Modern American Journal of Engineering, Technology, and Innovation
ISSN(E): 3067-7939
Volume 2, Issue 1, January, 2026
Website: usajournals.org

INTERACTION OF ETHANOLAMINES WITH COPPER(II) ACETATE MONOHYDRATE

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Abstract
Ethanolamines possess biological activity and also function as growth-promoting agents for plants. Copper(II) acetate monohydrate is known to exhibit fungicidal activity against root rot diseases of agricultural crops. A number of studies have been devoted to the interaction of ethanolamine with organic and inorganic salts; however, its interaction with copper(II) acetate monohydrate and the fungicidal activity of the complexes obtained on this basis have remained largely unexplored. Based on experimental investigations, new data were obtained on the solubility and interactions of components in two systems containing water, diethanolamine, triethanolamine, and copper(II) acetate monohydrate. These systems were studied using isomolar methods.

Keywords: Chemical analysis, IR spectroscopic analysis, X-ray phase analysis, thermographic analysis, refractive index, density, viscosity, pH medium.

Introduction

At the present stage, the treatment of cotton seeds and cereal crop seeds with seed disinfectants prior to sowing is widely used to increase agricultural crop productivity. Ethanolamines possess biological activity and play an important role in stimulating protein metabolism, participating in oxidation–reduction processes, and enhancing the activity of enzymatic systems; they are also considered

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effective plant growth stimulators [1]. According to literature data, copper(II) acetate monohydrate exhibits fungicidal activity against root rot diseases of agricultural crops.

It should be noted that a number of studies have been devoted to investigating the interactions of diethanolamine and triethanolamine with inorganic and organic salts [3,4]. However, the interaction of diethanolamine and triethanolamine with copper(II) acetate monohydrate, as well as the fungicidal properties of the complexes obtained on this basis, have remained largely unexplored.

In this regard, the aim of the present study is to investigate the interaction of ethanolamines with copper(II) acetate monohydrate.

Materials and Methods

To determine the mechanism of interaction between copper(II) acetate monohydrate and ethanolamines, solutions of the following systems were studied using the isomolar series method: diethanolamine–copper(II) acetate monohydrate–water and triethanolamine–copper(II) acetate monohydrate–water. In the course of this study, concentrated ethanolamines of analytical grade (“ch”), dehydrated under vacuum at 80–90 °C, and recrystallized copper(II) acetate monohydrate were used for the synthesis of the complex compounds. The concentrations of aqueous solutions of ethanolamines and copper(II) acetate monohydrate were 1 mol L^{-1}. All measurements were carried out in a thermostated aqueous bath at 20 ± 0.1 °C.

The kinematic viscosity of the solutions was measured using a VPZh-2 capillary viscometer, the refractive index was determined with an IRF-454 BM refractometer, the relative density was measured by the pycnometric method, and the pH was determined using a 150MI pH meter.

Elemental analysis of carbon, hydrogen, nitrogen, and oxygen was performed for the synthesized compounds. Nitrogen content was determined by the Kjeldahl method [5], carbon and hydrogen by the Dumas micromethod [6], while oxygen content was calculated by difference from the total composition.

IR spectra were recorded using a SPECORD-75 spectrophotometer in the range of 4000–400 cm^{-1}. The KBr pellet technique was applied to obtain the spectra of both the initial and synthesized compounds [7,8].

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X-ray diffraction patterns of the studied compounds were obtained using a DRON-2.0 diffractometer with a Cu anode. Interplanar spacings were calculated using Hiller tables [9,10], and relative intensities were determined as percentages of the most intense reflection. Thermal analysis was carried out on a Paulik–Paulik–Erdey derivatograph at a heating rate of 10 °C min^{-1} under a protective atmosphere.

Results and Discussion

To determine the mechanism of interaction between copper(II) acetate monohydrate and di- and triethanolamines, the system was investigated using the isomolar series method [11].

Based on the data obtained from the study of the copper(II) acetate monohydrate–diethanolamine–water system in isomolar solutions [2]—including refractive index, density, viscosity, and pH isotherms—it was established that the system is characterized by two inflection points corresponding to 10.0 and 60.0 mol% diethanolamine. This confirms the formation of a new compound at a diethanolamine : copper(II) acetate monohydrate molar ratio of 1:1 (Figure 1).

These characteristic inflection points are less pronounced on the viscosity isotherms. A solid complex compound with a 1:1 composition, copper(II) acetate monohydrate–diethanolamine, $NH(C_2H_4OH)_2 \cdot (CH_3COO)_2Cu \cdot H_2O$, was isolated and identified using various analytical methods.

Table 1. Elemental composition of the complex $NH(C_2H_4OH)_2 \cdot (CH_3COO)_2Cu \cdot H_2O$

Component	Found, wt.%	Calculated for 1:1 composition, wt.%
$NH(C_2H_4OH)_2$	33.74	33.79
$(CH_3COO)_2Cu$	57.79	57.85
H_2O	8.29	8.34

The study of the solubility of the synthesized complex $NH(C_2H_4OH)_2 \cdot (CH_3COO)_2Cu \cdot H_2O$ in organic solvents showed that it is readily soluble in alcohols, poorly soluble in ethers and acetone, insoluble in benzene and

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toluene, and soluble in water at 0, 10, and 20 °C with solubilities of 6.6, 16.0, and 28.0 wt.%, respectively. The dissolution process was found to be congruent.

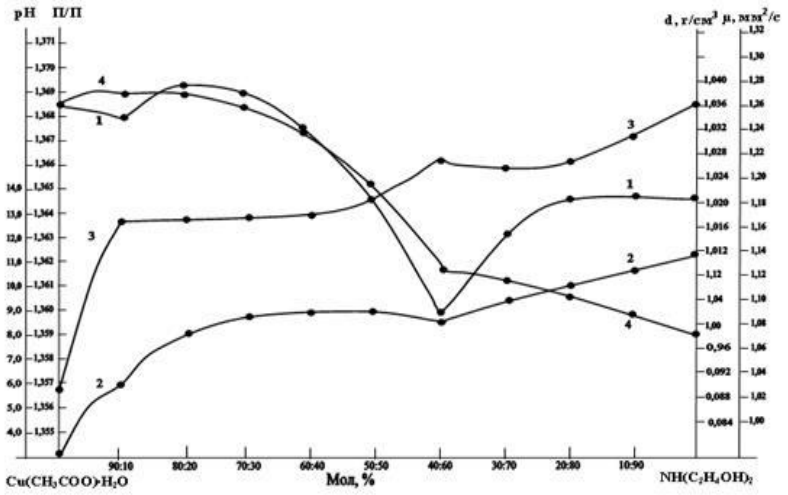

Figure 1. Copper(II) acetate monohydrate–diethanolamine–water system: 1 – refractive index, 2 – pH medium, 3 – density, 4 – viscosity.

X-ray phase analysis showed that the obtained compound, in comparison with the initial components, is characterized by new values of interplanar spacings, which confirms its individuality (Figure 2).

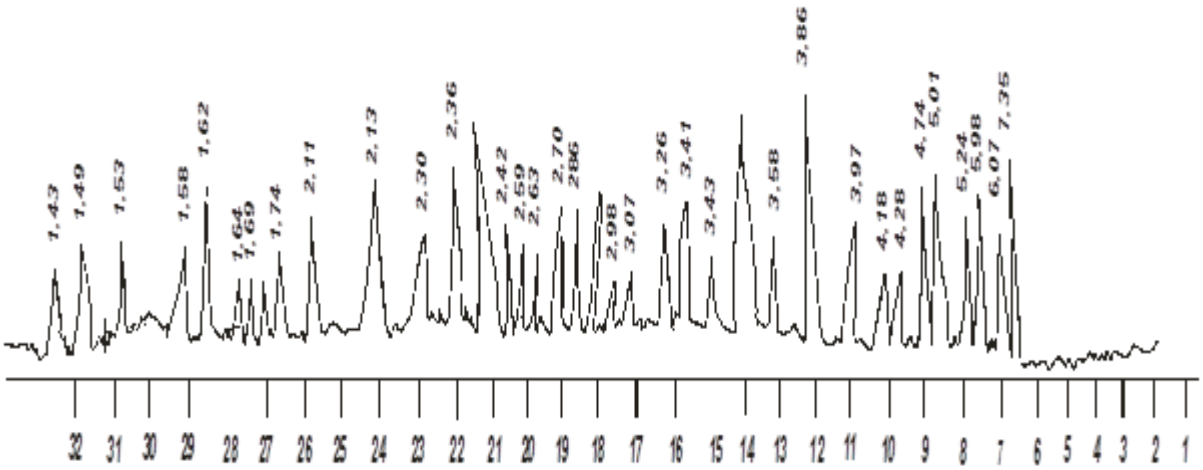

Figure 2. X-ray diffraction pattern of the compound $NH(C_2H_4OH)_2 \cdot (CH_3COO)_2Cu \cdot H_2O$.

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Thermal analysis showed that the heating curve of the obtained compound exhibits two endothermic effects at 109 and 170 °C and five exothermic effects at 268, 360, 490, 565, and 653 °C. The first endothermic effect is not accompanied by mass loss and is associated with the melting of the salt, whereas the second endothermic effect at 170 °C corresponds to the decomposition of the compound with a mass loss of 2.0%. The subsequent exothermic effects are related to the continuation of the decomposition process and the combustion of the thermolysis products (Figure 3).

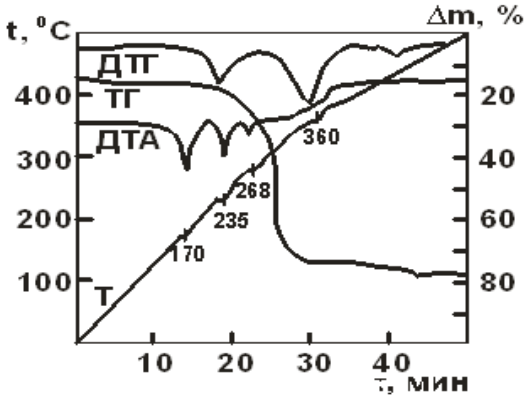

Figure 3. Derivatogram of the complex salt $NH(C_2H_4OH)_2 \cdot (CH_3COO)_2Cu \cdot H_2O$.

When studying the $N(C_2H_4OH)_3$–$(CH_3COO)_2Cu$–H_2O system, it was found that the refractive index, density, viscosity, and pH isotherms are characterized by two inflection points corresponding to 30.0 and 70.0 mol% [12]. This indicates the formation of a new compound at a triethanolamine : copper(II) acetate monohydrate molar ratio of 1:1 (Figure 9).

A solid complex compound of copper(II) acetate monohydrate with triethanolamine, $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$, was isolated.

Table 2. Elemental composition of the complex $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$

Component	Found, wt.%	Calculated for 1:1 composition, wt.%
$N(C_2H_4OH)_3$	42.51	42.78
$(CH_3COO)_2Cu$	52.21	52.08
H_2O	5.29	5.17

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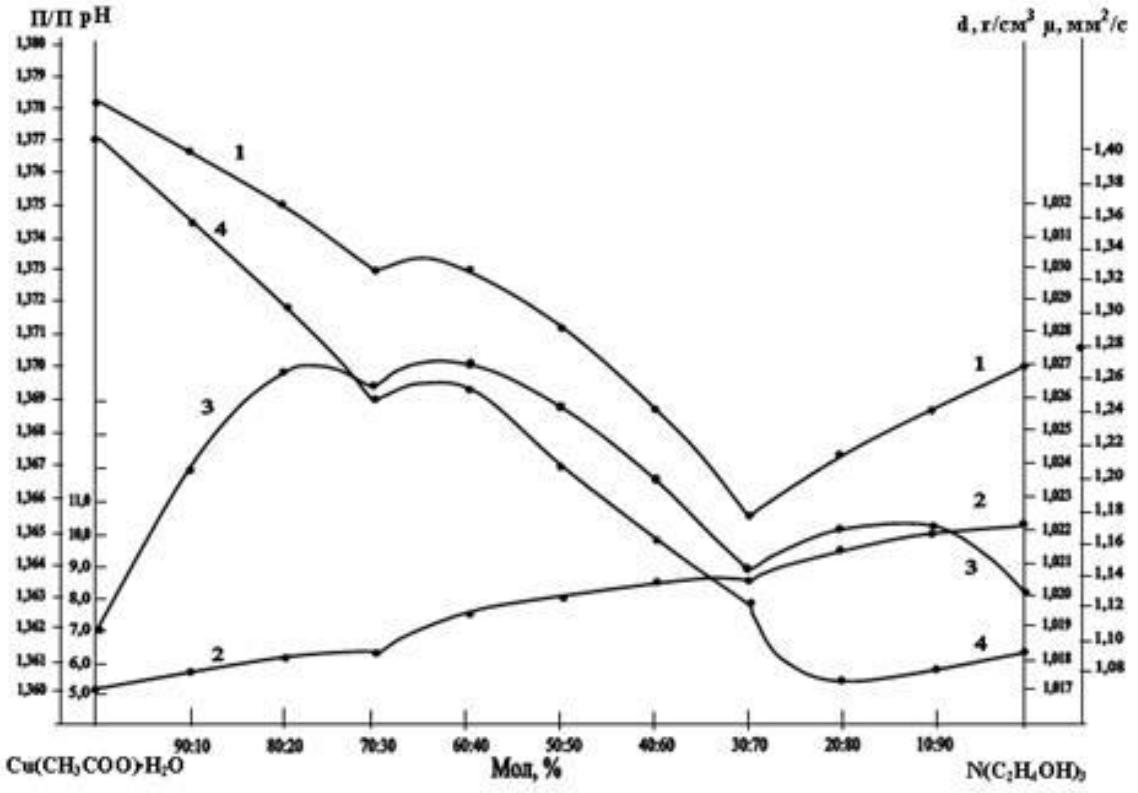

Figure 4. **Copper(II) acetate monohydrate–triethanolamine–water system.**

The solubility of the synthesized complex compound $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$ in various solvents was investigated. The compound is readily soluble in alcohols, poorly soluble in ethers and acetone, insoluble in benzene and toluene, and soluble in water with solubilities of 5.0 and 10.0 wt.% at 0.9 and 2.1 °C, respectively. The dissolution process was found to be congruent.

A comparison of the X-ray phase analyses of the initial substances showed that the obtained compound is characterized by new values of interplanar spacings, confirming the formation of an individual compound (Figure 5).

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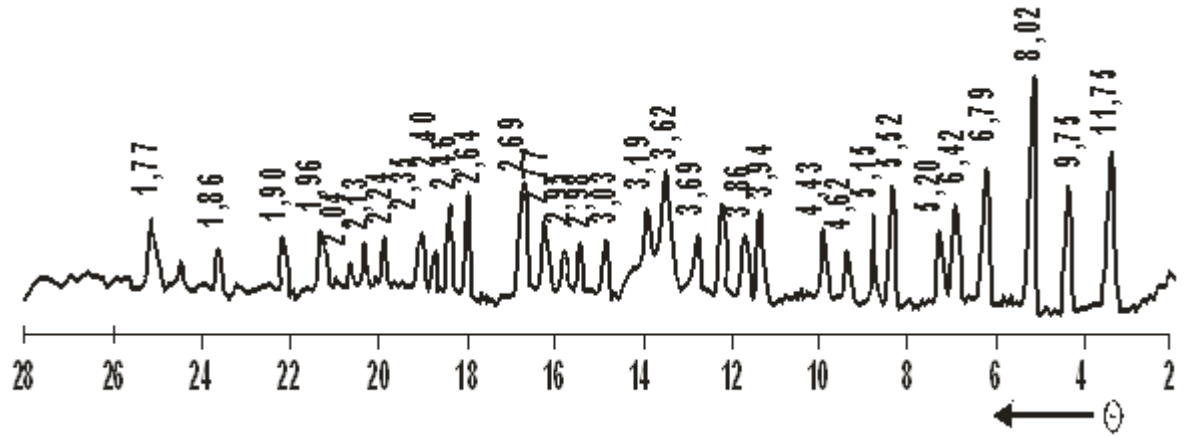

Figure 5. **X-ray diffraction pattern of the compound $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$.**

Based on thermal analysis, the obtained thermogram shows that the heating curve of the compound $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$ exhibits five endothermic effects at 92, 161, 272, 340, and 394 °C, as well as three exothermic effects at 462.5, 526, and 610 °C. The first endothermic effect is accompanied by a mass loss of up to 5.6% and is associated with the onset of dehydration of triethanolamine in the copper(II) acetate monohydrate salt. The second endothermic effect at 161.5 °C corresponds to the melting of the compound, with a mass loss of 10.6%. The three observed exothermic effects are related to the continuation of the decomposition process and the combustion of thermolysis products (Figure 6).

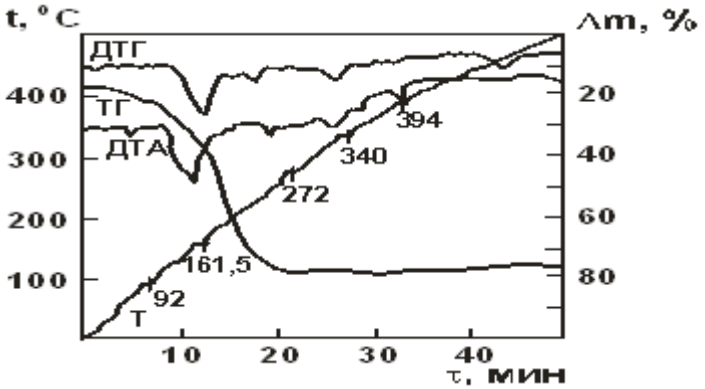

Figure 6. **Derivatogram of the 1:1 complex salt $N(C_2H_4OH)_3 \cdot (CH_3COO)_2Cu \cdot H_2O$.**

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A comparative analysis of the IR spectra of the complex and the initial substances—copper(II) acetate monohydrate and triethanolamine—shows that the formation of a complex between copper(II) acetate monohydrate and triethanolamine leads to significant changes in the IR spectrum in the region characteristic of the synthesized complex (Figure 8).

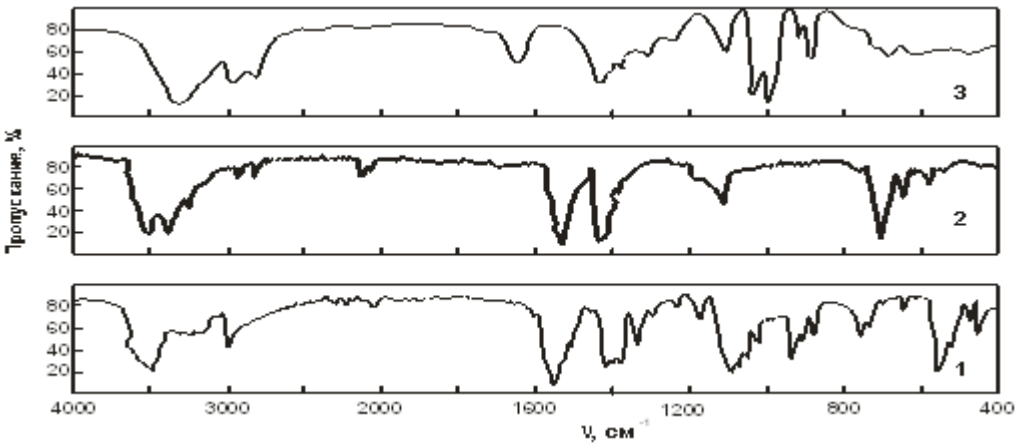

Figure 7. IR spectra: 1 – copper(II) acetate monohydrate with triethanolamine, 2 – copper(II) acetate monohydrate, 3 – triethanolamine.

As expected, the IR spectrum of the copper(II) acetate monohydrate complex differs significantly from those of the initial components when mono- and diethanolamines are used (Figures 4 and 8). Due to the absence of an NH group in triethanolamine, the main changes are observed in the region of valence bond vibrations at around 3600 cm^{-1}. In this region, a narrow and intense band at 3400 cm^{-1} is observed, which is characteristic of intramolecular hydrogen bonding involving the OH group of triethanolamine. This is reflected in a shift of the C=O band toward the lower-frequency region by approximately 20 cm^{-1} and is clearly detected at 1580 cm^{-1}.

Conclusion

Thus, to substantiate the synthesis of a plant growth stimulant with fungicidal properties, the conditions and mechanism of interaction between ethanolamines and copper(II) acetate monohydrate were investigated and established using the

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isomolar series method. The formation of three new compounds was confirmed using physicochemical analysis methods, including X-ray phase analysis, solubility studies in various solvents, thermal analysis, and IR spectroscopy. Based on the physicochemical analysis results, it can be concluded that ethanolamines bind to $(CH_3COO)_2Cu$ through the formation of intermolecular hydrogen bonds. The obtained preparations were tested as plant growth regulators and fungicidal microelement formulations.

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