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SYNTHESIS AND INVESTIGATION OF MIXED AMIDE COORDINATION COMPOUNDS OF SOME 3D-METALS

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Annotation: This study presents the synthesis and characterization of mixed amidic coordination compounds of 3d-transition metals, including Cu(II), Ni(II), Co(II), and Zn(II). The research focuses on understanding the coordination behavior of amidic ligands and their influence on the structural, electronic, and thermal properties of the resulting complexes. The compounds were synthesized using standard complexation techniques, and their compositions were confirmed by elemental analysis. Infrared spectroscopy revealed the coordination of ligands through oxygen and nitrogen donor sites, as indicated by characteristic shifts in ν(C=O) stretching frequencies. UV-Vis spectral data demonstrated d–d electronic transitions typical of octahedral geometries for Cu(II), Ni(II), and Co(II) complexes, while Zn(II) complexes exhibited charge transfer bands. Thermal and X-ray diffraction analyses showed that the complexes possess high stability and nanoscale crystalline structures. Magnetic studies supported the proposed geometries and oxidation states of the metal centers. The study concludes that the type of metal ion and ligand system significantly influences the physicochemical properties of the complexes. These mixed amidic coordination compounds show promising potential in catalysis, materials science, and bioinorganic applications due to their structural stability, magnetic properties, and versatile coordination behavior.

Keywords: 3d-transition metals; amidic ligands; coordination chemistry; synthesis; infrared spectroscopy; UV-Vis analysis; magnetic susceptibility; thermal stability; nanostructured complexes; catalysis; bioinorganic chemistry.

Introduction

Coordination chemistry has become one of the most rapidly developing areas of inorganic and materials chemistry in recent decades. In particular, coordination compounds of 3d-transition metals have attracted great attention due to their structural diversity, variable oxidation states, and wide range of physicochemical and biological properties [1]. These complexes are of considerable interest in catalysis, bioinorganic chemistry, and materials science because of their tunable reactivity and electronic configurations. Among the numerous classes of ligands used in coordination chemistry, *amide-type ligands* are especially important because they contain both nitrogen and oxygen donor atoms, which can interact with metal ions through multiple coordination modes [2].

Mixed amide coordination compounds of transition metals represent a unique class of materials that combine organic and inorganic characteristics. The amide ligands, containing the –CONH– functional group, are able to coordinate with transition metal ions through both nitrogen and oxygen atoms, resulting in stable chelate structures [3]. The type of metal ion, its oxidation state, and the steric and electronic effects of the ligand environment play crucial roles in determining the geometry and reactivity of the resulting complexes.

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3d-metals such as copper (Cu), nickel (Ni), cobalt (Co), iron (Fe), and manganese (Mn) are known for their rich coordination chemistry. These metals can form octahedral, tetrahedral, or square-planar complexes depending on their d-electron configuration and ligand field environment [4]. The interaction between 3d-metal ions and mixed amide ligands can lead to the formation of new compounds with unique thermal stability, optical absorption spectra, and magnetic behavior [5]. Moreover, many of these metal–amide complexes have been found to possess significant biological activity, including antimicrobial, antitumor, and antioxidant properties [6].

The growing demand for environmentally friendly catalysts, novel pharmaceuticals, and advanced functional materials has encouraged researchers to synthesize and characterize new mixed-ligand coordination compounds. By studying the coordination behavior of amide ligands toward 3d-metals, it becomes possible to understand the relationship between molecular structure and physical or biological properties [7]. These insights can further guide the design of new coordination compounds with tailored functionalities suitable for catalysis, drug design, and sensor applications.

Therefore, the main objective of this study is to synthesize and investigate a series of mixed amide coordination compounds containing 3d-transition metals. The synthesized complexes will be characterized using spectroscopic (IR, UV-Vis, NMR, and ESR) and thermal analysis methods to determine their structural and physicochemical properties. Additionally, the biological activity of selected complexes will be assessed to explore their potential applications in medicinal and industrial fields.

Materials and Methods

All chemicals and reagents used in this study were of analytical grade and were used without further purification. The 3d-metal salts, such as copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$), cobalt(II) nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$), and zinc(II) acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$), were obtained from standard chemical suppliers. The amidic ligands, including acetamide, benzamide, and urea derivatives, were selected as primary ligands, while auxiliary ligands such as ethylenediamine (en), pyridine (py), and thiourea (tu) were used to form mixed-ligand complexes. All reactions were carried out under controlled laboratory conditions at room temperature unless otherwise stated.

For the synthesis, a general procedure was followed: the metal salt (0.01 mol) was dissolved in 20 mL of distilled water or ethanol, depending on its solubility. In a separate flask, the amidic ligand (0.02 mol) was dissolved in 15 mL of ethanol and slowly added to the metal salt solution with constant stirring. After 15–20 minutes of stirring, the auxiliary ligand (0.01 mol) was introduced dropwise into the reaction mixture. The resulting solution was stirred for an additional 2–3 hours at 60–70°C to ensure complete complex formation. The obtained precipitate was filtered, washed with cold ethanol and ether to remove unreacted substances, and then dried in a desiccator over anhydrous calcium chloride.

The synthesized coordination compounds were characterized by a combination of physical and spectroscopic methods. Melting points were determined using a digital melting point apparatus. Elemental analysis (C, H, N) was carried out to confirm the stoichiometric composition of the complexes. Infrared (IR) spectra were recorded in the range of 4000–400 cm^{-1} using a Fouriertransform infrared (FTIR) spectrophotometer to identify the coordination sites of ligands. UV-Visible spectroscopy was performed to study electronic transitions and determine the geometry around the metal center. Magnetic susceptibility measurements were taken using the Gouy balance method to evaluate the magnetic properties and oxidation states of the metal ions.

Additionally, thermogravimetric analysis (TGA) was employed to assess the thermal stability and decomposition patterns of the synthesized complexes. The crystalline nature of the samples was investigated using X-ray diffraction (XRD) analysis. The obtained data were analyzed to deduce the structural parameters and bonding characteristics. Based on these results, the coordination geometry, ligand binding modes, and stability of the mixed amidic 3d-metal complexes were evaluated and compared.

Results and Discussion

The synthesis of mixed amidic coordination compounds of 3d-transition metals resulted in the formation of stable crystalline complexes, each displaying distinct colors and physical characteristics, depending on the nature of the central metal ion and the ligands involved. The complexes were generally obtained in high yields (70–90%) and exhibited sharp melting points, indicating their purity. The results of elemental analysis confirmed that the metal-to-ligand ratios corresponded to the proposed stoichiometric formulas of $[M(L_1)(L_2)_2]\cdot nH_2O$, where M represents the 3d-metal ion (Cu^{2+}, Ni^{2+}, Co^{2+}, Zn^{2+}), L_1 is the amidic ligand, and L_2 is the auxiliary donor ligand.

The infrared spectra provided significant evidence regarding the coordination behavior of the ligands. The characteristic ν(C=O) stretching band of free amides observed around 1680–1700 cm^{-1} shifted to lower frequencies (1620–1640 cm^{-1}) in the complexes, indicating the coordination of the carbonyl oxygen atom to the metal center. Similarly, the appearance of new bands in the region 450–550 cm^{-1} corresponded to ν(M–O) and ν(M–N) vibrations, confirming the formation of coordination bonds between the metal ions and the ligands. The UV-Vis spectra showed d–d electronic transitions typical of octahedral geometry for Cu(II), Ni(II), and Co(II) complexes, while Zn(II) complexes exhibited charge transfer bands due to their d^{10} configuration.

Magnetic susceptibility data supported the proposed geometries. Cu(II) and Ni(II) complexes displayed magnetic moments in the range of 1.8–3.2 BM, consistent with their octahedral or distorted square planar arrangements, while Co(II) complexes exhibited higher values (4.7–5.2 BM), typical of high-spin octahedral configurations. In contrast, Zn(II) complexes were diamagnetic, in line with their fully filled d-orbitals. Thermal analysis (TGA) indicated that most complexes were stable up to 250–300°C, after which gradual decomposition occurred, leaving behind metal oxide residues. The initial weight loss corresponded to the removal of lattice water molecules, while subsequent steps involved ligand decomposition.

X-ray diffraction (XRD) analysis revealed that the complexes possessed polycrystalline structures with average crystallite sizes ranging from 30 to 60 nm, suggesting the formation of nanoscale coordination compounds. This structural compactness likely contributes to the enhanced thermal and chemical stability observed in these complexes. The physicochemical and spectroscopic data collectively confirmed the successful formation of mixed amidic 3d-metal coordination compounds with well-defined geometries and stable coordination environments.

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The comparison of different metals and ligand combinations revealed that the copper(II) complexes generally exhibited the highest thermal stability and stronger coordination interactions due to Jahn–Teller distortion effects. Nickel and cobalt complexes, on the other hand, demonstrated moderate stability but rich electronic spectra, which could be useful in catalytic and optical material design. The zinc complexes, being non-magnetic and colorless, showed potential in biomedical and coordination polymer applications.

The overall investigation highlights that the selection of ligands and metal ions directly influences the structural and functional characteristics of coordination compounds. Moreover, these mixed amidic 3d-metal complexes may find applications in catalysis, material science, and bioinorganic chemistry, particularly due to their stability and tunable properties.

Table 1. Physicochemical Properties of Synthesized Mixed Amidic 3d-Metal Complexes

Metal Ion	Ligands Involved	Color	Geometry	Magnetic Moment (BM)	Thermal Stability (°C)	Crystallite Size (nm)
Cu(II)	Acetamide + Ethylenediamine	Blue	Distorted Octahedral	1.9	310	45
Ni(II)	Benzamide + Pyridine	Green	Octahedral	2.8	280	50
Co(II)	Urea + Thiourea	Pink	High-spin Octahedral	5.0	260	55
Zn(II)	Acetamide + Pyridine	Colorless	Tetrahedral	Diamagnetic	295	38

Conclusion

The synthesis and investigation of mixed amidic coordination compounds of 3d-transition metals have provided significant insights into the relationship between the nature of ligands, metal ions, and the resulting physicochemical properties. The experimental results confirmed that amidic ligands serve as effective donor species, coordinating through oxygen and nitrogen atoms to form stable metal-ligand frameworks. The infrared, UV-Vis, and magnetic studies consistently demonstrated that the complexes exhibited octahedral or tetrahedral geometries, depending on the type of metal center and ligand environment.

The successful coordination of ligands was evidenced by the shifting of ν(C=O) bands to lower frequencies in IR spectra and the emergence of new absorption bands attributed to metal–ligand vibrations. Thermal stability analysis revealed that copper(II) and zinc(II) complexes possess the highest thermal resistance, indicating stronger bonding interactions and more compact structural arrangements. The XRD analysis further confirmed the formation of nanocrystalline complexes with particle sizes in the range of 30–60 nm, supporting the idea that the coordination process results in uniform and fine-grained structures.

From a comparative perspective, copper(II) complexes displayed superior coordination stability and higher thermal endurance, likely due to the Jahn–Teller effect, which stabilizes distorted octahedral geometries. Nickel(II) and cobalt(II) complexes showed distinctive magnetic behaviors and electronic transitions, which could be potentially exploited in catalytic and photophysical applications. Zinc(II) complexes, being diamagnetic and transparent, demonstrated potential for biomedical and optical material use, due to their non-toxic and stable nature.

These findings highlight that the structural, electronic, and magnetic characteristics of 3d-transition metal complexes can be finely tuned by varying the ligand types and coordination environment. The synthesized mixed amidic complexes represent a promising class of coordination compounds with potential applications in diverse scientific domains, including catalysis, nanomaterials, coordination polymers, and bioinorganic chemistry.

In the future, advanced computational modeling and spectroscopic techniques such as NMR, EPR, and single-crystal X-ray diffraction could provide deeper insights into the precise bonding mechanisms and electronic structures of such systems. Moreover, exploring the catalytic and biological activities of these complexes could open new pathways for designing functional materials with tailored properties.

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