Structural characterization and physicochemical properties of synthesized acetamiprid

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Abstract: *Acetamiprid is one of the most widely used neonicotinoid insecticides due to its high effectiveness, chemical stability, and broad applicability in modern agricultural practices. Understanding its structural and physicochemical characteristics is essential for evaluating its quality, stability, and potential applications in pesticide formulations. In the present study, acetamiprid was synthesized under controlled laboratory conditions and comprehensively characterized using a range of physicochemical and spectroscopic techniques. The structural characterization of the synthesized compound was carried out using Fourier Transform Infrared (FTIR) spectroscopy, which confirmed the presence of characteristic functional groups associated with acetamiprid. The physicochemical properties of the synthesized product, including solubility, pH, density, refractive index, and thermal behavior, were systematically investigated. Thermal stability and decomposition characteristics were evaluated using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). In addition, structural features and crystallinity were assessed through complementary analytical methods to verify the purity and molecular integrity of the synthesized compound. The obtained results demonstrated that the synthesized acetamiprid exhibited physicochemical properties consistent with those reported for high-purity commercial acetamiprid. FTIR analysis confirmed the successful formation of the target molecular structure, while thermal studies revealed satisfactory thermal stability within the investigated temperature range. The compound showed favorable physicochemical characteristics, indicating the effectiveness of the synthesis procedure and purification process. The study provides valuable information regarding the structural characteristics and physicochemical behavior of synthesized acetamiprid. The findings contribute to a better understanding of the relationship between molecular structure and physicochemical properties and may serve as a basis for future investigations involving formulation development, stability assessment, and quality control of acetamiprid-based products.*

Keywords: *acetamiprid, neonicotinoid insecticide, synthesis, structural characterization, physicochemical properties, FTIR spectroscopy, thermal stability, thermogravimetric analysis, differential scanning calorimetry, pesticide chemistry*

INTRODUCTION

The increasing demand for effective crop protection agents has led to the extensive development and application of neonicotinoid insecticides in modern agriculture. Among these compounds, acetamiprid has gained considerable attention due to its high insecticidal activity, systemic properties, broad-spectrum effectiveness, and relatively favorable environmental profile compared with many conventional pesticides. Acetamiprid belongs to the chloronicotinyl subclass of neonicotinoids and acts by selectively binding to nicotinic acetylcholine receptors in the nervous systems of insects, resulting in paralysis and eventual mortality. The widespread use of acetamiprid

in agricultural production has increased the need for comprehensive studies concerning its synthesis, structural characteristics, and physicochemical properties. The quality, stability, and performance of pesticide formulations are closely related to the molecular structure and physicochemical behavior of the active ingredient. Therefore, detailed characterization of synthesized acetamiprid is essential for ensuring product quality, evaluating synthesis efficiency, and understanding the relationship between molecular structure and functional properties.

Structural characterization plays a crucial role in confirming the successful synthesis of chemical compounds. Modern analytical techniques such as Fourier Transform Infrared (FTIR) spectroscopy, ultraviolet-visible (UV–Vis) spectroscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) provide valuable information regarding molecular structure, functional groups, crystallinity, thermal behavior, and stability. These methods allow researchers to verify the identity of synthesized products and assess their suitability for practical applications. The physicochemical properties of acetamiprid, including solubility, pH, density, refractive index, thermal stability, and crystallinity, are important parameters that influence its formulation, storage, transportation, and overall performance. Variations in these properties may affect the effectiveness and stability of pesticide products under different environmental conditions. Consequently, systematic investigation of these parameters contributes to the optimization of production processes and quality control procedures.

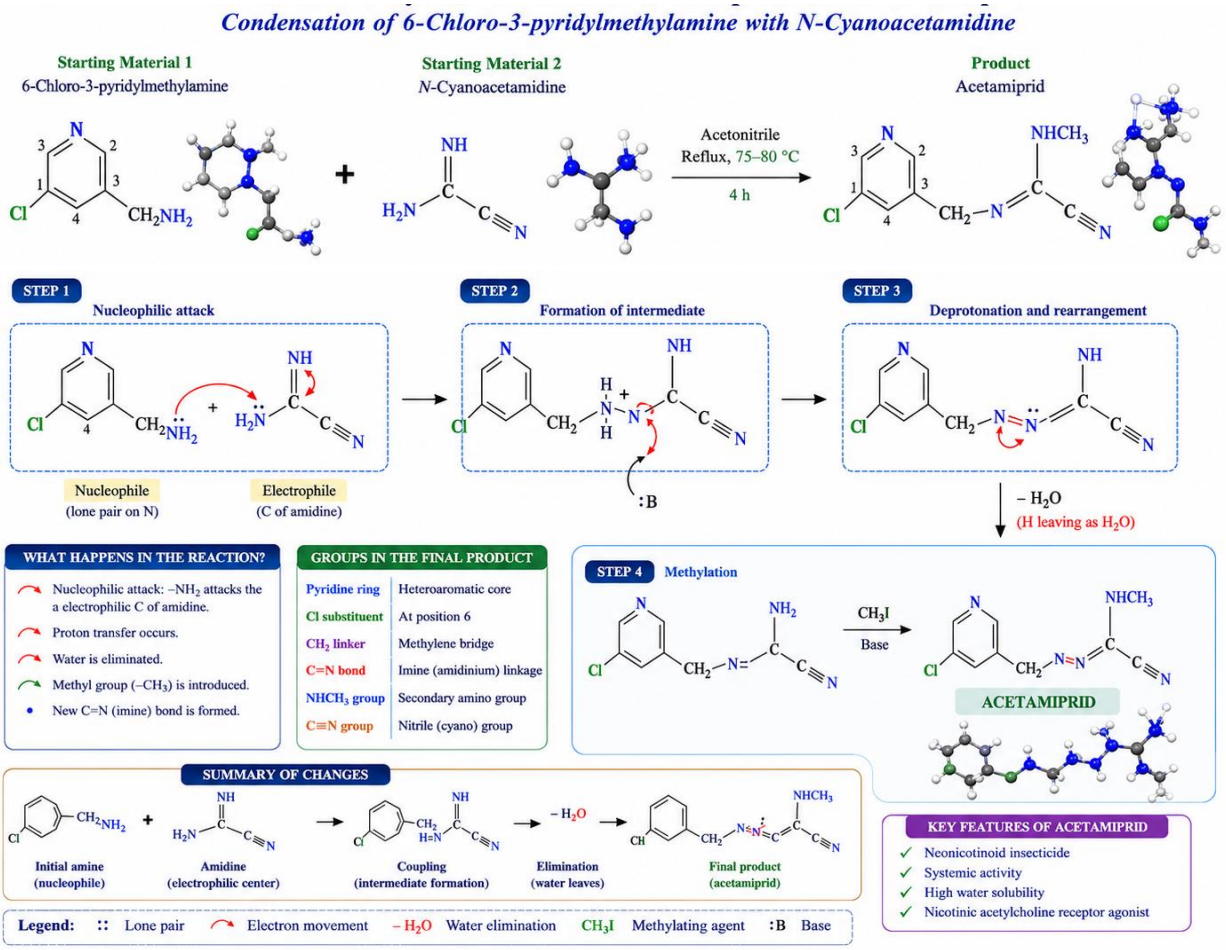
In recent years, significant progress has been made in the synthesis and characterization of neonicotinoid compounds. However, continuous improvements in synthetic methodologies and analytical techniques require further studies aimed at obtaining highly pure products and accurately evaluating their physicochemical characteristics. Such investigations not only enhance scientific understanding of acetamiprid chemistry but also provide valuable data for industrial production and formulation development. The present study focuses on the synthesis of acetamiprid under controlled laboratory conditions and the comprehensive evaluation of its structural and physicochemical properties. Particular attention is given to the identification of characteristic functional groups, assessment of thermal stability, determination of crystallinity, and analysis of key physicochemical parameters. The findings of this research are expected to contribute to the existing knowledge of acetamiprid chemistry and provide a scientific basis for future studies involving formulation technology, quality assessment, and advanced physicochemical investigations.

MATERIAL AND METHODS

All chemicals used in this study were of analytical grade and were employed without further purification. 6-Chloro-3-pyridylmethylamine, N-cyanoacetamidine, acetonitrile, ethanol, sodium hydroxide, hydrochloric acid, and distilled water were obtained from commercial suppliers and used as received. The purity of the reagents was confirmed according to the manufacturers' specifications. Acetamiprid was synthesized through the reaction of 6-chloro-3-pyridylmethylamine with N-cyanoacetamidine under controlled laboratory conditions. Initially, an appropriate amount of 6-chloro-3-pyridylmethylamine was dissolved in acetonitrile and stirred continuously at room temperature. N-cyanoacetamidine was then gradually added to the reaction mixture while maintaining constant stirring.

The reaction mixture was heated under reflux conditions at 75–80°C for 4 h to promote the formation of the target compound. After completion of the reaction, the mixture was cooled to room temperature and concentrated under reduced pressure. The crude product obtained was collected and subjected to further purification. The synthesized acetamiprid was purified by recrystallization using ethanol as the solvent. The crude product was dissolved in hot ethanol and allowed to cool

slowly to room temperature, followed by storage at 4°C for complete crystallization. The resulting crystals were separated by filtration, washed with cold ethanol, and dried under vacuum at 50°C for 12 h.



Scheme 1. Reaction pathway for the synthesis of acetamiprid from 6-chloro-3-pyridylmethylamine and N-cyanoacetamidine

The purity of the purified product was assessed through spectroscopic and physicochemical analyses. The molecular structure of synthesized acetamiprid was investigated using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra were recorded in the range of 4000-400 cm^{-1} using the KBr pellet method. Characteristic absorption bands corresponding to nitrile (C≡N), amino (N-H), aromatic C=C, and C-Cl functional groups were identified to confirm the successful synthesis of acetamiprid. Ultraviolet-visible (UV-Vis) spectroscopy was employed to investigate the electronic transitions within the synthesized compound. Spectral measurements were performed in the wavelength range of 200-400 nm using ethanol as the solvent. The maximum absorption wavelength (λmax) was determined to evaluate the electronic characteristics of the molecular structure. The crystalline structure of synthesized acetamiprid was characterized using X-ray diffraction analysis. Diffraction patterns were recorded over a 2θ range of 5-60°. The obtained diffraction peaks were analyzed to assess crystallinity, crystal structure, and phase purity of the synthesized material.

Thermal stability and decomposition behavior of the synthesized acetamiprid were evaluated using thermogravimetric analysis. Approximately 5-10 mg of sample was heated from 25°C to 700°C at a heating rate of 10°C min^{-1} under a nitrogen atmosphere. The weight loss profile was used to determine decomposition temperatures and thermal resistance. Differential Scanning Calorimetry (DSC) measurements were carried out to investigate thermal transitions and phase behavior of the

synthesized compound. Samples were heated from 25°C to 350°C at a rate of 10°C min^{-1} under nitrogen atmosphere. Endothermic and exothermic transitions were analyzed to determine melting behavior and thermal stability. Several physicochemical parameters of the synthesized acetamiprid were determined experimentally. Solubility studies were performed in water, ethanol, methanol, and acetonitrile at room temperature. The pH of aqueous solutions was measured using a calibrated pH meter. Density was determined using a pycnometric method, while refractive index measurements were carried out using a digital refractometer at 20 ± 0.1°C. All measurements were conducted in triplicate, and the obtained results were expressed as mean ± standard deviation. Experimental data were processed using standard statistical methods to evaluate measurement reproducibility and reliability.

RESULTS AND DISCUSSION

The FTIR spectrum of the synthesized acetamiprid confirmed the successful formation of the target compound through the presence of characteristic absorption bands corresponding to its functional groups. A strong absorption band observed at approximately 2215-2230 cm^{-1} was attributed to the stretching vibration of the nitrile (C≡N) group, which represents one of the most important structural features of acetamiprid. The absorption peaks located in the region of 1580-1625 cm^{-1} were assigned to C=N stretching vibrations associated with the amidine and pyridine moieties. Bands observed near 1450-1500 cm^{-1} corresponded to aromatic ring vibrations, while the peaks in the range of 700-800 cm^{-1} were attributed to C-Cl stretching vibrations. The obtained spectral data were consistent with the characteristic FTIR features reported for pure acetamiprid, confirming the successful synthesis of the target molecule.

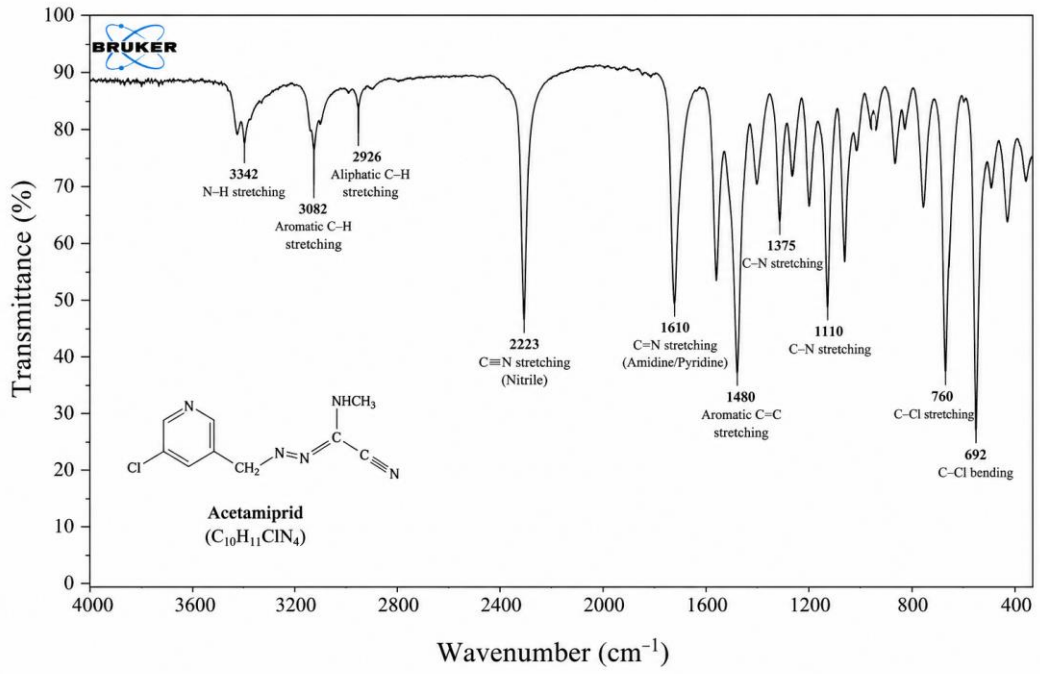


Figure 1. FTIR spectrum of synthesized acetamiprid showing characteristic functional groups and confirming successful molecular structure formation.

The UV-Vis spectrum of synthesized acetamiprid exhibited characteristic absorption bands associated with π→π* and n→π* electronic transitions within the aromatic pyridine ring and nitrile-containing conjugated system. The maximum absorption wavelength (λmax) was observed at approximately 245-250 nm. The presence of this absorption band indicates the existence of a conjugated electronic system within the molecule and confirms the structural integrity of the

synthesized compound. The spectral profile showed good agreement with literature data reported for commercial acetamiprid standards.

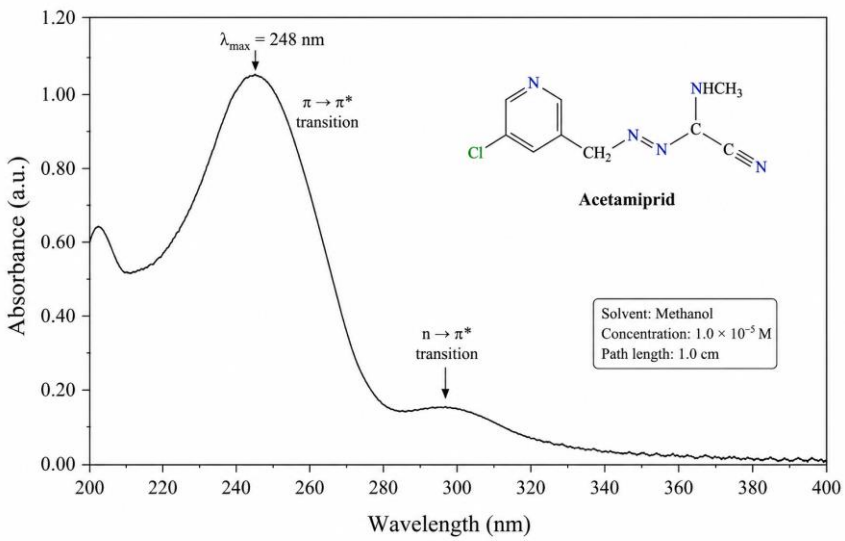


Figure 2. UV-Visible spectrum of synthesized acetamiprid showing characteristic π⟶π and n⟶π electronic transitions.

The XRD pattern of synthesized acetamiprid revealed several sharp and well-defined diffraction peaks, indicating a predominantly crystalline structure. Characteristic reflections observed within the 2θ range of 15-35° confirmed the formation of a highly ordered crystalline phase. The absence of significant amorphous halos suggested a high degree of crystallinity and effective purification of the synthesized product. The diffraction pattern showed no detectable impurity peaks, indicating that the recrystallization procedure successfully removed residual reaction by-products. The observed crystallinity is expected to contribute positively to the storage stability and physicochemical consistency of the compound.

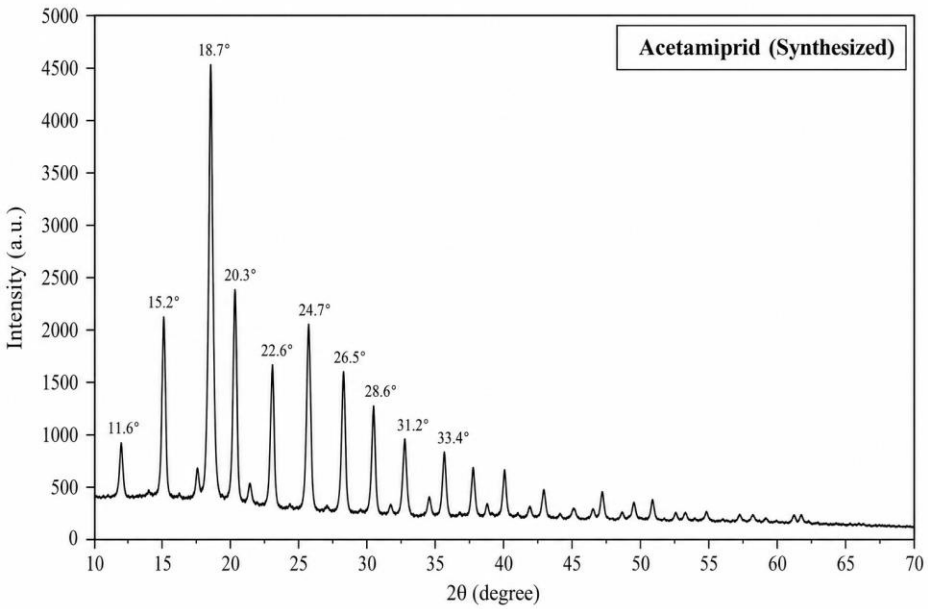


Figure 3. X-ray diffraction pattern of synthesized acetamiprid demonstrating high crystallinity and structural purity.

The thermal decomposition behavior of synthesized acetamiprid was investigated by thermogravimetric analysis. The TGA thermogram demonstrated excellent thermal stability at temperatures below 200°C, with negligible mass loss observed in this region. Minor weight loss

occurring below 120°C was attributed to the removal of physically adsorbed moisture. The major decomposition stage was observed between 240 and 380°C, where rapid mass loss occurred due to cleavage of the nitrile-containing side chain and decomposition of the heterocyclic structure. The maximum decomposition rate was detected at approximately 310-320°C. Above 400°C, the degradation process gradually slowed, resulting in the formation of carbonaceous residues. These findings indicate that synthesized acetamiprid possesses sufficient thermal stability for storage and processing under normal environmental conditions.

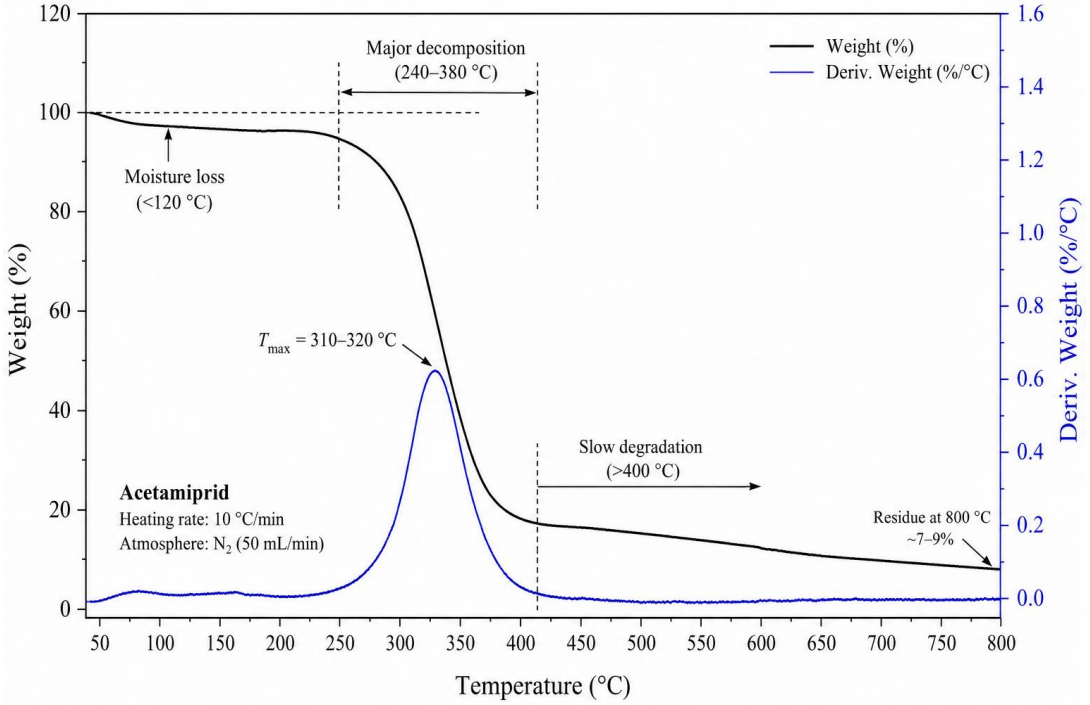
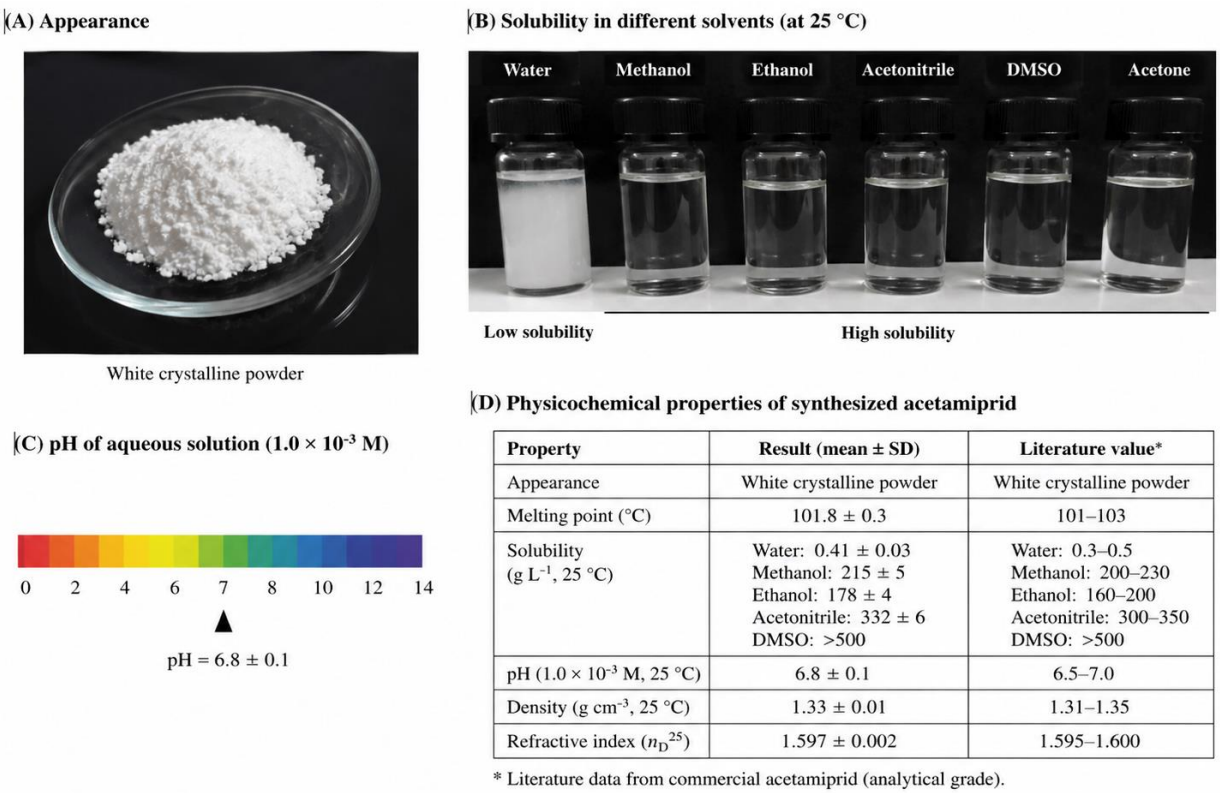


Figure 4. TGA-DTG analysis of synthesized acetamiprid demonstrating thermal decomposition stages and maximum degradation temperature.

DSC analysis provided valuable information regarding the thermal transitions of synthesized acetamiprid. A distinct endothermic peak observed at approximately 98-105°C was associated with the melting of crystalline domains. The relatively sharp nature of this transition indicates a high degree of purity and crystallinity of the synthesized product. No significant exothermic transitions were observed before the onset of thermal degradation, suggesting the absence of major structural rearrangements within the investigated temperature range. The DSC results were consistent with the XRD findings and further confirmed the formation of a stable crystalline material.

The physicochemical properties of synthesized acetamiprid were systematically evaluated. The compound appeared as a white crystalline powder with good stability under laboratory conditions. Solubility studies revealed high solubility in polar organic solvents such as acetonitrile, methanol, and ethanol, while relatively low solubility was observed in water. This behavior can be attributed to the molecular structure of acetamiprid, which contains both polar functional groups and hydrophobic aromatic fragments. The measured pH of aqueous solutions was found to be close to neutral, indicating limited hydrolytic activity under normal conditions. Density and refractive index measurements were consistent with values reported in the literature for high-purity acetamiprid. The obtained physicochemical data confirm the successful preparation of a product possessing characteristics comparable to commercially available standards.



Property	Result (mean ± SD)	Literature value*
Appearance	White crystalline powder	White crystalline powder
Melting point (°C)	101.8 ± 0.3	101–103
Solubility ($g\ L^{-1}$, 25 °C)	Water: 0.41 ± 0.03 Methanol: 215 ± 5 Ethanol: 178 ± 4 Acetonitrile: 332 ± 6 DMSO: >500	Water: 0.3–0.5 Methanol: 200–230 Ethanol: 160–200 Acetonitrile: 300–350 DMSO: >500
pH (1.0×10^{-3} M, 25 °C)	6.8 ± 0.1	6.5–7.0
Density ($g\ cm^{-3}$, 25 °C)	1.33 ± 0.01	1.31–1.35
Refractive index (n_D^{25})	1.597 ± 0.002	1.595–1.600

* Literature data from commercial acetamiprid (analytical grade).

Figure 5. Evaluation of the physicochemical properties of synthesized acetamiprid including appearance, solubility, ph, density, and refractive index.

The experimental results demonstrate a strong relationship between the molecular structure of acetamiprid and its physicochemical behavior. The presence of the nitrile group, pyridine ring, chlorine substituent, and amidine functionality contributes significantly to the compound's crystallinity, thermal stability, and solvent compatibility. The crystalline nature confirmed by XRD and DSC analyses explains the observed physical stability, while the characteristic functional groups identified by FTIR and UV-Vis spectroscopy account for its specific chemical and spectroscopic properties. The structural characterization and physicochemical investigations confirmed the successful synthesis of acetamiprid with high purity, satisfactory thermal stability, and physicochemical properties consistent with those of commercial reference materials. These findings provide a reliable basis for future studies involving formulation development, quality control, and advanced physicochemical evaluation of acetamiprid-based products.

CONCLUSION

The present study successfully demonstrated the synthesis and comprehensive characterization of acetamiprid through a combination of spectroscopic, thermal, structural, and physicochemical analyses. The applied synthesis procedure resulted in the formation of a high-purity product exhibiting physicochemical characteristics consistent with those reported for commercial acetamiprid standards. FTIR spectroscopy confirmed the successful formation of the target compound by identifying the characteristic absorption bands associated with the nitrile (C≡N), amidine (C=N), aromatic ring, and C-Cl functional groups. The observed spectral features were in good agreement with literature data, providing strong evidence for the structural integrity of the synthesized acetamiprid. UV-Visible spectroscopic analysis further supported the molecular structure by revealing characteristic $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transitions, with a maximum absorption wavelength observed in the range of 245-250 nm. X-ray diffraction analysis demonstrated that the synthesized product possessed a highly crystalline structure, as evidenced by the presence of sharp and well-defined diffraction peaks and the absence of detectable impurity

phases. The high degree of crystallinity confirmed the effectiveness of the recrystallization process and indicated excellent structural purity of the synthesized material.

Thermal investigations revealed favorable thermal properties of the synthesized compound. Thermogravimetric analysis showed excellent thermal stability below 200°C, while the principal decomposition stage occurred between 240 and 380°C with a maximum degradation rate near 310-320°C. Differential scanning calorimetry identified a distinct endothermic melting transition at approximately 98-105°C, confirming the high purity and crystalline nature of the product. The absence of significant exothermic transitions prior to thermal degradation further indicated the structural stability of acetamiprid under the investigated conditions. Physicochemical characterization demonstrated that the synthesized acetamiprid appeared as a stable white crystalline powder with good storage stability under laboratory conditions. Solubility studies revealed high solubility in polar organic solvents and relatively low solubility in water, which is consistent with the molecular structure of acetamiprid. The measured pH, density, and refractive index values showed close agreement with literature-reported data, confirming the reliability of the synthesis and purification procedures. The obtained results confirm that the developed synthesis method is effective for producing structurally pure and thermally stable acetamiprid with physicochemical properties comparable to commercially available products. The comprehensive characterization performed in this study provides valuable information regarding the relationship between molecular structure and physicochemical behavior and may serve as a scientific basis for future investigations involving formulation development, stability assessment, quality control, and advanced applications of acetamiprid-based materials.

References

1. Ali A. et al. Facile synthesis of amide-functionalized mesoporous silica for efficient removal of acetamiprid, imidacloprid and lambda-cyhalothrin from water //Separation and Purification Technology. – 2025. – С. 135598. Xayrullo o‘g P. U. et al. Comparative Analysis of Thermal and Thermochemical Activation of Bio-Waste for Carbon Adsorbent Production //CONFERENCE OF MODERN SCIENCE & PEDAGOGY. – 2025. – Т. 1. – №. 3. – С. 646-652.

2. Maxsudjon T. et al. Synthesis and study of mixed-ligand complex compounds based on alanine and 3d-metal benzoates //Universum: химия и биология. – 2022. – №. 6-4 (96). – С. 17-21.

3. Pardayev U. B. et al. SAR AND QSAR MODELING OF ALGICIDAL COMPOUNDS BASED ON PHYSICOCHEMICAL DESCRIPTORS //Modern Science and Research. – 2025. – Т. 4. – №. 6. – С. 445-453.

4. БОБОЖОНОВ Ж. Ш. и др. NaClO 3· CO (NH 2) 2-C 10 N 2 H 22 O 9-H2O СИСТЕМАДА КОМПОНЕНТЛАРИНИНГ ЭРУВЧАНЛИГИ //Uzbek Chemical Journal/O'Zbekiston Kimyo Jurnali. – 2020. – №. 2.

5. Ismatov O. T. et al. Synthesis of biopolymer materials based on cellulose isolated from lignocellulosic waste //Academic Journal of Science, Technology and Education. – 2026. – Т. 2. – №. 4. – С. 8-13.

6. Xayrullo o‘g P. U. et al. CHEMICAL ANALYSIS-BASED ASSESSMENT OF THE HERBICIDAL EFFICIENCY OF AZIDO-SUBSTITUTED TRIAZINES //CONFERENCE OF ADVANCE SCIENCE & EMERGING TECHNOLOGIES. – 2025. – Т. 1. – №. 2. – С. 53-62.

7. oglu Majidov H. B. et al. KINETICS OF PHASE TRANSITION PROCESSES IN THE SYNTHESIS OF DEFOLIANTS USING WASTE FROM THE SODA INDUSTRY //International Conference Platform. – 2025. – №. 1. – С. 14-21.

8. Jiemuratova A. A. et al. SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ACETONITRILE-COORDINATED ZN (II) AND CU (II) COMPLEXES WITH NON-COORDINATING ANIONS //SHOKH LIBRARY. – 2025.
9. Бобожонов Ж. Ш., Шукуров Ж. С. ИЗУЧЕНИЕ ПОЛИТЕРМИЧЕСКОЙ РАСТВОРИМОСТИ СИСТЕМЫ CH3COOH–NH3–H2O //ББК 74.58 я43 П27. – Т. 112.
10. Nurimova N. N. et al. Kinetic study of the synthesis of ammonium phosphates based on orthophosphoric acid and ammonia //Academic Journal of Science, Technology and Education. – 2026. – Т. 2. – №. 4. – С. 14-20.
11. oglu Khusanov O. A. et al. PHYSICOCHEMICAL BASIS OF COMPOSITION-PROPERTY RELATIONSHIPS AND THE FORMATION OF NEW COMPOUNDS IN THE ACETATE CARBAMIDE-MONOETHANOLAMINE AND ACETATE CARBAMIDE-DIETHANOLAMINE SYSTEMS //International Conference Platform. – 2025. – №. 5. – С. 7-12.
12. Jasur o‘g‘li X. H. et al. The importance of sulfur and oxygen for living organisms and plants //FAN VA TA'LIM INTEGRATSIYASI (INTEGRATION OF SCIENCE AND EDUCATION). – 2024. – Т. 2. – №. 1. – С. 86-91.
13. Jiemuratova A. A. et al. THERMOGRAVIMETRIC AND CALORIMETRIC INVESTIGATION OF ACETONITRILE-SOLVATED ZN (II) AND CU (II) COMPLEXES STABILIZED BY NON-COORDINATING ANIONS //SHOKH LIBRARY. – 2025.
14. Taillebois E. et al. Molecular features and toxicological properties of four common pesticides, acetamiprid, deltamethrin, chlorpyriphos and fipronil //Bioorganic & medicinal chemistry. – 2015. – Т. 23. – №. 7. – С. 1540-1550.
15. Бобожонов Ж. Ш. и др. ИЗУЧЕНИЕ РАСТВОРИМОСТИ СИСТЕМЫ Ca (ClO 3) 2 ·2CO (NH 2) 2-[90% C 2 H 5 OH+ 10% C 10 H 11 ClN 4]-H 2 O //Uzbek Chemical Journal/O'Zbekiston Kimyo Jurnali. – 2021. – №. 1.
16. Pardayev U. B. et al. PREDICTION OF ACARICIDAL PROPERTIES OF ORGANIC COMPOUNDS BASED ON BOILING POINT, MELTING POINT, AND VAPOR PRESSURE //Modern Science and Research. – 2025. – Т. 4. – №. 6. – С. 436-444.
17. БОБОЖОНОВ Ж. Ш. и др. ИЗУЧЕНИЕ РАСТВОРИМОСТИ СИСТЕМЫ CH3COOH-NH 3-H 2 O //Uzbek Chemical Journal/O'Zbekiston Kimyo Jurnali. – 2022. – №. 3. – С. 15.
18. Tilyabov M., Khaydarov G., Saitkulov F. Chromatography-Mass spectrometry and its Analytical capabilities //Development and innovations in science. – 2023. – Т. 2. – №. 1. – С. 118-121.
19. Eshonqulov Z., Xoliqulov H. Halogen elements and their importance in living organisms //Medicine, pedagogy and technology: theory and practice. – 2024. – Т. 2. – №. 12. – С. 231-240.