QO‘QON DAVLAT PEDAGOGIKA INSTITUTI ILMIY XABARLARI
(2026-yil 2-son)

TABIIY FANLAR

NATURAL SCIENCES

SYNTHESIS, STRUCTURAL CHARACTERIZATION AND ANTIMICROBIAL EVALUATION OF A NOVEL COPPER(II) ACETATE–ISONICOTINAMIDE COORDINATION COMPLEX

https://doi.org/10.70728/a.series.v08.i02.004

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Abstract. In this article, a coordination compound of copper acetate with isonicotinamide was synthesized and its structure was analyzed using physicochemical methods.

Keywords: Cu(II) acetate monohydrate, isonicotinamide, absorption bands, IR spectrum, X-ray diffraction (XRD)

Аннотация.В статье синтезировано новое координационное соединение ацетата меди(II) с изоникотинамидом. Структура полученного комплекса исследована с использованием физико-химических методов анализа.

Ключевые слова: Ацетат меди(II) моногидрат, изоникотинамид, полосы поглощения, ИК-спектр, рентгеновская дифракция (XRD).

Annotatsiya. Mazkur ishda mis(II) atsetat va izonikotinamid asosida yangi koordinatsion birikma sintez qilindi. Olingan kompleksning tuzilishi fizik-kimyoviy tahlil usullari yordamida o‘rganildi.

Kalit so‘zlar: Mis(II) atsetat monogidrat, izonikotinamid, yutilish polosalari, IQ-spektr, rentgen difraksiyasi (XRD).

Introduction. Coordination chemistry of transition metals continues to attract considerable attention due to the structural diversity and functional properties of metal–organic compounds. Among nitrogen-containing ligands, isonicotinamide (INA) represents an important heterocyclic amide capable of participating in versatile coordination modes. Owing to the presence of both a pyridine nitrogen atom and an amide carbonyl group, INA can act as a potential donor ligand toward metal ions, leading to the formation of stable coordination architectures.

Copper(II) ions are well known for their variable coordination geometries and biological relevance. As an essential trace element, copper plays a key role in numerous enzymatic processes, redox reactions, and electron-transfer mechanisms. The ability of Cu(II) to interact with N- and O-donor ligands makes it particularly suitable for constructing coordination compounds with potential biological and catalytic applications.

Complexes of isonicotinamide with transition metals have been widely investigated due to their structural flexibility and supramolecular hydrogen-bonding capabilities. In many reported systems, INA coordinates through the pyridine nitrogen atom, while the amide group contributes to stabilization of the crystal lattice via intermolecular interactions. Such features make INA-based complexes attractive candidates in crystal engineering and bioinorganic chemistry.

The present study focuses on the synthesis of a new Cu(II) coordination compound with isonicotinamide using a solvent evaporation approach. The structural features of the obtained complex were examined by infrared spectroscopy and X-ray diffraction analysis. Special attention was given to identifying coordination sites and confirming the formation of a new crystalline phase. Additionally, the antimicrobial properties of the synthesized compound were evaluated against selected clinical opportunistic pathogenic strains to assess its potential biological relevance.

Materials and Methods

To determine the individuality and phase composition of the synthesized complex compound, a high-resolution X-ray diffractometer **Rigaku MiniFlex 600** (Rigaku Corporation, Japan) was used. The instrument was operated under the following conditions: X-ray radiation source – Cu (Kα) radiation, $\lambda = 1.5406$ Å; scanning range: $2\theta = 3°–90°$; detector: software-controlled system.

To analyze changes in the functional groups of the ligand, a 32-scan **PerkinElmer Spectrum Two** (UK) IR spectrophotometer was employed. The spectra were recorded in the range of 400–4000 cm^{-1}.

For the synthesis of the complex, $Cu(CH_3COO)_2 \cdot H_2O$ and isonicotinamide were taken in a 1:2 molar ratio. The starting materials were dissolved in DMF and stirred at 50 °C for 4–6 hours using a magnetic stirrer. The resulting solution was slowly evaporated at room temperature, yielding blue-colored crystals. The crystals were filtered and dried under vacuum.

Results and Discussion

In the IR spectrum of the free isonicotinamide ligand, broad absorption bands observed in the 3300–3200 cm^{-1} region correspond to the ν(N–H) stretching vibrations characteristic of the amide group. These signals indicate the presence of intra- and intermolecular hydrogen bonding.

An intense absorption band observed in the 1660–1680 cm^{-1} region is attributed to the ν(C=O) stretching vibration of the amide carbonyl group. In addition, absorptions in the 1600–1550 cm^{-1} region are associated with the ν(C=N) and ν(C=C) stretching vibrations of the pyridine ring (Figure 2).

In the IR spectrum of the free isonicotinamide molecule, the characteristic carbonyl group vibration ν(C=O) appears at 1655 cm^{-1}, the N–H stretching vibration at 3364 cm^{-1}, and the C–N stretching vibration at 1218 cm^{-1}.

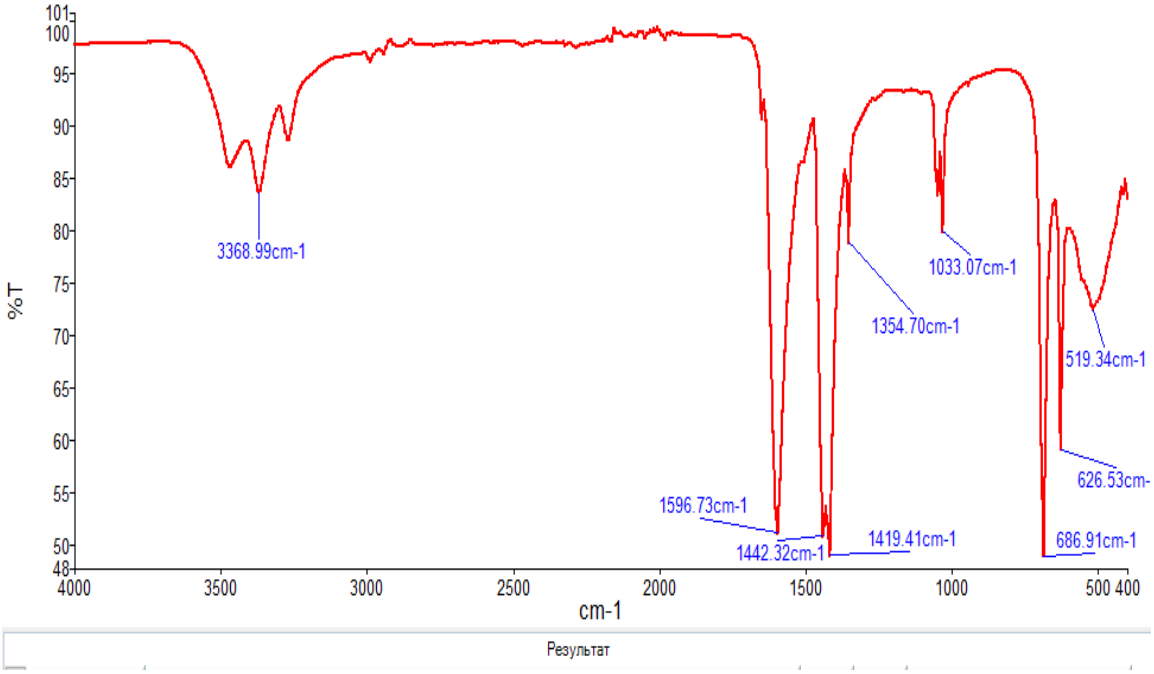


Figure 1. IR Spectrum of $Cu(CH_3COO)_2 \cdot H_2O$

In the IR spectrum of copper(II) acetate, strong absorption bands are observed in the regions 1550–1600 cm^{-1} and 1400–1450 cm^{-1}, which correspond to the asymmetric and symmetric ν(COO^-) stretching vibrations of the carboxylate ion, respectively. These signals indicate that the Cu(II) ion is coordinated through oxygen donor atoms. The spectral characteristics of the carboxylate groups confirm the formation of a stable coordination environment around the metal center (Figure 1).

In the IR spectra of isonicotinamide-based complexes, shifts in the characteristic ν(C=O) and ν(N–H) stretching frequencies of the amide group confirm the coordination of the ligand to the metal ion. Additionally, changes in the ν(C=N) stretching vibrations of the pyridine ring are considered important evidence of the coordination process.

Electronic and magnetic studies are also widely used to determine the geometric structure of these complexes.

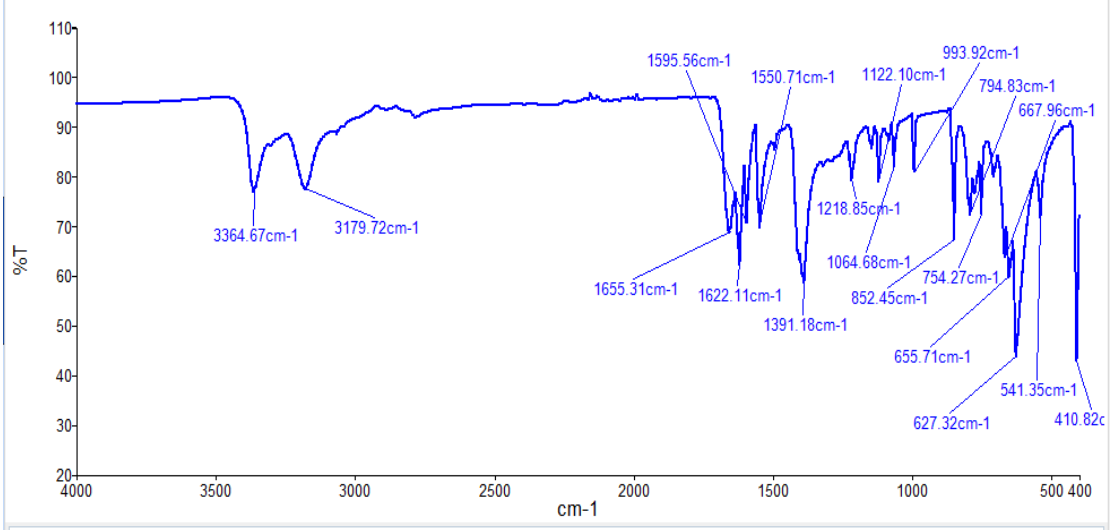


Figure 2. FTIR spectrum of Isonicotinamide

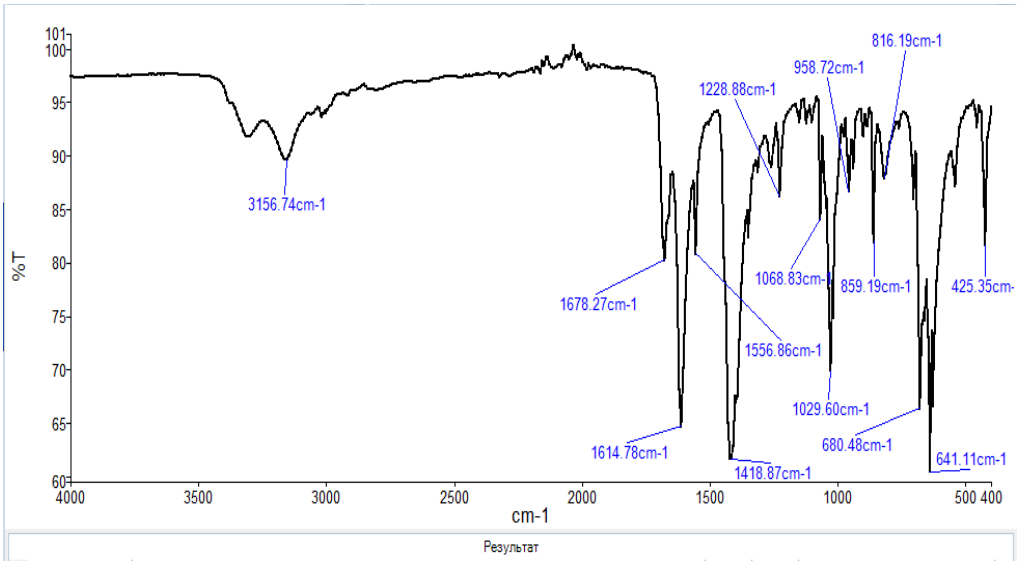


Figure 3. FTIR spectrum of the $[Cu(CH_3COO)_2 \cdot NC_5H_4CONH_2] \cdot 2H_2O$ complex

The FTIR spectrum of the synthesized $[Cu(CH_3COO)_2 \cdot NC_5H_4CONH_2] \cdot 2H_2O$ complex was compared with those of the free ligand, isonicotinamide, and copper(II) acetate monohydrate. Significant differences were observed between the spectra.

In particular, the amide ν(C=O) stretching vibration shifts from 1655 cm^{-1} to 1615 cm^{-1}, i.e., toward lower frequencies, indicating the coordination of the carbonyl oxygen atom to the Cu(II) ion. This shift to lower wavenumbers can be attributed to the weakening of the C=O bond upon coordination.

Furthermore, the shift in the ν(C=N) vibrations of the pyridine ring confirms the formation of a metal–nitrogen (Cu–N) coordination bond. These changes indicate that the isonicotinamide ligand acts as a donor toward the Cu(II) ion, coordinating through nitrogen and oxygen donor atoms.

New absorption bands appearing in the 500–600 cm^{-1} region in the complex spectrum are assigned to Cu–N and Cu–O metal–ligand vibrations. Such bands are absent in the

spectrum of the free ligand, which further supports the formation of the coordination compound.

The presence of characteristic ν_as(COO⁻) and ν_s(COO⁻) vibrations of the acetate groups suggests that acetate ions also participate in the coordination environment (Figure 3).

X-ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) patterns of isonicotinamide, copper(II) acetate monohydrate, and the synthesized complex were compared in terms of interplanar spacings and relative intensities. Significant differences were observed among the diffraction patterns, confirming that the obtained complex is an individual crystalline compound distinct from the starting materials.

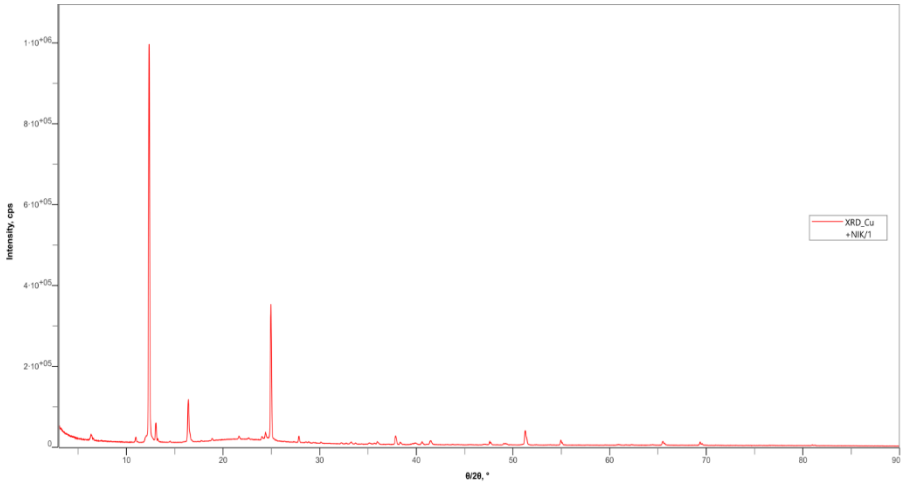


Figure 5. X-ray diffraction pattern of $[Cu(CH_3COO)_2 \cdot NC_5H_4CONH_2] \cdot 2H_2O$

The X-ray diffraction (XRD) analysis of the synthesized sample was carried out in the 2θ range of 5–90°. The diffractogram shows a series of sharp and high-intensity diffraction peaks, indicating that the sample possesses a well-developed crystalline structure.

The most intense peaks are observed at approximately 2θ ≈ 11–13°, 17–18°, and 26–27°. These reflections differ significantly from the characteristic diffraction maxima of the starting materials, confirming the formation of a new crystalline phase. The disappearance or decrease in intensity of certain peaks characteristic of the initial Cu(II) salt indicates that the metal ion has formed a coordination complex with the ligand.

The low background level and the sharpness of the peaks suggest that the sample contains little to no amorphous phase. The narrow diffraction peaks indicate relatively well-ordered crystallites and a high degree of crystallinity. At diffraction angles above 30°, additional low-intensity but clearly distinguishable peaks are observed, which suggest that the complex compound possesses a relatively intricate crystal structure (Figure 5).

Moreover, the absence of strong peaks corresponding to foreign impurity phases confirms that a pure coordination complex was obtained during the synthesis. The XRD results are consistent with the infrared spectral analysis, further supporting the formation of a new coordination complex.

Evaluation of Antimicrobial Activity Against Clinical Opportunistic Pathogenic Strains.

The antimicrobial activity of the synthesized compounds was evaluated against clinical opportunistic pathogenic strains using the agar diffusion method. Prior to testing, the compounds were dissolved in distilled water to obtain the required working concentrations. Microbial suspensions with a density of not less than 1×10^7 CFU/mL were prepared and uniformly inoculated onto meat–peptone agar (MPA) plates using the lawn culture technique.

Distilled water served as the negative control. An aliquot of 100 μL of each test solution was applied onto the agar surface by the drop diffusion method. The inoculated plates were incubated aerobically at 30°C for 24 h.

Following incubation, the diameters of the inhibition zones were measured in millimeters using a digital caliper. All experiments were conducted in triplicate to ensure reproducibility. The obtained data were expressed as mean ± standard deviation (SD), and statistical analysis was performed using appropriate significance tests.

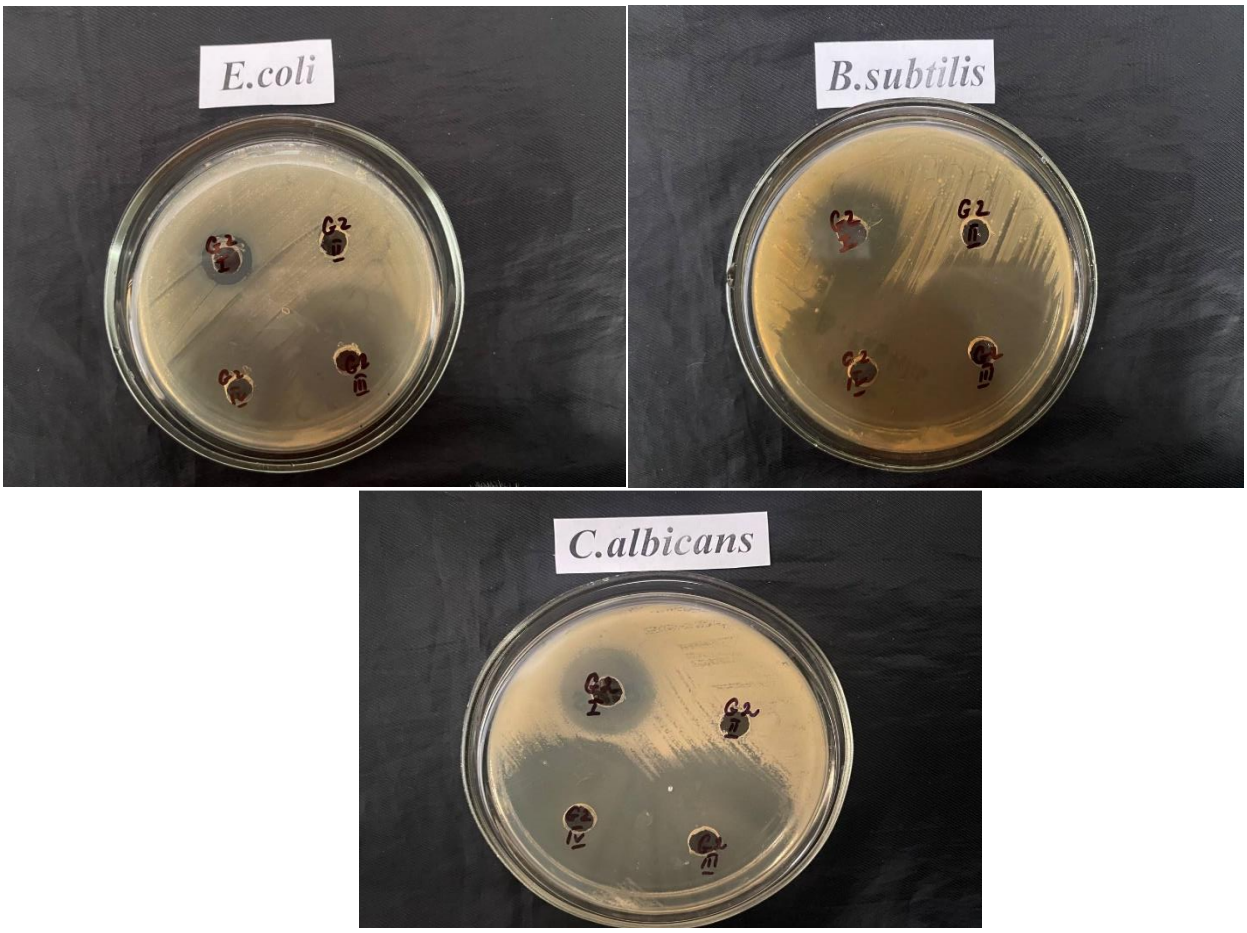
The results demonstrated that the negative control (distilled water) exhibited no inhibitory effect on microbial growth. In contrast, the tested compounds showed moderate to pronounced antimicrobial activity, as evidenced by the formation of distinct inhibition zones. These findings indicate that the investigated compounds contain biologically active moieties capable of suppressing the growth of opportunistic pathogenic microorganisms.

Overall, the data suggest that the studied compounds represent promising candidates for further pharmacological and microbiological investigations. Nevertheless, additional studies, including minimum inhibitory concentration (MIC) determination and mechanistic evaluation, are required to validate their potential for practical applications.

Table 1

(The concentration of opportunistic pathogenic strains was not less than 1×10^7 CFU/mL)

№	Materials	E. coli	C. albicans	B. subtilis
1	Cu(CH3COO)2·2H20	10mm ± 0,1mm	25mm ± 0,3mm	25mm ± 0,3mm
2	Water	0 mm	0 mm	0 mm
3	Complex	30mm ± 0,3mm	40mm ± 0,4mm	40mm ± 0,4mm
4	Isonicotinamide	30mm ± 0,3mm	40mm ± 0,4mm	35mm ± 0,4mm



Conclusion

New coordination complexes based on copper(II) acetate monohydrate and isonicotinamide were successfully synthesized. Spectroscopic and thermal analyses confirmed the presence of ligand–metal coordination, the formation of a new solid phase, and enhanced stability of the resulting complexes. The obtained results indicate that these complexes may have potential applications in future biological and pharmaceutical studies.

The biological and catalytic activity of the Cu(II) ion increases the interest in its complexation with various nitrogen-containing ligands. Isonicotinamide, due to the presence of a pyridine ring and an amide functional group, acts as an efficient donor–acceptor ligand. Therefore, Cu(II)–isonicotinamide complexes are of considerable importance in the fields of crystal engineering, pharmaceutical chemistry, and materials science. The present study demonstrates that the synthesized coordination compound exhibits significant antimicrobial activity against clinical opportunistic pathogenic strains. In comparison with the precursor salt $Cu(CH_3COO)_2 \cdot 2H_2O$ and the free ligand isonicotinamide, the complex showed enhanced inhibitory effects, particularly against *C. albicans* and *B. subtilis*, where inhibition zones reached up to 40 mm.

The absence of antimicrobial activity in the negative control confirms that the observed effects are exclusively associated with the tested compounds. The increased activity of the complex relative to the starting materials suggests that metal coordination plays a crucial role in enhancing biological efficacy. This improvement may be attributed to

chelation effects, which can increase lipophilicity and facilitate penetration through microbial cell membranes, thereby improving interaction with intracellular targets.

Overall, the results indicate that the synthesized complex possesses promising antimicrobial potential and may serve as a valuable candidate for further pharmacological development. Nevertheless, additional investigations, including minimum inhibitory concentration (MIC) determination, cytotoxicity assessment, and mechanistic studies, are required to fully elucidate its therapeutic applicability.

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