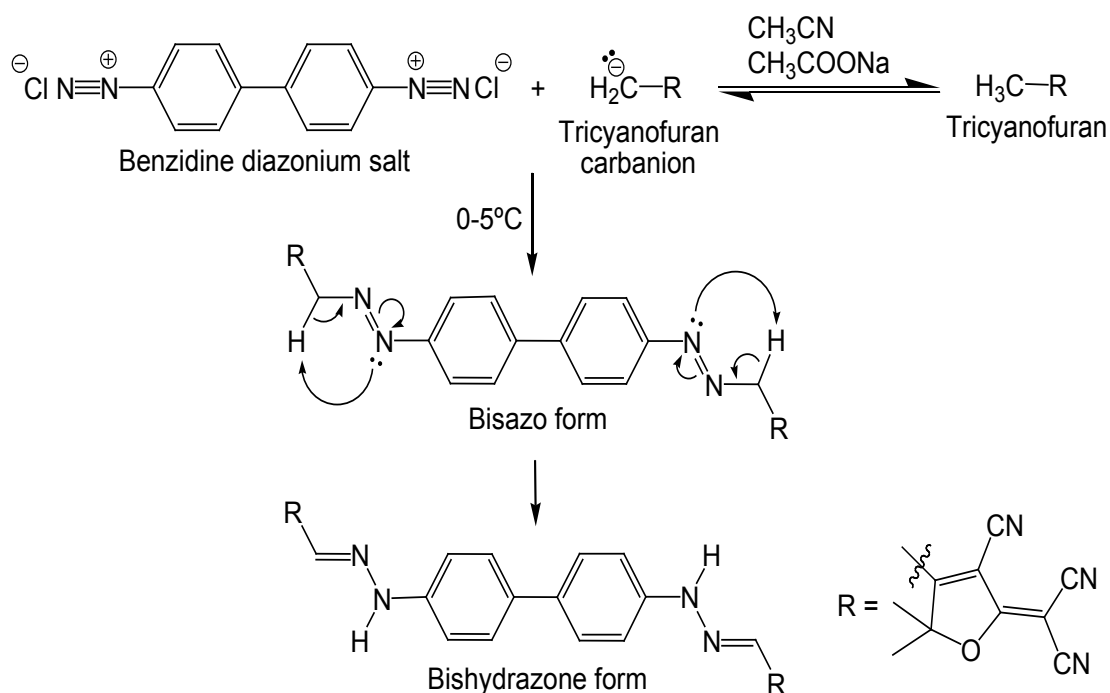
bis(diazonium) salt to introduce the consequent unstable bis(azo) dye form that was converted directly into the stable bis(hydrazone) dye form as shown in Scheme 1.

The molecular structure of the symmetrical bis(tricyanofuran)hydrazone chromophore was verified according to its spectroscopic results. FTIR spectrum showed an absorption band at 3385cm^{-1} which is attributed to hydrazone NH. The absorption band at 1584cm^{-1} is owing to the hydrazone C=N stretch, while the cyano substituent appeared at 2207cm^{-1} . The ^1H NMR spectrum displayed singlet broad peak at 12.78ppm for the hydrazone NH proton, while the singlet signal at 7.79ppm was attributed to two aliphatic vinyl protons (=C-H). The downfield shift of this singlet peak from the aliphatic vinyl protons can be attributed to the strong effect of the electron-withdrawing TCF heterocycle.

Solvatochromic measurements

The solvatochromic effect of the bis(tricyanofuran)hydrazone chromophore in some selected solvents exhibited UV-Vis absorption bands in the wavelength range between 457 and 543nm (Table 1 and Fig. 2). The chromophore demonstrated colors between red and violet in the selected pure solvents. A characteristic solvatochromism in protic and

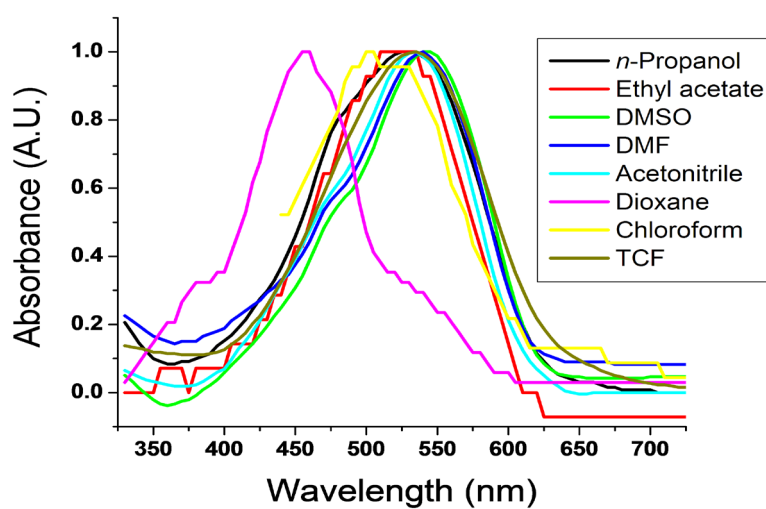
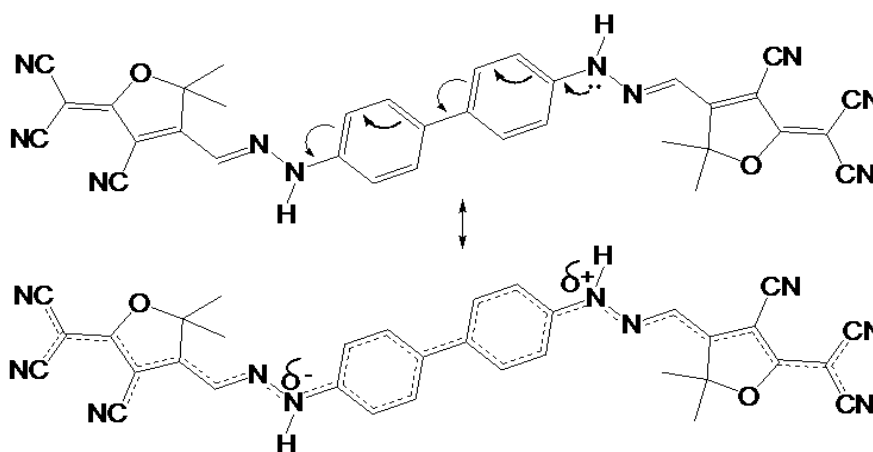
aprotic solvents was detected. As expected, protic solvents exhibited partial protonation to hydrazone NH by forming H-bond between the hydroxyl group proton of the alcoholic solvent and the lone-pair of electrons on the nitrogen atom of the hydrazone NH [27]. This resulted in decreasing the charge on the nitrogen atom of the hydrazone NH, which is translated into a partial delocalized charge transfer leading to bathochromic shift of the UV-Vis absorption band. On the other hand, in case of non-polar and aprotic solvents, this hydrogen bonding effect was negligible and our thought was change to the dipolar character of solvents for explaining solvatochromism phenomena. In general, the solvatochromic behavior demonstrated by bis(tricyanofuran)hydrazone chromophore, was due to the contribution degree of the lone-pair of electrons on the nitrogen atom of the hydrazone NH to delocalized over the whole molecule, which can partially acts as a bridge between highly electron-deficient TCF (electron pull system) and partially electron-rich hydrazone (electron push system) in a semi push-pull molecular system leading to positive solvatochromism [15]. Such semi push-pull effect can simply be explained as an extended electron inductive effect as shown in Scheme 2.



Scheme 1. Synthesis of bis(tricyanofuran-hydrazone) chromophore.

TABLE 1. UV-Vis absorption maximum wavelength (λ_{\max}) in some selected solvents.

Solvent	λ_{\max} (nm)
<i>n</i> -Propanol	530
Ethyl acetate	522
DMSO	543
DMF	540
Acetone	535
Chloroform	502
Tetrahydrofuran	535
1,4-Dioxane	457

**Fig. 2.** UV-Vis absorption maxima in some selected solvents.**Scheme 2.** Extended electron inductive property demonstrated in a semi push-pull bis(tricyanofuran) hydrazone.

Assessment of pH sensing effect

Upon increasing the pH to the alkaline range, one of the two highly electron-withdrawing tricyanofuran heterocycles on both sides of the symmetrical biphenyl bishydrazone core generated an acidic hydrazone NH proton able to produce a conjugate base, or so called a biphenylhydrazone anion (BTCFHA), with an electron-donating ability to the other TCF heterocycle. This biphenylhydrazone anion acted as an electron-donor in conjugation with the other strong electron-deficient TCF leading to an interesting spectral switch between BTCFH and BTCFHA molecular systems under pH stimulus [14]. Figure 3 displays UV-Vis absorption spectra and color changes of BTCFH dissolved in acetone (*ca.* 1.4×10^{-5} mol L⁻¹) at different pH values in the range between 5.9 and 7.5. This was accompanied by a color change from red to violet upon increasing the pH value from 5.9 to 7.5. To raise the pH value to 7.5, we gradually added a 1.0 mol L⁻¹ of methanolic tetrabutylammonium hydroxide to a solution of BTCFH in acetone, thus the maximum absorbance at 500nm was bathochromically shifted to 535nm. In order to reduce the pH value to 5.9, we gradually added a 1.0 mol L⁻¹ methanolic trifluoroacetic

acid, which led to the maximum absorbance at 500nm to reappear.

As shown in Fig. 3, the shift of the absorption maximum wavelength from 500 to 535nm upon increasing the pH value proved that there are two distinct molecular structures, BTCFH and BTCFHA, coexist in equilibrium and therefore the spectral changes were due to the acidity of the hydrazone NH proton. Proposing that the single bonds on both sides of the hydrazone NH obstructed the resonance to occur between the biphenylhydrazone core and TCF, BTCFHA generated in an alkaline environment introduced a considerable increase in the internal molecular charge which can be explained as an extended electronic resonance (Scheme 3).

To study the reversibility and stability of BTCFH, methanolic solutions of tetrabutylammonium hydroxide and trifluoroacetic acid (1.0 mol L⁻¹) were added dropwisely to an acetone solution of BTCFH to shift the pH between ~5.9 and ~7.5, while monitoring the ratio of the absorbance at 500nm (~0.130) and at 535nm (~1.78) as shown in Fig. 4. It was apparent that this course of action was reversible proving the stability of BTCFH under acid/base conditions.

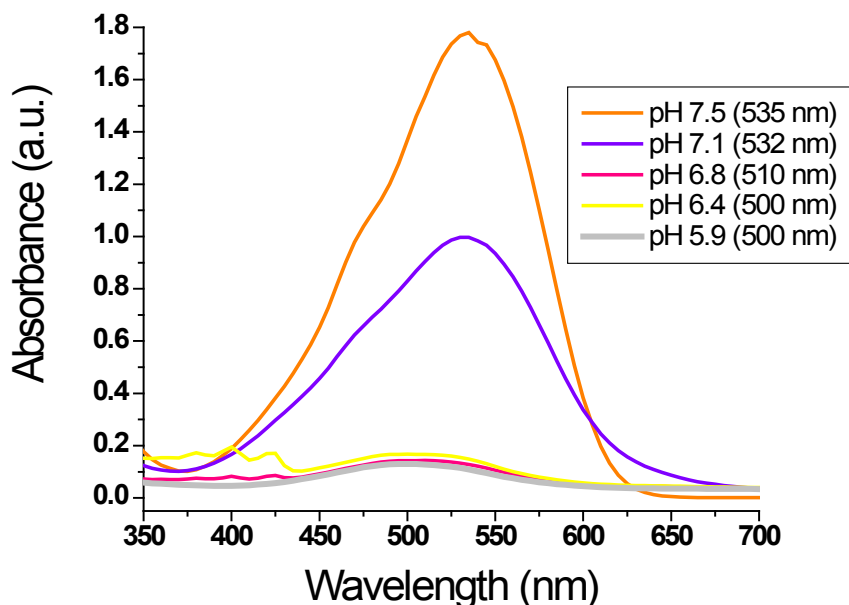
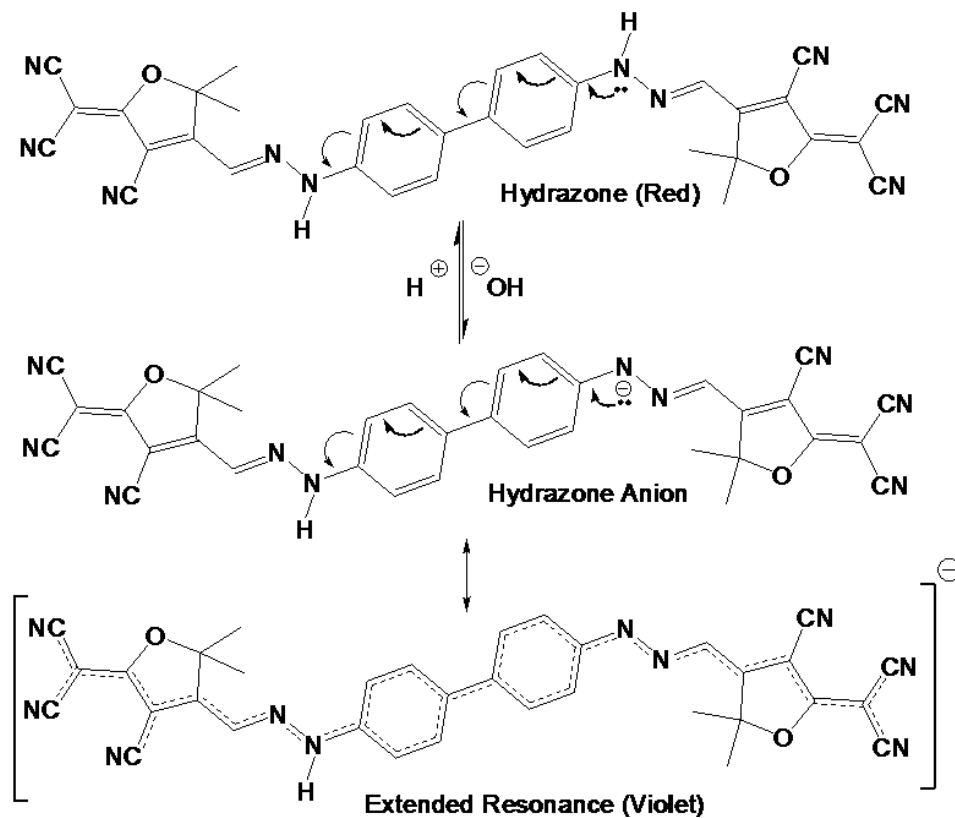
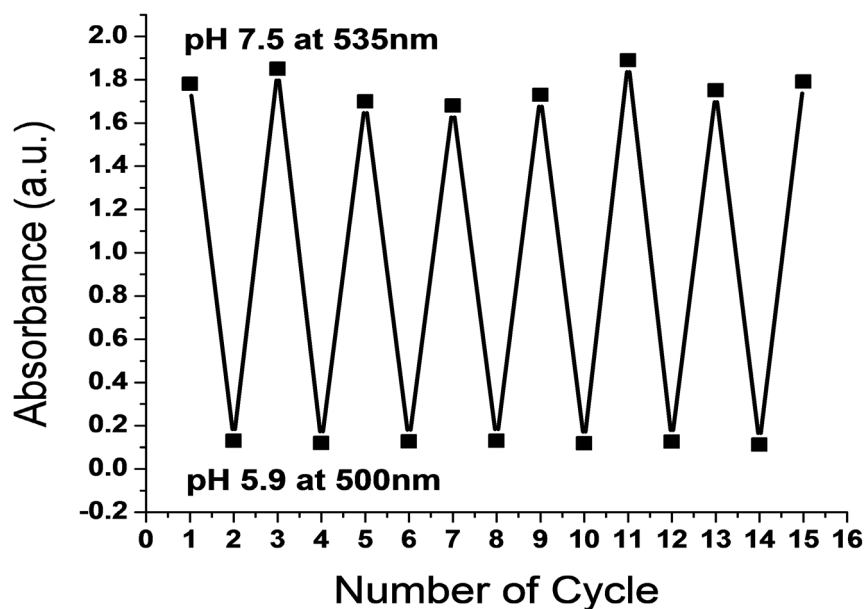


Fig. 3. UV-Vis absorption spectra of the symmetrical bis(tricyanofuran)hydrazone in acetone (*ca.* 1.4×10^{-5} mol L⁻¹) at different pH values between 5.9-7.5.



Scheme 3. pH molecular switch mechanism.

Fig. 4. Ratio of absorbance at 500 and 535nm for an acetone solution of BTCFH (ca. 1.4×10^{-5} mol L⁻¹), while altering the pH back and forth among 5.9 and ~7.5.

Conclusions

Novel bis(tricyanofuran)hydrazone chromophore was synthesized and fully characterized by ^1H and ^{13}C NMR spectrum, elemental analysis, and FTIR spectrum. It was prepared using a simple approach via a diazo-coupling of the tricyanofuran starting material with the benzidine diazonium chloride. A positive solvatochromism was recorded by UV-Vis absorption spectra in different solvents. The pH molecular switching effect, accompanied by reversible color changes from red to violet upon increasing pH value, was monitored by UV-Vis absorption spectra. Upon changing the pH value from acidic to alkaline, a charge delocalization was generated on the bishydrazone chromophore to result in an extended conjugation through a quinoid resonance form. This stimulated planarity of bis(tricyanofuran)hydrazone chromophore resulted from each strongly electron-withdrawing tricyanofuran heterocycle on the biphenylhydrazone core producing a higher conjugation extent of the hydrazone anion form than that of the hydrazone chromophore. Therefore, the pH sensory effect was displayed via modulating the intramolecular charge transfer altered by deprotonation-protonation structural changes.

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تحضير ودراسة خصائص كروموفور ثنائي التراي سيانو فيوران هيدرازون كمجس للـسولفاتوكروميك ودرجة الاس الهيدروجيني

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قسم الصباغة والطباعة والمواد الوسيطة ، شعبة بحوث الصناعات النسيجية ، المركز القومي للبحوث ، الدقي ، القاهرة
22621 ، مصر

تم تحضير مركب جديد متمائل يضم مجموعتي التراي سيانو فيوران والهيدرازون تضم من أجل دراسة وخصائصها كمجس للـسولفاتوكروميك ودرجة الاس الهيدروجيني. تم تحضير كروموفور ثنائي التراي سيانو فيوران هيدرازون عن طريق تفاعل اقتران الديازو لملح ثنائي بنزدين الديازونيوم. تم تأكيد التركيب الكيميائي للكروموفور المحضر بواسطة التحليل الطيفي للرنين المغناطيسي النووي ، التحليل الطيفي للأشعة تحت الحمراء ، التحليل الأولي للعناصر. دراسة أطياف الامتصاص المرئي للأشعة فوق البنفسجية في مذيبات مختلفة اعتماداً على قطبية المذيب المطبق. تم تحفيز التبدل الجزيئي لدرجة الاس الهيدروجيني في ظل التغيير الجزيئي لنزع واكتساب البروتون العكسي في محلول الأسيتون مما يدل على تغير اللون من الأحمر إلى البنفسجي اعتماداً على درجة الاس الهيدروجيني عند تغير الوسط من الحمضية إلى القلوية ، على التوالي.