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RESEARCH PAPER

Latent Fingerprint Detection by New Azochalcone Dye for 4-Aminoacetopheneone and Its Palladium Nano Complex

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ABSTRACT

This study presents the synthesis of an azo-chalcone derivative obtained from 4-aminoacetophenone, 6-bromo-2-naphthol, and cinnamic aldehyde, designated as BHND, together with its palladium nanocomplex. Structural characterization was carried out using a range of techniques including X-ray diffraction (XRD), elemental chemical analysis, molar conductivity, electronic spectroscopy, infrared (IR) spectroscopy, and ^1H-NMR spectroscopy. The dyeing resistance of both the ligand and its palladium complex was examined on wool and polyamide fibers, revealing promising fastness properties. The synthetic route involved the condensation of 4-aminoacetophenone with benzaldehyde to yield a chalcone, which was subsequently converted into a diazonium salt, coupled with chalcones, and then reacted with thiobarbituric acid to afford the target azo-chalcone dye. The synthesized compounds demonstrated strong stability and excellent performance on multiple surfaces, showing higher efficiency than the conventional black powder method used for latent fingerprint detection. In addition, the palladium nanocomplex exhibited desirable crystalline nanomaterial features, highlighting its potential applications in dyeing, forensic science, and nanomaterials research

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INTRODUCTION

Azo dye is an important class of compounds which possess two organic fragments connected by azo chromophore (-N=N-) [1]. They use typical pH indicators such as methyl orange and methyl red. The studies refer to efficiency prepared, good yields, low cost and biologically active of azo dyes and their coordination compounds [2, 3]. The development of fingerprint dyes requires the study and implementation of new types of azo dyes with improved properties and superior results in terms of yield, resistance to light, and humidity. Chalcones show potential such as anti-inflammatory, anti-cancer, antimalarial and antibacterial properties [4, 5]. Chalcones showed a sweat-responsive when exposed to human sweat that lead for determination the fingerprint liveness [6]. Cardiovascular diseases cause the death for many people across the globe. Many chalcones are deserving of recognition for their potential to inhibit various cardiovascular types [7]. Azo chalcones showed effective inhibition of angiotensin converting enzyme, which means effective control of blood pressure escalation

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which showed important potent inhibition with IC50 values of 0.25 μM [8]. Azo ligands, chalcone ligands and azo-chalcone ligands possess effective coordination with metal ions, stability upon chelation, and capable biological properties [9–13]. Identifying fingerprints at crime scenes is a fundamental pillar of forensic science. Due to their uniqueness and stability, fingerprints are a highly reliable means of personal identification. Their detection and identification at a crime scene establishes a direct link between the suspect and the perpetrator, providing crucial evidence. Furthermore, the analysis of latent fingerprints those invisible to the naked eye significantly enhances the accuracy of investigations. This ability not only allows for reconstructing the sequence of criminal events but also enhances the legal process by contributing to the establishment of guilt or innocence [14, 15]. The azo dyes are colored due to π-delocalization which increases the color intensity and the interaction of azo dyes with surfaces. The chalcone group with azo chromophore in the same structure increases the conjugation and gives brighter colors on surfaces [16]. Therefore, we are interested in synthesis and fingerprint detection of new azo chalcone dye of 4-aminoacetophenone and its palladium complex.

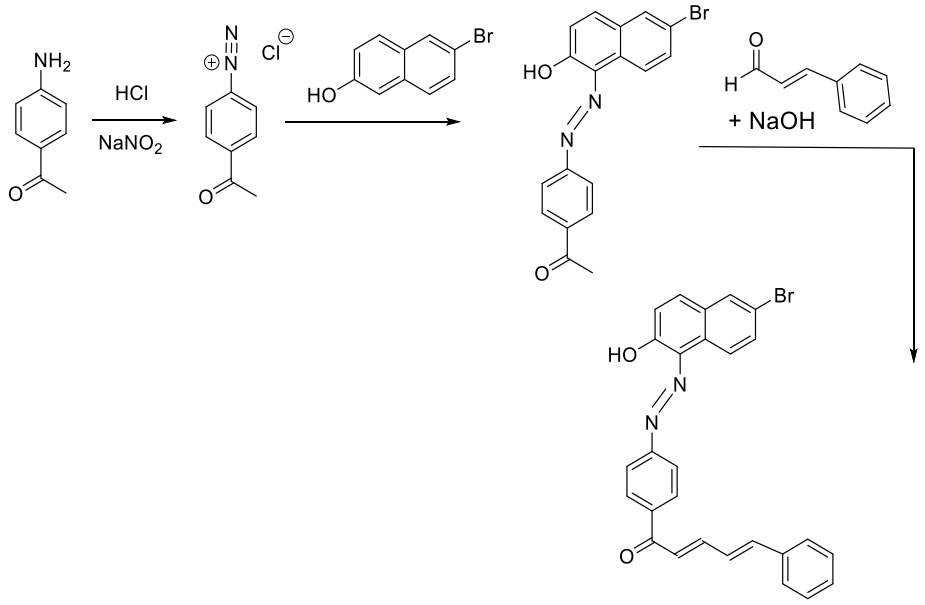
MATERIALS AND METHODS

Materials and Methods

Highly pure chemicals of analytical reagent grade (AR) were utilized. The 4- Aminoacetophenone, 6-bromo-2-naphthole and palladium (II) chloride and Cinnamic aldehyde were purchased from Merck (purity 96%). Sodium hydroxide (NaOH, 98%) was supplied from Thomas baker. Hydrochloric acid (HCl, 38%) was purchased from CGH company. Dimethyl sulfoxide (DMSO, 99%) was purchased from LOBA.

Instruments

An Elementar Vario EL III analyzer was utilized to conduct chemical analyses on carbon, hydrogen and nitrogen, elements. KBr discs were used to record FT-IR spectra on a Perkin-Elmer 1650 spectrometer within the range of 4000-400 cm^{-1}. The ^{1}H-NMR spectra were obtained using TMS as an internal standard and recorded using a 400 MHz Varian-Oxford Mercury instrument Palo Alto, as a solution in DMSO-d6. The UV-Visible spectra were recorded in the range of 1000-200 nm on Shimadzu spectrometer. We use CRIMSCOPE UV-CS-16-500 and FX10AC to determine the latent fingerprint on different surfaces. The XRD spectra for the synthesized compounds were recorded



(2*E*,4*E*)-1-(4-((*E*)-(6-bromo-2-hydroxynaphthalen-1-yl)diazenyl)phe
-5-phenylpenta-2,4-dien-1-one

Fig. 1. Preparation steps of BHND ligand.

with an UltimaIV X- ray diffractometer (Rigaku model, CuKα=0.1540562 nm). Grain sizes of the complexes were calculated with by Scherer's equation.

Preparation of BHND ligand

This ligand was prepared by the reaction of diazonium salt of (4-aminoacetophenone) with the 6-chloro-1-naphthole to form the azo dye. The azo dye was precipitated in a neutral to slightly basic medium with yield equal to 95%. Azo dye was reacted with cinnamic aldehyde in basic aqueous under stirring at 30 °C for 3 hours. The mixture was neutralized by hydrochloric acid. Then the azo-chalcone of 4-aminoacetophenone was filtrated and dried under vacuum with yield equal to 86%. IR(cm^{-1}) ligand 3334, 3058, 2974, 2880, 1674, 1597, 1507, 1432, 1393. Elemental Analysis (expected) C, 67.09; H, 3.96; N, 5.80%. Elemental Analysis (experimental) C, 66.82; H, 3.73; N, 5.78%. The preparation steps of the ligand are depicted in Fig. 1.

Synthesis of palladium (II) complex for (BHND) azo chalcone

The preparation of the palladium complex was performed by the addition of the appropriate palladium chloride $PdCl_2$ (0.5 g, 0.002 mol), in 25 mL water with two drops of hydrochloric acid for perfect solubility to the 0.2 g, 0.1 mol, of ligand which was dissolving in 40 mL of ethanol. The reaction mixture was refluxed for three hours. A dark brown palladium complex was obtained after filtering and washing with water, followed by drying. The yield was 79%, dark brown powder. IR complex 3062, 3027, 2974, 2873, 1672, 1588, 1497, 1418, 1360, 440, 418. Elemental Analysis (expected) C, 60.55; H, 3.39; N, 5.23%. Elemental

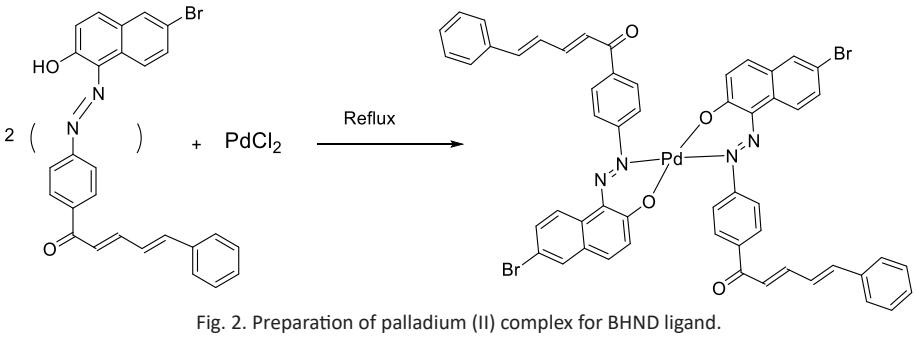


Fig. 2. Preparation of palladium (II) complex for BHND ligand.

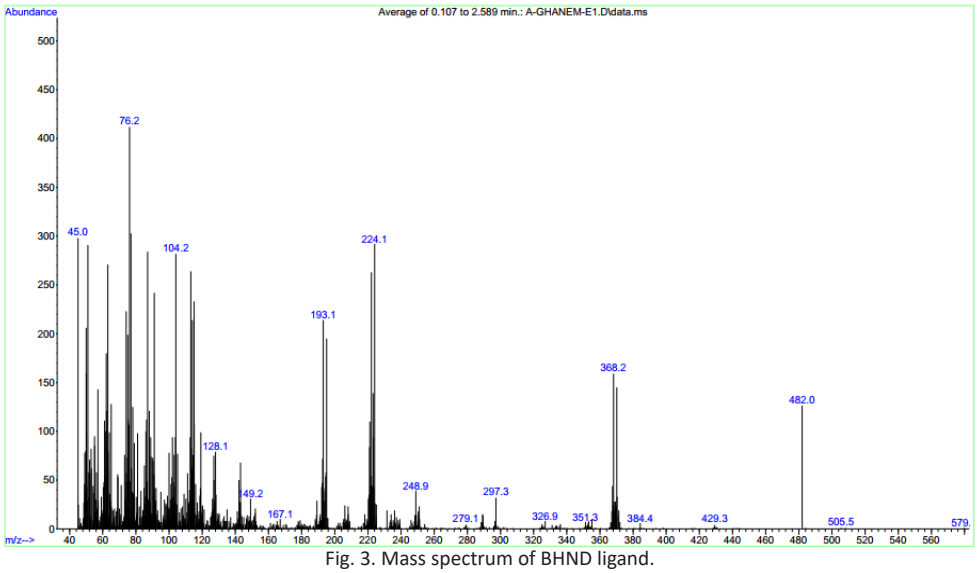


Fig. 3. Mass spectrum of BHND ligand.

Analysis (experimental) C, 60.38; H, 3.19; N, 5.18%. The preparation steps of the palladium complex are depicted in Fig. 2.

RESULTS AND DISCUSSION

The ligand and its prepared metal complexes were identified through accurate quantitative elemental analysis (C.H.N.) and by matching the theoretically calculated results with the practically obtained results, a relative convergence was observed, which proves the correctness of the added molar ratios of (metal:ligand) and confirms the correctness of the proposed formula for the prepared complex. These results are included in the table.

The mass spectrum of the ligand (BHND) as shown in Fig. 3, was recorded. The BHND under study gave a signal at 482.0, which represents the expected formula C27H19BrN2O2 of the ligand. and is consistent with the proposed formula ($C_{13}H_{11}N_3O_3$).

Infrared spectra of the ligand and its prepared palladium (II) complex

Infrared spectroscopy is an important technique for identifying the functional groups in compounds. The ligand exhibited several characterisation peaks as shown in Fig. 4. One of the important groups appearing in the spectrum of the ligand (2E,6E)-1-(4-((E)-(6-bromo-2-hydroxynaphthalen-1-yl) diazenyl)phenyl)-7-phenylhepta-2,6-dien-1-one) is the O-H group, which appeared at 3334 cm^{-1} and was not observed in the palladium complex [17–19]. Other groups such as aromatic CH at 3058 cm^{-1} and CH at 2880, 2974 cm^{-1} and peak at (1674) cm^{-1} belonging to the carbonyl group which did not suffer a change in the palladium complex, and C=C groups were at 1597, 1507 cm^{-1} as well as in-plane bending CH at (1027) cm^{-1} and out-of-plane bending CH at 828 cm^{-1}. As well as the ph-O group was at (1393) cm^{-1} which in turn suffered a change after the coordination of the ligand to the metal ions in the prepared palladium complex as shown in Fig. 5. Where it decreased by a frequency of about 30 cm^{-1} [20–23]. The ligand (BHND) also showed absorption of the N=N group at (1432) cm^{-1}, which suffered a change in position and intensity in the palladium complex spectrum, indicating its coordination with the metal ions in the palladium complex. New bands also appeared

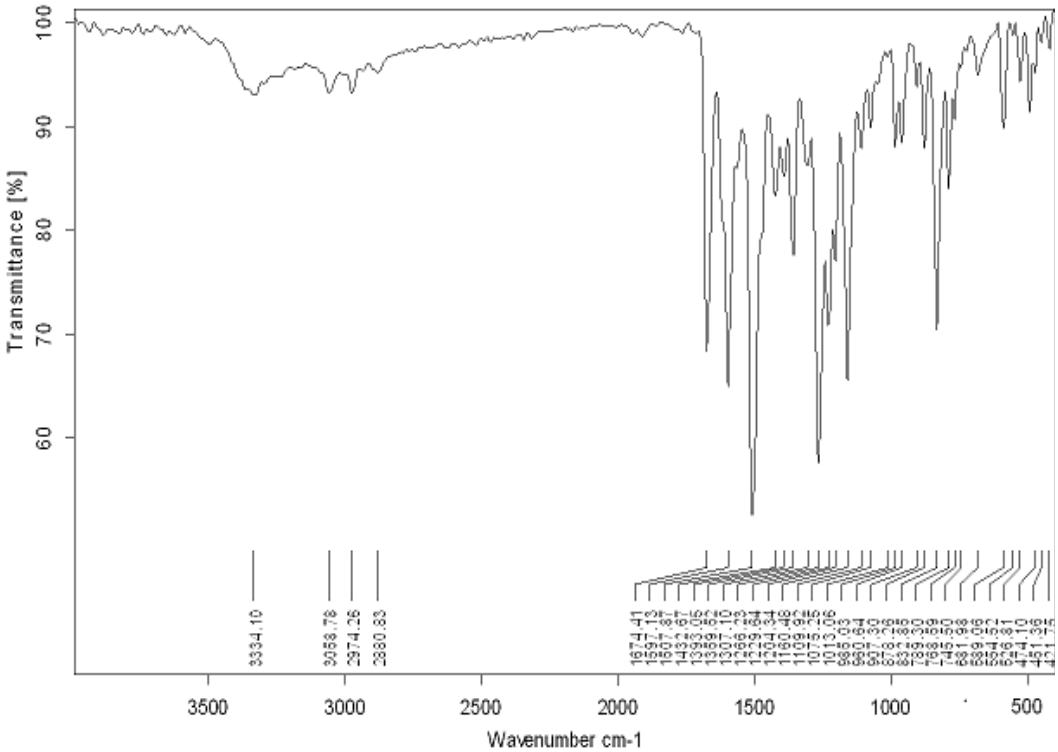


Fig. 4. Infrared spectrum of BHND dye in KBr pellet.

at low frequencies at 416-471 cm^{-1}, which are due to the absorptions of M-O and M-N [24–27].

Electronic Spectra of the BHND Ligand and Its palladium (II) complex

UV-visible spectra are an important spectroscopic technique in organic chemistry, where absorption is preferably in the visible region or near the red region, which facilitates its use in medicine and sensors. In inorganic chemistry, they are also diagnostic of the complex's structure through d-d electronic transitions. In addition to d→d electronic transitions in transition metal complexes, other transitions specific to the ligand may occur, such as π→π and n→π*, as well as charge transfer transitions from the metal to the ligand (M-L) or vice versa. The BHND ligand in dimethyl sulfoxide (DMSO) at laboratory

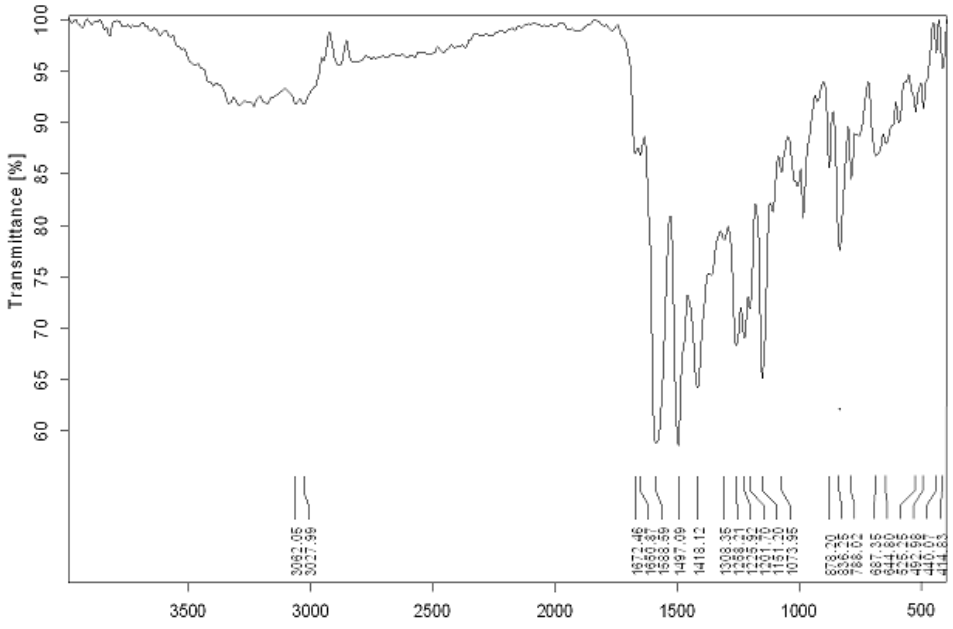


Fig. 5. Infrared spectrum of palladium complex for dye in KBr pellet.

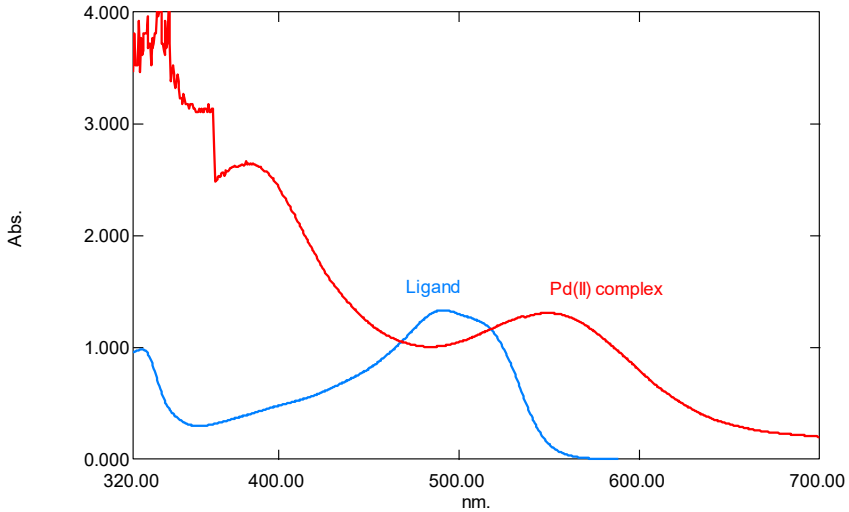


Fig. 6. UV-Visible spectra of BHND ligand and its palladium complex.

temperature exhibited two electronic transitions. The first electronic transition is at 326 nm and the second one is at 395 nm. These electronic transitions are attributed to the π→π* electronic transition [28, 29]. The third band appeared at a wavelength of 495 nm, which is attributed to the n→π* electronic transition [30–32]. The UV-Vis spectrum of the palladium complex as shown in Fig. 4, showed several transitions at 360 nm and 390 nm due to the BHND ligand transitions and an

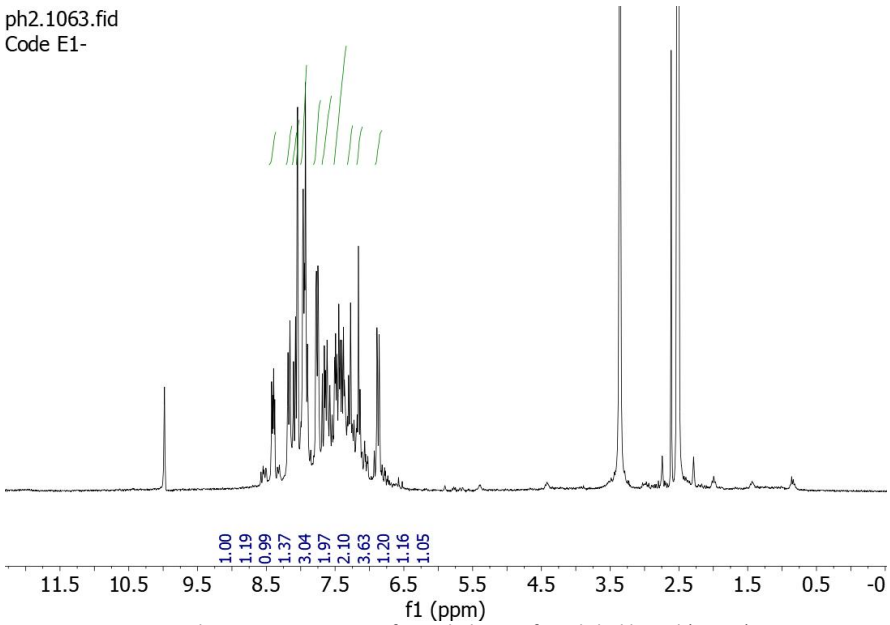


Fig. 7. The H-NMR spectrum of Azo chalcone of Naphthol ligand (BHND).

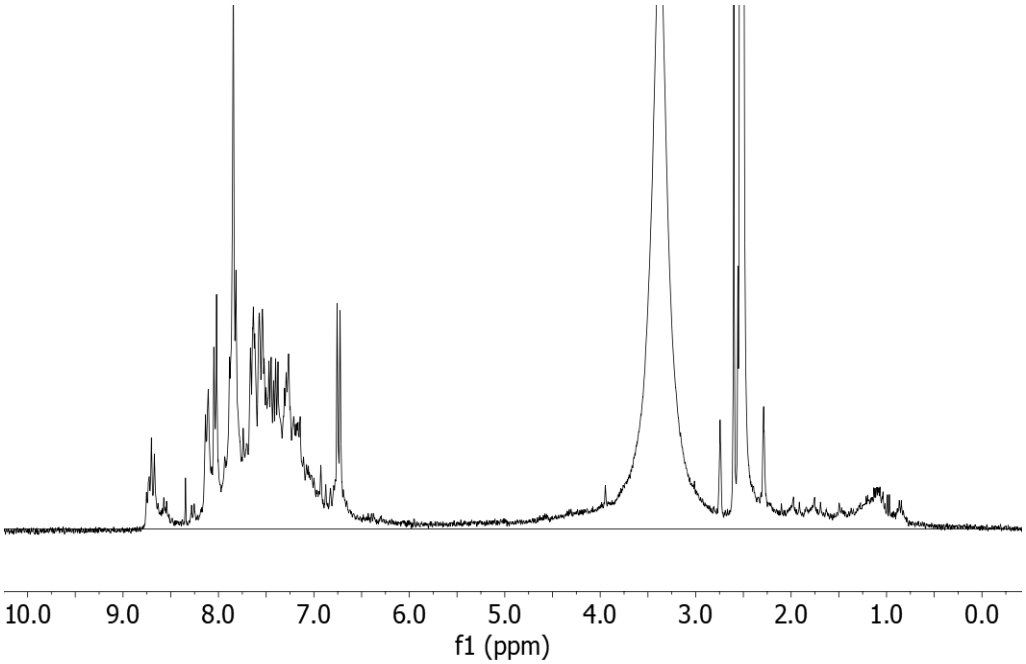


Fig. 8. The H-NMR spectrum of Palladium complex for BHND ligand.

electronic transition at 567 nm due to a $^1A_{1g} \rightarrow {}^1B_{1g}$ electronic transition, with a value of dia, which corresponds to the magnetic properties of the palladium complex. These results are consistent with the square-planar palladium complexes [33–36].

H-NMR spectra of BHND and its complex with palladium (II)

The BHND ligand was characterized by H-NMR spectroscopy (as shown in Fig. 7) using dimethyl sulfoxide (DMSO-d6) as the solvent and tetramethylsilane (TMS) as the reference standard.

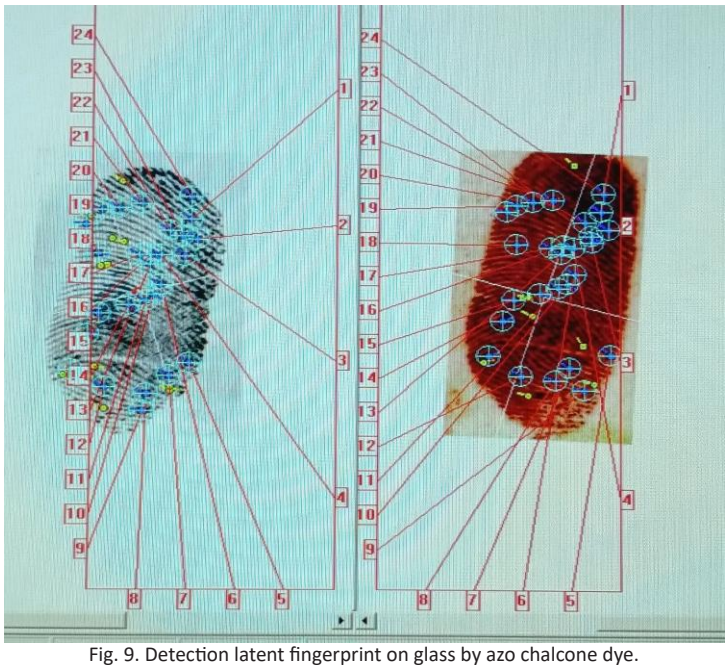


Fig. 9. Detection latent fingerprint on glass by azo chalcone dye.

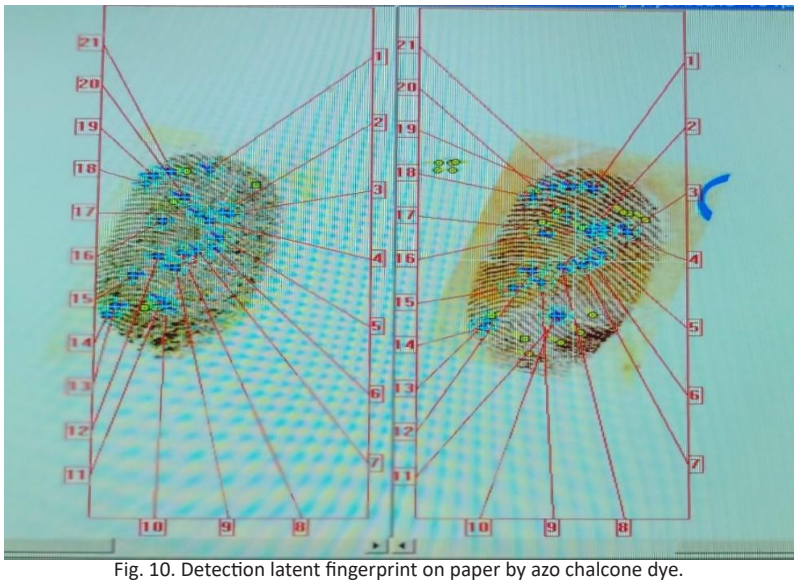


Fig. 10. Detection latent fingerprint on paper by azo chalcone dye.

The ^{1}H-NMR spectrum of the ligand showed a set of signals representing the number of protons in the ligand. In the ^{1}H-NMR spectrum of the ligand, a singlet signal at 9.97 ppm indicates to presence of a hydroxyl group (OH) in the ligand structure. Aromatic protons were present in the range of 8.39 to 6.87 ppm. A singlet signal appeared at 7.15 ppm as a singlet with multiple protons, which corresponds to a proton in the ligand. The signal at 2.5 ppm δ is due to the solvent (DMSO-d6), and a

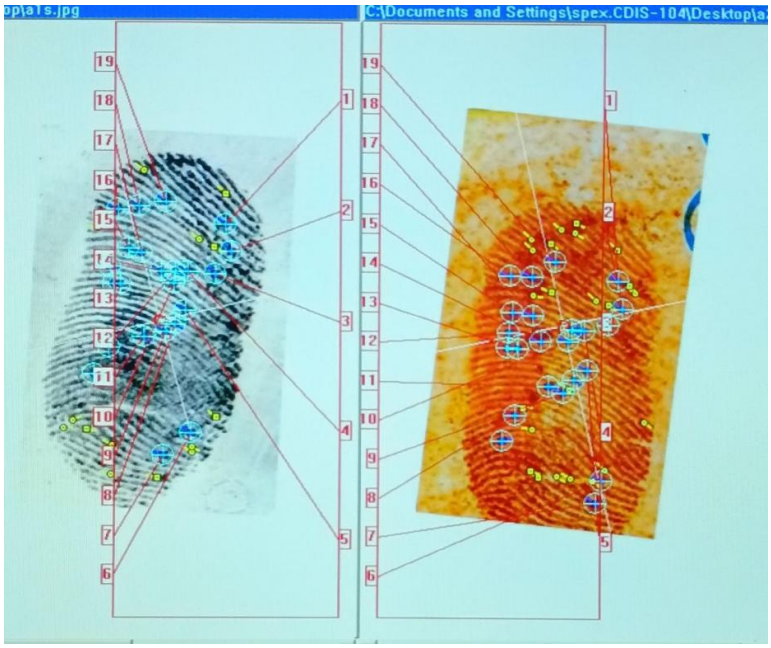


Fig. 11. Detection latent fingerprint on CD disk by azo chalcone dye.

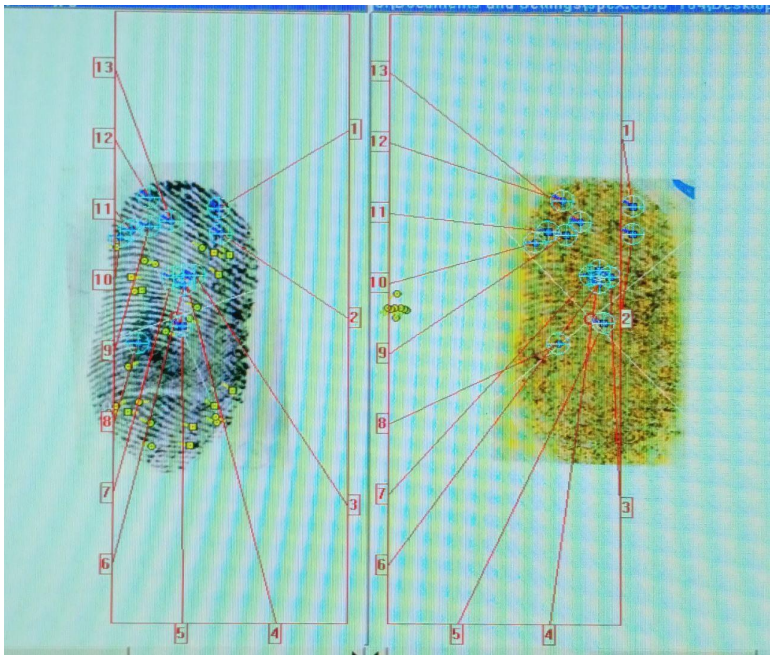


Fig. 12. Detection latent fingerprint on glass by palladium complex.

signal at 3.34 ppm is due to the residual water in the DMSO solvent.

The palladium complex exhibited H-NMR spectrum similar to the BHND ligand as shown in Fig. 8, except for the disappearance of the -OH proton. The aromatic protons appeared in the range of 6.97–8.74 ppm. The protons appeared at higher chemical displacement levels than in the

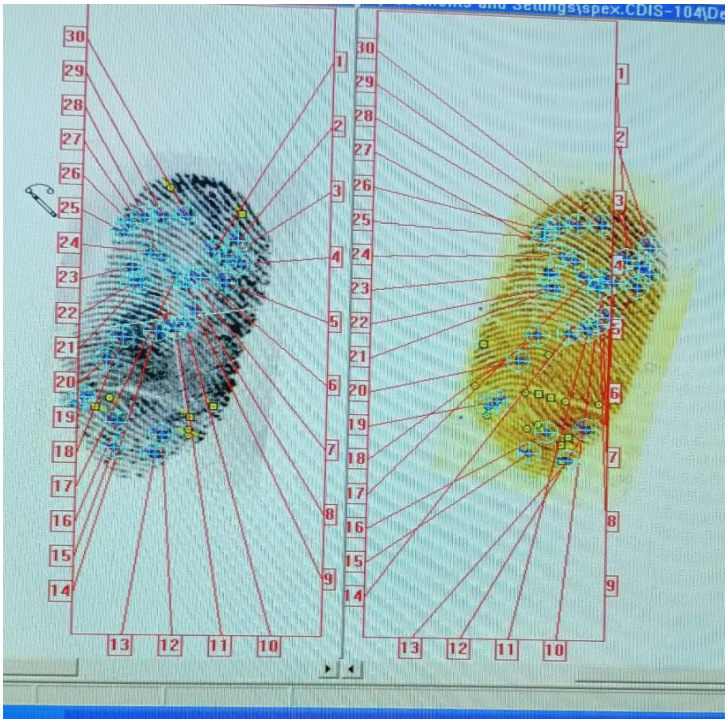


Fig. 13. Detection latent fingerprint on paper by palladium complex.

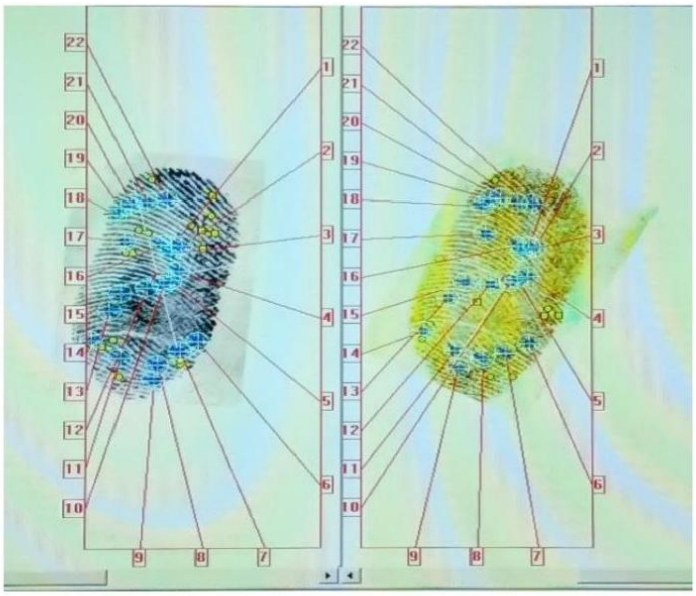


Fig. 14. Detection latent fingerprint on CD disk by Palladium complex.

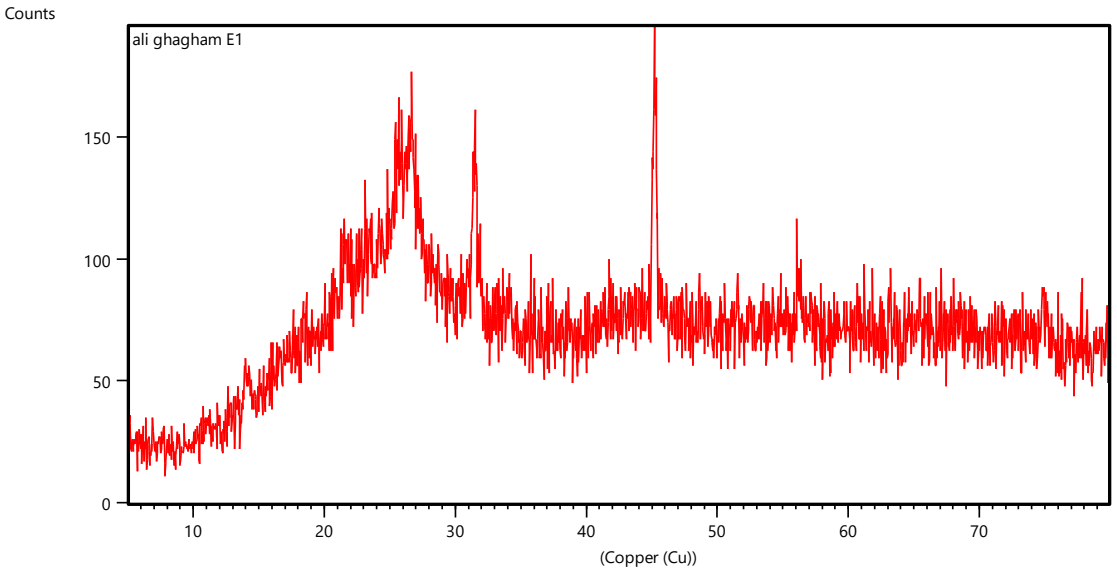


Fig. 15. XRD spectrum for azo chalcone ligand.

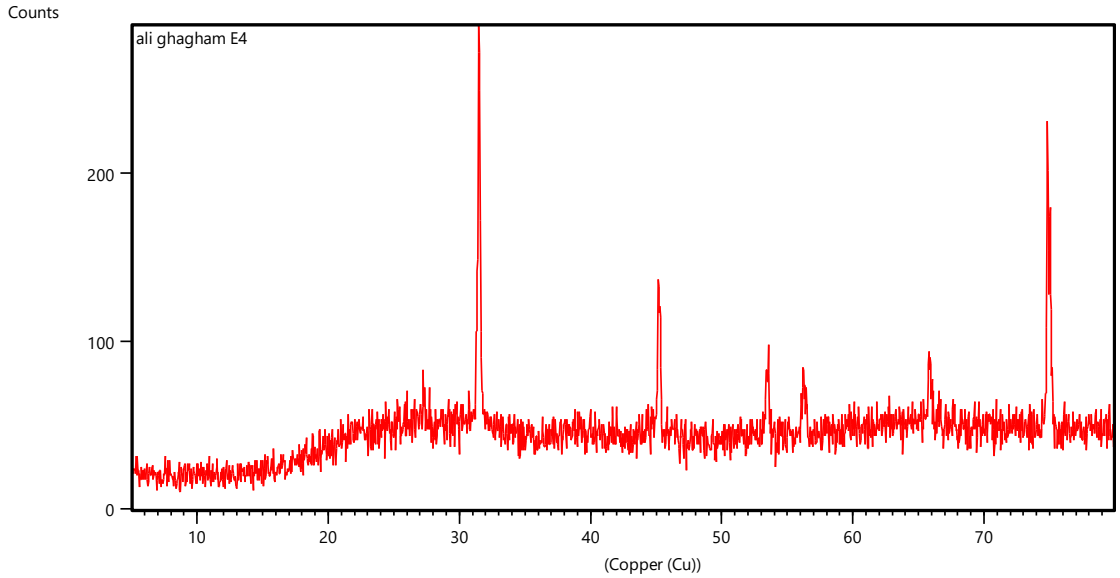


Fig. 16. XRD spectrum for palladium Nano complex of azo chalcone ligand.

BHND ligand. These indicators provide evidence of complex formation, and the number of signals in the complex indicates the symmetry of the BHND ligands around the palladium ion.

Detection of latent fingerprint

Fingerprints produced using the azo-chalcone dye and its palladium complex were compared to the standard material, black powder, using the powder method. The results demonstrated a clear match between the two fingerprints in terms of feature distribution and pattern, as well as the number of identical dots, and on different surfaces, with high stability over time as shown in the Figs. 9-14. The fingerprint of the prepared material contained a large number of dots identical to the standard fingerprint, and even included new dots not seen in the black powder case. These results

reflect the success of the material preparation process under the experimental conditions followed and confirm that the prepared material represents a very close model of the standard material. Therefore, the prepared material can be considered of great practical importance, both for its use as an alternative source of standard materials in applications, especially due to its high stability and feasibility [12, 37, 38].

XRD characterization

XRD spectrum of ligand and its nano complex are presented in Figs. 15 and 16. The ligand showed the XRD peaks at 2θ equals to 25.463°, 26.561°, 31.504° and 45.439°. The palladium complex showed the XRD peaks at 2θ equals to 31.504°, 45.370°, 53.676° and 75.025°. The ligand and its complex showed high intensity and sharp peaks which may be attributed to long range order of the molecules and high degree of the crystallite nature. The crystallite size was calculated using Scherrer equation where the ligand showed a crystallite size 45.6 nm and the complex showed crystallite size (20.4 nm) in the range of nanoparticle materials. The palladium complex is more crystalline nature and nanomaterial nature than was observed for palladium complex.

CONCLUSION

We reported herein synthesis of a novel azo-chalcone of 4-aminoacetone and its palladium complex. The synthesized compounds characterized by various spectroscopic and analytical techniques. The data suggested a square planar geometry of Pd(II) complex with 1:2 (Metal:Ligand) stoichiometry. The azo chalcone ligand is bidentate via oxygen and nitrogen atoms. They showed potential ability to detect the latent fingerprint on different surfaces with the observed coloration remaining stable over time. The crystalline nature and nanoparticle properties for palladium complex were more than the azo chalcone ligand.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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