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SULFONE BASED COMPLEXES: SYNTHESIS, CHARACTERIZATION AND MAGNETIC STUDIES OF Co (II) AND Ni(II) COMPLEXES

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ABSTRACT

Two Complexes were synthesized by using solvothermal method namely $[Co\text{-}DADPS]_n$ and $[Ni\text{-}DADPS]_n$ where DADPS is a ligand (DADPS=4,4-diaminodiphenyl sulfone). The properties of the samples were investigated by powder X-ray diffraction (PXRD), Fourier transform infrared (FT-IR) spectroscopy, UV-Visible spectrophotometer and magnetic measurement by the vibrating sample magnetometer (VSM) technique at room temperature. The average crystallite size of [Co-DADPS] and $[Ni\text{-}DADPS]_n$ samples were 66.2 nm and 36.4 nm respectively. The VSM results showed magnetic saturation 0.99amu/g and 46.68amu/g were $[Co\text{-}DADPS]_n$ and $[Ni\text{-}DADPS]_n$ respectively. The two coordination polymer complexes [Co-DADPS] and [Ni-DADPS] were synthesized via a solvothermal method using 4,4′-diaminodiphenyl sulfone (DADPS) as the organic ligand. The structural and physicochemical properties of the synthesized materials were characterized using powder X-ray diffraction (PXRD), Fourier-transform infrared (FT-IR) spectroscopy, UV–Visible spectrophotometry, and magnetic measurements obtained with a vibrating sample magnetometer (VSM) at room temperature. The average crystallite sizes of the [Co-DADPS] and [Ni-DADPS] samples, calculated from PXRD data, were found to be 66.2 nm and 36.4 nm, respectively. VSM analysis revealed saturation magnetization values of 0.99 amu/g for [Co-DADPS] and 46.68 amu/g for [Ni-DADPS], indicating distinct magnetic behaviors among the synthesized complexes.

KEYWORD: Solvothermal Method, PXRD, UV-Visible Spectrophotometer, FT-IR, VSM

Coordination Polymers, specifically metal-organic frameworks (MOFs), are the remarkable materials of the 21st century, offering unprecedented characteristics (Allahabad *et al.*, 2017). They are often formed by metallic centers coordinated with bidentate organic ligands, resulting in porous materials (Lee *et al.*, 2013). They have exhibited various potential applications in drug delivery research and catalytic systems owing to their inherent large surface areas. The organic linkers in MOF repeating coordination entities can be expanded in one, two, or two dimensions, as indicated by earlier research on MOFs (Mohammadi *et al.*, 2023). MOFs were synthesized in this investigation using the ligand [L=4,4-diaminodiphenyl sulfone] (DADPS), an antibiotic with numerous therapeutic uses, including the treatment of skin conditions. Due to their diverse oxidation states and extensive applications in materials science, health, and catalysis, cobalt-based compounds are especially intriguing. Coordination chemistry depends on transition metal complexes. Among the ligands used to form stable metal complexes, 4,4'-diaminodiphenyl sulfone (DADPS) is unique due to its sulfone ($-SO_2-$) moiety and electron-donating amine ($-NH_2$) groups, which aid in establishing strong coordination with metal centers (Liu *et al.*, 2016). Cobalt and DADPS can collaborate to create stable coordination compounds with potential applications in antibacterial activity, redox catalysis, and materials engineering. Cobalt-DADPS metal complexes have attracted significant interest due to their potential applications and structural characterization. Among these, complexes featuring cobalt and nickel and are distinguished by their distinctive electronic structures, redox characteristics, and prospective applications in magnetic materials. 4,4-Diaminodiphenyl sulfone (DADPS), characterized by its sulfone ($-SO_2$) moiety and electron-donating amine ($-NH_2$) groups, exhibits remarkable coordination properties. These complexes are utilized in bioinorganic chemistry, among other fields of study. Nickel-based compounds are essential for the production of magnetic materials. The literature references nickel d8 electronic configuration, spin crossover phenomena, coordination compounds displaying ferromagnetic or antiferromagnetic interactions, and single-molecule magnet (SMM) behavior (Somnath *et al.*, 2022).

While cobalt based compounds in most studies show antiferromagnetic interactions. Nevertheless, for molecular magnetic materials, spin electrical devices, and data storage, nickel complexes are preferred choices. It has been thoroughly investigated how to enhance the stability and performance of transition magnetic complexes by incorporating sulphur-containing

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substances, polydentate amines, and Schiff bases. Here, however, a ligand called 4,4'-diaminodiphenyl sulfone is employed to synthesize a coordination polymer (Pandey and Narvi, 2017). This work investigates the synthesis, structural description, and magnetic examinations of compounds based on cobalt and nickel. By studying their coordination behavior, electronic transitions and magnetic properties, we aim to contribute to the expanding field of metal-organic framework coordination chemistry and its applications in advanced materials. In this study we have synthesized two new metal organic framework compounds using different metal ions like nickel and cobalt. The ligand 4,4-Diaminodiphyenl sulfone is used for synthesized Cobalt (Co-DADPS) Nickel-based (Ni-DADPS) metal-organic framework compounds. Cobalt-based compounds predominantly exhibit antiferromagnetic interactions. Nonetheless, nickel complexes have become preferred options for molecular magnetic materials, spinning devices, and data storage (Yadav *et al.*, 2017). The enhancement of stability and performance in transition magnetic complexes through the incorporation of sulfur-containing compounds, polydentate amines, and Schiff bases has been extensively studied. A ligand known as 4,4-diaminodiphenyl sulfone is utilized to synthesis a coordination polymer. Five this study explores the synthesis, structural characterization, and magnetic analysis of compounds containing cobalt, nickel, and chromium. Through the examination of their coordination behavior, electronic transitions, and magnetic properties, we seek to enhance the burgeoning domain of metal-organic framework coordination chemistry and its applications in advanced materials. This study synthesised two novel metal-organic framework compounds utilizing various metal ions, including nickel and cobalt. The ligand 4,4-Diaminodiphenyl sulfone is utilized for the synthesis of cobalt (Co-DADPS) and nickel-based (Ni-DADPS) metal-organic framework compounds (Mohammadi *et al.*, 2023; Nik *et al.*, 2020).

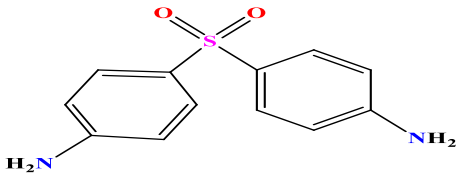


Figure 1: Structure of 4,4'-diaminodiphenyl sulfone

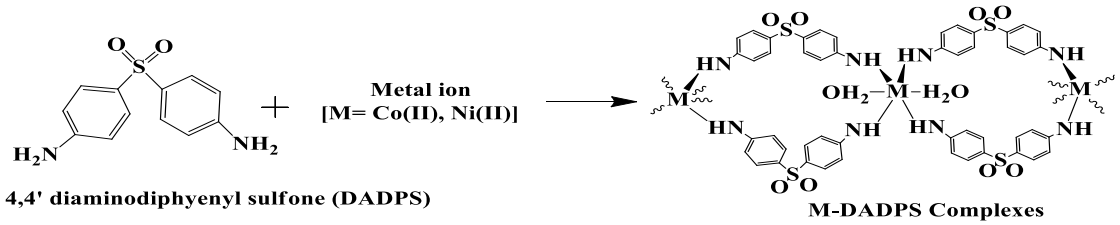
Experimental Section

Materials and Instrumentations

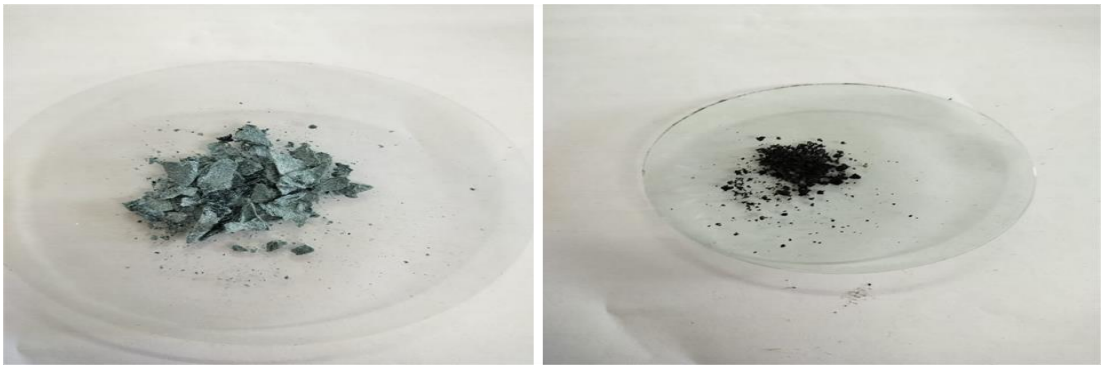
$Ni(OCOCH3)_2 4H2O$, ethanol, distilled water, 4,4'-diaminodiphenylsulfone (DADPS), and $C4H6COO_4 4H2O$ are the components of this mixture. It was not necessary to perform any additional purification on any of the reagents or solvents because they were all obtained from various commercial sources first. For the purpose of recording all infrared (IR) spectra in the range of 400–4000 cm−1, crushed KBr pellets and a Jasco FTIR-4X FTIR instrument were effectively utilized. In order to acquire the powder X-ray diffraction patterns (PXRD), a Philips X-ray diffractometer with the model number PW1840 utilized was utilized (Pandey,).

Syntheses of M-DADPS Complexes [where M = Co(II) and Ni(II)]

Complex compounds that are synthesised using 4,4-diaminodiphenyl sulfone (DADPS) and metal salts as the starting material. In a separate container, metal salt and ligand were dissolved, and then they were combined with each other drop by drop while being stirred continuously. After dissolving 1 mmole of metal salt, which consisted of cobalt acetate (0.249g) and nickel acetate (0.176g), in 10 ml of water was also added. A total of 0.496 grams of 4,4-diaminodiphenyl sulfone ligand were dissolved in 10 milliliters of methanol. A ligand known as 4,4-At the same time that the metal solution was being stirred continuously 4,4-diaminodiphenyl sulfone was being added drop by drop. At room temperature, the solution that was produced was allowed to remain on string for a period of two hours. Following filtration, the sample was washed two times with ethanol and dried in a vacuum oven. At this point, solid samples were collected and characterized using a variety of different techniques. The framework for the reaction is presented down below.



Reaction Scheme: Synthesis of M-DADPS complexes [M = Co(II), and Ni(II)]



Ni-DADPS **Co-DADPS**

RESULTS AND DISCUSSION

Fourier Transform Infrared (FT-IR) Analysis

The functional groups and coordination behavior of the synthesized materials were examined by recording their FT-IR spectra in the 4000–400 cm^{-1} range. The N–H stretching vibrations are responsible for the wide band seen in the free ligands (DADPS) spectra between 3360 and 3260 cm^{-1}. At 1636 cm^{-1} and 1581 cm^{-1}, respectively, the distinctive asymmetric and symmetric N–H stretching vibrations are seen. Additionally, a prominent band in the 1135–1058 cm^{-1} region is attributed to the sulfonyl ($–SO_2$) groups stretching vibration (Buzykin *et al.*, 2005). The coordination between the metal ion and the ligand is confirmed by the FT-IR spectra of the Ni-MOF, which exhibits discernible shifts in peak locations. At 3483, 3433, 3330, and 3234 cm^{-1}, the N–H stretching bands show potential hydrogen bonding interactions (Ji *et al.*, 2025). The stretching vibrations of the $–SO_2$ group are represented by the bands at 1273 cm^{-1} and 1135 cm^{-1}, whereas the N–H bending vibrations are responsible for the band at 1562 cm^{-1}. Additionally, the predicted area exhibits the aromatic C=C stretching vibrations. Significantly, a new band that emerges in the cobalt-based material at around 520 cm^{-1} is attributed to the Co–N stretching vibration, offering unambiguous proof of coordination between the ligand and the metal center (Rebrov, 2026). The successful construction of the metal–organic framework is strongly supported by the observed shifts in typical peaks and the emergence of new bands in the lower frequency area. The successful coordination and development of the metal–organic framework are unquestionably confirmed by these spectral signatures, which include peak shifts and the appearance of metal–ligand vibrations (Pandey *et al.*, 2014). (Figure 2)

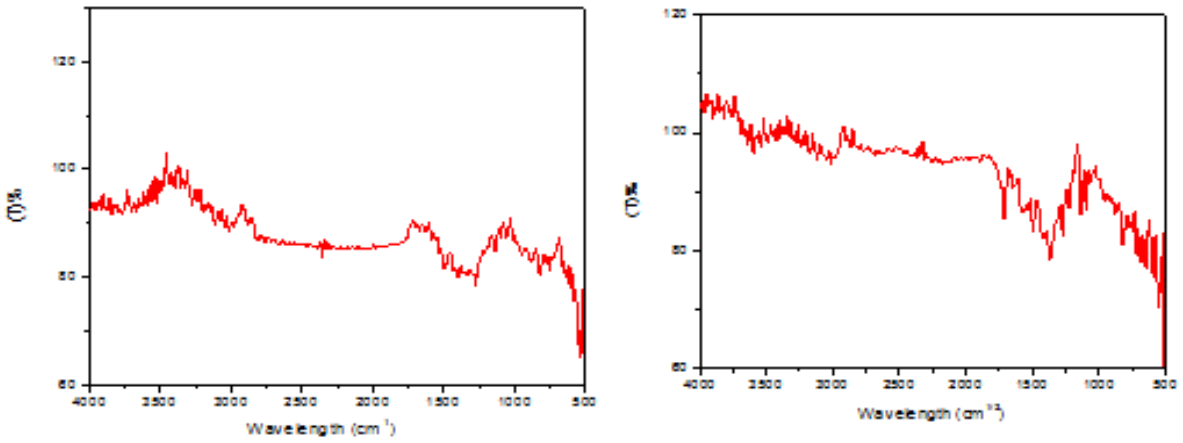
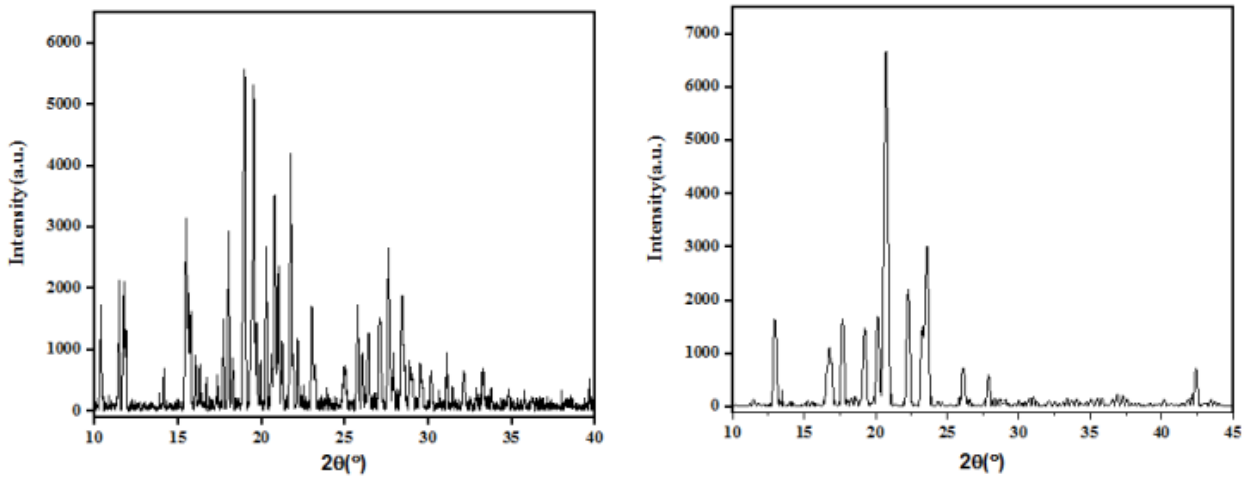


Figure 2: FT-IR spectra (a) Co-DADPS and (b) Ni-DADPS

Powder X-ray Diffraction (PXRD)

The generated materials were characterized by powder X-ray diffraction. These facilities are classified based on the ways in which X-rays interact with matter, specifically through absorption, refraction/reflection and scattering, because they technically employ optical processes. Although XRD is mostly used to determine a crystalline material's fingerprint (Bhatnagar *et al.*, 2024), Each complex has a unique structure since they are engineering materials with a high degree of crystalline. XRD analysis should be performed as the first characterization to determine the synthesized materials and their purities prior to doing further study. The two objectives of XRD analysis are to determine a unique crystal structure and validate known crystal structures. The XRD pattern of the generated Co-DADPS

compounds is shown in Fig 3. The crystallite sizes have been determined using Debye-Scherrer. D is equal to $0.94\lambda/(\beta\cos(\theta)$. where θ is the full width at half maximum (FWHM), D is the crystalline size in nanometers, 0.94 is the sheerer constant, θ is the position of the specific diffraction peak, and λ is the wavelength of the X-ray that was employed. The average crystallite size of Co-DADPS is 66.2 nm and Ni-DADPS is 36.4 nm.

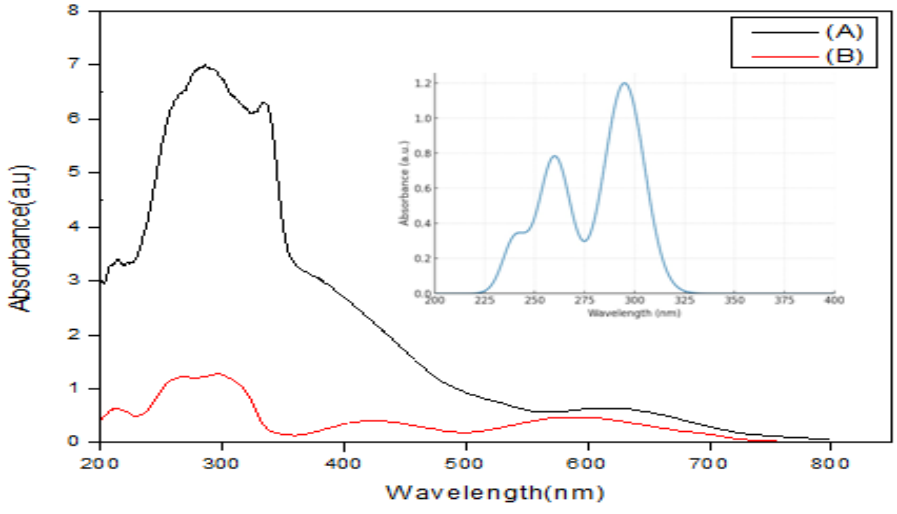


(a) **(b)**

Figure 3: PXRD Spectra (a)Co-DADPS and (b) Ni-DADPS

UV-Visible Spectroscopy

The electronic spectra of the ligand DADPS and its complexes with Co(II) and Ni(II) are displayed in Fig 4. These spectra were measured in DMSO (in the region of 251 nm) (Pai *et al.*, 2023). Located at 251 and 303 nm, the ligand exhibits two peaks that are particularly noticeable to the naked eye. This absorption band is caused by the π-π* transition that occurs in the benzene ring of the ligand, which has a wavelength of 251 nm. During the n-π* transition of the ligand, the maximum absorption (λmax) of Co-DADPS occurred at 312 nm, while the maximum absorption of Ni-DADPS occurred at 264 nm. This shift occurred at a higher wave length (Parveen *et al.*, 2025; Somnath *et al.*, 2022). The absorption band can be seen between 251 and 303 nanometers in wavelength. Additionally, the ligands π-π* transition was also a source of suggestion. During the complex, the n-π* transition of the sulfoxone group shifted to a longer wavelength frequency. It is necessary to check that the ligand and the metal ions are in coordination. An approximation known as the Tauc plot was utilized in order to determine the band gaps of each and every compound that was synthesized plot of $(\alpha h\upsilon)1/2$ versus hυ can be designed by employing this equation to create a plot. The band gaps of Co-DADPS and Ni-DADPS are 3.0 and 3.6, respectively, when represented in equivalent terms.



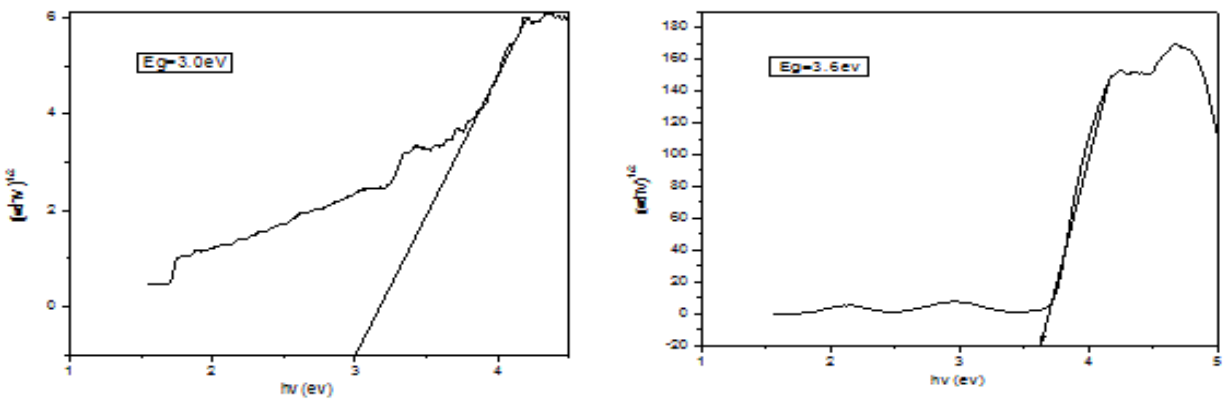


Figure 4: UV-Visible spectra of Co-DADPS (red line) and band gap (a); Ni-DADPS (black line) and band gap (b), Inset graph shows spectra of 4,4-Diaminodiphenyl sulfone(DADPS)

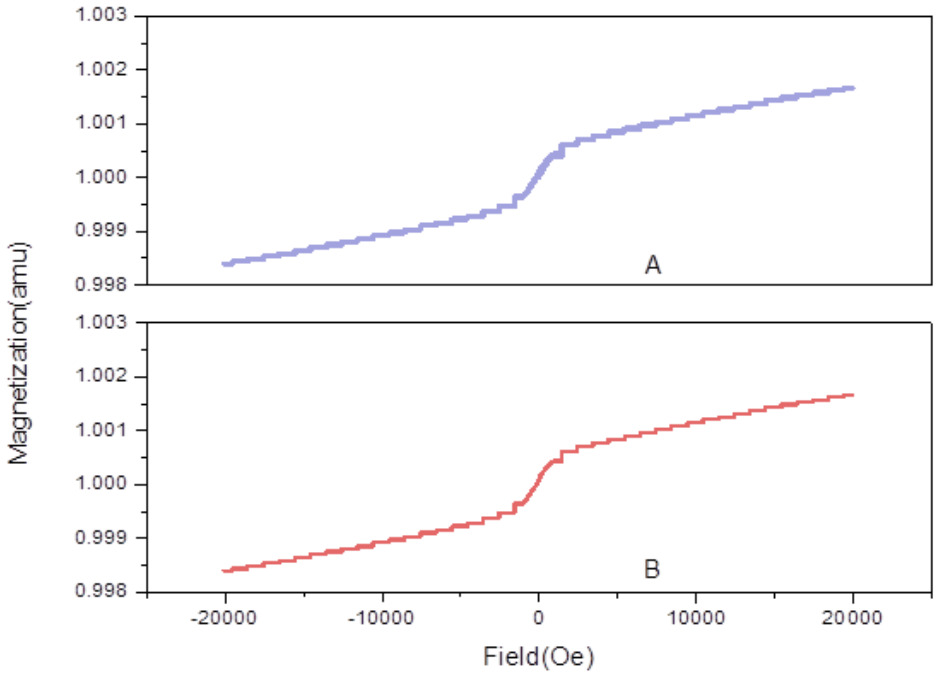


Figure 5: VSM plot of (a) Co-DADPS (b) Ni-DADPS

Magnetism

On the basis of the investigation into the magnetic properties of Ni-complexes and Co-complexes, the hysteresis loop that was obtained through VSM was utilized in order to assess the magnetic properties of Ni-complexes and Co-complexes (Abdollahi *et al.*, 2022). Fig 5 demonstrates that the remanence and coerecivity values of all materials were found to be 0. Given that this demonstrates that all materials possess super-magnetic properties, it follows that magnetic separation can be accomplished through the utilization of an external magnetic field (Vishwakarma *et al.*, 203). The magnetic moment of Co-DADPS and Ni-DADPS are 4.9µB and 3.1µB respectively. The small hysteresis loop that was obtained at room temperature on a 4-10k Oe field was used for the purpose of studying the magnetic properties of complexes (Yananose *et al.*, 2023). The value of saturation magnetization (Ms) was determined to be 0.99 emu/g for Co-DADPS and 46.68 emu/g for Ni-DADPS, respectively. The magnetic saturation value of Co-DADPS is reported to be low (1.6 emu/g), while the value for Ni-DADPS is reported to be high (55.1 emu/g) (Archana *et al.*, 2019; Fecher *et al.*, 2026).

CONCLUSION

Co(II) and Ni(II) complexes were successfully created in this work using 4,4-diaminodiphenyl sulfone (DADPS) as a ligand. A thorough investigation of the ligand's coordination behavior with respect to the metal ions revealed its efficient binding capacity through donor

sites, resulting in the creation of stable metal–ligand frameworks. FT-IR, UV–visible spectroscopy, and powder X-ray diffraction (PXRD) were among the analytical methods used to characterize the produced compounds. Through noticeable changes in distinctive vibrational bands and the emergence of novel metal–ligand bonding characteristics, the FT-IR study verified the coordination. By demonstrating variations in electronic transitions in comparison to the free ligand, the UV–visible spectra provided additional evidence for complex formation, suggesting ligand-to-metal charge transfer and potential d–d transitions. Powder XRD patterns confirmed successful complexation and structural organization by revealing the crystalline character of the produced complexes, with unique diffraction peaks indicating the creation of new phases in comparison to the ligand. A vibrating sample magnetometer (VSM) was used to examine the magnetic characteristics of the Co-DADPS and Ni-DADPS complexes. Compared to Ni-DADPS (1.38 emu per molecule), the Co-DADPS compound showed a substantially higher magnetic saturation value (46.1 emu per molecule). The differential in the amount of unpaired electrons and the electronic configuration of Co(II) and Ni(II) ions are responsible for this discrepancy. Stronger magnetic interactions are suggested by the cobalt complex's greater magnetic moment, while nickels comparatively lower value denotes weaker paramagnetic activity. The effective synthesis of Co(II) and Ni(II) complexes with DADPS is strongly supported by the combined spectroscopic, structural, and magnetic investigations, which further highlight the unique physicochemical characteristics of these complexes.

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