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Extraction Of Complex Salts Of Microelement (Cu, Zn) And Microelemental Secondary Raw Materials

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***Annotation**:* The study solves the problem of developing a rational technology for the production of complex microelement fertilizers with high absorption form for plants on the basis of microelement salts (Cu, Zn, Mo) salts currently produced in the Republic, secondary microelement raw materials formed by molybdenum processing of JSC "Almalyk MMC". The article presents the results of research on complex methods of microelement salts produced at the Almalyk Mining and Metallurgical Combine and microelement secondary raw materials formed in the production process in the presence of monoethanolamine or citric acid salts, their production of highly microelement complex compounds. As well as the results of IR spectral analysis of the obtained complex compounds. The resulting complex form of microelement salts and industrial microelement secondary raw materials were found to have a high level of plant absorption when added to macro-fertilizers.

Experiments show that the effectiveness of mineral salts of microelements is lower than that of their chelate compounds. Numerous experiments have shown that microelement complexes (chelates) can increase the yield of agricultural crops even when used 2-10 times less than the amount of microelement salts.

Currently, the main chelating agents are HEDP (hydroxyethy lenendy phosphonic acid), ATMP (nitriltrimethyl phosphonic acid), DTPA (diethylenetriaminepentaacetic acid), EDTA (from Ethylenediaminetetraacetic acid), HMDT (hydroxy methylene diamine tetraacetic acid), DTMP (diethylenetriamine pentasiric acid). Microelement chelates are easier to assimilate by plants than inorganic salts (sulfates, carbonates, etc.), lignin-based chelates are 4 times easier to digest, and citrate-based chelates are 6-8 times easier to assimilate. Plants assimilate them much better and more efficiently because simple salts of microelements react with each other in the soil and form compounds that are difficult to assimilate. Chelates do not undergo such a reaction and also do not combine with soil. As a result, 30-40% of simple microelements are assimilated by plants, while 90% and more are assimilated by chelate microelements.

When nitrogen, phosphorus, and potassium, which are macroelements, are replenished by adding mineral fertilizers to plants, it is not possible to meet the demand for plants due to the lack of microelement production. Despite the great importance of microelement in the life ofplants, microelement are almost not produced in the country.

The following are the reasons for delaying the production of microelement fertilizers. Cheap and usable copper, zinc, cobalt, nickel, molybdenum, manganese, barium and other

microelement raw materials are not fully studied, there is a lack of annual data on the mobile forms of microelement in the soil, insufficient scientific and technological developments, recommendations and complex insufficient data on the status of micro elements in the production of fertilizers.
The use of various wastes and products of non-ferrous metallurgy, sour effluents, used catalysts and other types of secondary products in the production of mineral fertilizers is also an important problem.
Based on these data, it can be said that microelement (Cu, Zn) salts produced in the Republic, the acquisition of complexes of microelement secondary raw materials formed during the processing of molybdenum by JSC "Almalyk MMC", the development of rational technology for microelement fertilizers with high absorbency.
Keywords: Microelement salts, microelement secondary raw materials of industry, citric acid, monoethanolamine, reaction, micronutrients in a complex form with a high form that the plant can assimilate, the results of IR spectral analysis.

I. INTRODUCTION

Complex fertilizers containing microelements (Cu Zn Mo and others) play an important role in increasing plant productivity and improving their quality. They serve as a key determinant in the rapid development of plants, in the process of photosynthesis, in the synthesis of carbohydrates, proteins, vitamins, in the regulation of growth, in the assimilation of various nutrients and water. Due to the lack of microelements in the soil, high efficiency cannot be achieved even with the use of high doses of nitrogen-phosphorus fertilizers and all agrochemical methods. When microelements are used in combination with mineral fertilizers, complete assimilation of nitrogen, phosphorus, potassium and other nutrients by plants can be achieved. Each microelement has its own function and can not be replaced by others.

At present, all greenhouses and intensive orchards in the country use only imported microelement fertilizers. This is because the technology of obtaining cheap and available local microelement fertilizers has not been introduced in the country.

Based on these problems, it is necessary to develop a rational technology for the production of copper, zinc, molybdenum and other microelement fertilizers on the basis of secondary raw materials and microelements of the industry, which contain microelements.

Scientific research is being organized in the country to create and develop technologies for obtaining fertilizers containing nitrogen, phosphorus, potassium and other nutrients on the basis of local raw materials. Extensive theoretical and practical results are being achieved in the development of cost-effective methods of their application in agriculture. The third direction of the Action Strategy of the Republic of Uzbekistan for 2017-2021 identifies important tasks such as "... production of high value-added finished products based on deep processing of high-tech industries, especially local raw materials ..." [1]. In this regard, it is important to create and improve the technology of production of new varieties of microelement fertilizers, including on the basis of local microelement salts and industrial microelement secondary raw materials.

II. MATERIALS AND METHODS

Scientific and technical literature provides extensive information on the technology of production, chemical composition and properties, application, storage and transportation of microelement fertilizers.

Eighty percent of the elements in the Mendeleev periodic table are microelements, which do not exceed 0.01% in the earth's crust, plants and animals (in terms of dry matter). Some heavy metals (mercury, lead) are harmful to plants and living organisms, while the remaining elements have a positive effect on their development. Mineral fertilizers, the main nutrient of which is a microelement, are called microelements. In the following literature [2, 3] it is stated that for the adequate development of organisms, living organisms assimilate microelements from food, and plants from soil. Microorganisms enter the soil through soil-forming rocks, natural waters, and space dust. In addition, microelements are given artificially.

In A.I.Fateev's work, it is stated that a certain amount of microelements is released from the soil as a result of assimilation by plants or for other reasons, which are not replenished at present, which is due to non-introduction of organic fertilizers into the soil [4, 5]. It should be noted that the presence of microelements and their assimilation by plants synthesize all of the enzymes that allow them to effectively use soil energy, water, fertilizers and nutrients in the soil.

I.B.Karmazin, B.A.Yagodin, S.P.Polyanchikov emphasize that the presence of nitrogen, phosphorus and potassium alone is not enough for the normal functioning of the plant organism. Microelements play an important role in plant nutrition. They are involved in the synthesis of proteins, carbohydrates, vitamins. Under their influence, photosynthesis is improved, drought resistance is increased, immunity against disease pathogens is increased, resulting in increased productivity and improved quality [6, 7, 8].

Experiments show that the effectiveness of mineral salts of microelements is lower than that of their chelate compounds. Numerous experiments have shown that microelement complexes (chelates) can increase the yield of agricultural crops even when used 2-10 times less than the amount of microelement salts.

Currently, the main chelating agents are HEDP (hydroxyethy lenendy phosphonic acid), ATMP (nitriltrimethyl phosphonic acid), DTPA (diethylenetriaminepentaacetic acid), EDTA (from Ethylenediaminetetraacetic acid), HMDT (hydroxy methylene diamine tetraacetic acid), DTMP (diethylenetriamine pentasiric acid). Microelement chelates are easier to assimilate by plants than inorganic salts (sulfates, carbonates, etc.), lignin-based chelates are 4 times easier to digest, and citrate-based chelates are 6-8 times easier to assimilate. Plants assimilate them much better and more efficiently because simple salts of microelements react with each other in the soil and form compounds that are difficult to assimilate. Chelates do not undergo such a reaction and also do not combine with soil. As a result, 30-40% of simple microelements are assimilated by plants, while 90% and more are assimilated by chelate microelements [9].

Microelement complexes are more easily assimilated by plants than inorganic salts (sulfates, carbonates etc.).

In recent years, the importance of metalloorganic complex compounds in biochemical processes has begun to be determined. Recently, as a result of the discovery of the use of internal complex compounds called anti-chlorosis chelates and complexes, which occur due to lack of iron and other microelements in the soil, great attention has been paid to metalloorganic complexes.

A.A. Jacobson [10] attached great importance to the use of chelates or complexes in the prevention of chlorosis in plants. He attached great importance to EDTA (the disodium salt of ethylenediamine tetraacetic acid). This compound forms complexes with almost all complexing metals. Such complexes have good solubility in any environmental soils. Currently, methods have been developed for the preparation of synthetic chelates such as CuEDTA, MoEDTA, MnEDTA, ZnEDTA and their application in practice.

In the researches of I.A.Gaysin, R.N.Sagitova, R.R.Khabibullin the complex forms of different proportions of copper, molybdenum, as well as copper and molybdenum microelements in the presence of monoethanolamine were obtained and their positive effect on crop yields and plant development was studied [11]. About 15 copyright certificates and patents for inventions were obtained on the basis of these preparations. At present, in the Republic of Tatarstan, microelements in complex form are used on a large scale. Microelements in this complex form allow the rapid growth of plants, increase productivity, reduce the application of pesticides to plants by 3-4 times.

In the scientific work of I.A.Gaysin and others [12] it was shown that the solution obtained by adding boric acid and copper sulfate to an aqueous solution of monoethanolamine to stimulate plant growth is more effective than the use of individual ingredients when applied to plants.

Globally A.I.Fateev, A.S.Zarishnyak, B.A.Yagodin, S.P.Torshin, S.P.Polyanchikov, G.Gospadarenko, I.V.Jerdetskiy, E.A.Krylov, I. A number of scientists, such as A. Gaysin, have conducted research on the chemical composition and brand properties of microelement fertilizers, as well as the development of technologies for improving their quality.

In this regard, a large scientific school on the technology of microelement fertilizers under the leadership of Academician M.N. Nabiev has been established in Uzbekistan and is still operating. M.R.Adilova, E.K.Badalova, A.M.Amirova, M.T.Saibova, T.X.Taksanova, V.K.Khakimova, S.To'xtaev, S.M.Tadjiev, I.I. Usmanov, Z. Turaev and others are conducting large-scale research on the technology of microelement fertilizers, the scientific development of this field. Scientific sources on the production of microelement mineral fertilizers with high absorption properties by the above-mentioned scientists have not yet been studied.

When nitrogen, phosphorus, and potassium, which are macroelements, are replenished by adding mineral fertilizers to plants, it is not possible to meet the demand for plants due to the lack of microelement production. Despite the great importance of microelement in the life of plants, microelement are almost not produced in the country.

The following are the reasons for delaying the production of microelement fertilizers. Cheap and usable copper, zinc, cobalt, nickel, molybdenum, manganese, barium and other microelement raw materials are not fully studied, there is a lack of annual data on the mobile forms of microelement in the soil, insufficient scientific and technological developments, recommendations and complex insufficient data on the status of micro elements in the production of fertilizers.

The use of various wastes and products of non-ferrous metallurgy, sour effluents, used catalysts and other types of secondary products in the production of mineral fertilizers is also an important problem.

Based on these data, it can be said that microelement (Cu, Zn) salts produced in the Republic, the acquisition of complexes of microelement-containing secondary raw materials (MECSRM) formed during the processing of molybdenum by JSC "Almalyk MMC", the development of rational technology for microelement fertilizers with high absorbency.

III. RESULT AND DISCUSSION.

The object of research was the use of salts of Cu, Zn, State Standart 19347-2014 copper sulfate salt ($CuSO_4 \cdot 5H_2O$), State Standart 8723-82 zinc sulfate salt ($ZnSO_4 \cdot 7H_2O$) currently produced in our country.

Table 1 Description of microelement salts

Components	Copper sulfate	Zinc sulfate
Molecular mass. g / mol	249,68	161,47
Boiling point °C	650	740
Melting point °C	110	100
Density g / cm^3	2,29	3,54

Also, secondary raw materials (MECSRM) of JSC "Almalyk MMC" formed in the process of molybdenum processing, type 1 pH acidic environment and sample 2 pH environment neutral microelements were used.

Table 2 Chemical composition of MECSRM samples of hydrometallurgy, %

sample №	N_{total}	Microelemnts									H_2O
		total	Fe	Co	Mn	Cu	Zn	Mo	Ni	Mg	
1	4,87	0,35	0,0371	0,00012	0,0086	0,24	0,012	0,0371	0,00086	-	87,61
2	3,80	0,13	0,0012	-	0,0002	0,08	0,003	0,006	-	0,014	87,96

technical conditions (TC) 2423-002-78722668-2010 monoethanolamine and State Standart 32113-2013 - citric acid were used for complex transformation of salts of microelement (Cu, Zn) and MECSRM of hydrometallurgy.

Monoethanolamine: chemical formula C_2H_2NO, molecular weight 61.08 g / mol, density 1.012 g / cm3, viscosity 0.019Pa • s, melting point 10.3°C, boiling point 170°C, soluble in water.

Citric acid: chemical formula $Ca_3(C_6H_5O_7)_2$, white colorless crystalline substance, soluble in water and alcohol, molecular weight 192.12 g / mol, density 1.63 g / cm3, liquidus temperature 153.5°C.

Nitrogen content of raw materials, intermediates and finished products, various forms of phosphorus, calcium, magnesium, sulfur, aluminum, iron fluoride, carbonates, insoluble residues, water and others were analyzed.

Total nitrogen was determined according to the method given in the literature [13, 14]. This method is based on the reduction of nitrate nitrogen to ammonia nitrogen using a Devard alloy and the subsequent extraction of ammonia and its titrometric determination. Ammonia nitrogen was determined according to the method given in the literature [15]. This method is based on the oxidation of ammonia nitrogen to chloramine in the presence of a brominated potassium and a phosphate buffer solution with a pH of 6.7 to elemental nitrogen; The amount of excess chloramine is determined by the iodometric method.

Nitrate nitrogen [16] was determined according to the method given in the literature. The method is based on the reduction of nitrate nitrogen in an acidic medium in the presence of ammonium molybdate as a catalyst using a solution of ferrous sulfate (II) followed by titration of the excess amount of ferrous sulfate (II) with a solution of potassium permanganate.

The amount of water in the solid samples was determined by drying to constant weight in a drying cabinet at a temperature of 100–105°C [17].

Detection of elements Su, Zn, Mo [18, 19] was carried out by spectrometry and photocolorimetry (photometer on the device KFK-3). The method of photocolorimetric analysis is based on the law of light (wavelength of light Cu=λ-580, Zn=λ-530, Mo=λ-460) absorption, which is based on measuring the optical density of a solution, ie the attenuation of light (intensity, decrease in brightness) when light passes through a colored solution.

The density of solutions and slurries was determined using a pycnometer PJ-2 [20] and their kinematic viscosity was determined using glass capillary viscometers VPJ-1 and VPJ-2 [21]. Their pH values were determined in the METTLER TOLEDO FE20 / EL20 pH meter quick guide device [22].

In our research, certain complex compounds of microelement raw materials were used to obtain microelement fertilizers. Monoethanolamine and citric acid were used to obtain microelement complexes. Below are the reactions of microelement salts with monoethanolamine and citric acid.

Reaction of copper and zinc sulfate salts with monoethanolamine.

$CuSO_4 \cdot 5H_2O + 2HO\text{-}CH_2\text{-}CH_2\text{-}NH_2 \rightarrow [Cu(\text{-}O\text{-}CH_2\text{-}CH_2\text{-}NH_2 \rightarrow)_2]+$

$SO^{2-}_4 + 5H_2O + 2H^+$ (1)

$ZnSO_4 \cdot 7H_2O + 2HO\text{-}CH_2\text{-}CH_2\text{-}NH_2 \rightarrow [Zn(\text{-}O\text{-}CH_2\text{-}CH_2\text{-}NH_2 \rightarrow)_2]+$

$+SO^{2-}_4 + 7H_2O + 2H^+$ (2)

Copper and zinc sulfate salts form citrate complexes in varying proportions with citric acid.

$CuSO_4 \cdot 5H_2O + HOOC\text{-}CH_2\text{-}C(OH)(COOH)\text{-}CH_2\text{-}COOH+$

$+ [\text{-}OOC\text{-}CH_2\text{-}C(OH)\ (COO\text{-})\text{-}CH_2\text{-}COOH] \cdot Cu + H_2SO_4 + 5H_2O$ (3)

$ZnSO_4 \cdot 7H_2O + HOOC\text{-}CH_2\text{-}C(OH)(COOH)\text{-}CH_2\text{-}COOH+$

$+[-OOC-CH_2-C(OH)(COO-)-CH_2-COOH]Zn+H_2SO_4+7H_2O$ (4)

In the process of conducting experimental work, it is advisable to carry out microelement salts with monoethanolamine in a ratio of 1: 2 mol. The reaction of copper sulfate and citric acid in the ratio of 0.65: 1 mol, and the reaction of zinc sulfate and citric acid in the ratio of 0.75: 1 mol was considered the optimal option.

The study of the physiological significance of microelements and their assimilation into plants, known from the literature, requires consideration of their ability to form complex compounds. They form a wide variety of complex compounds with many types of organic compounds. The physiological activity of microelements in combination with organic complexes is thousands and millions of times stronger than their ionic state. Therefore, their IR spectral analysis was studied, taking into account the formation of complex compounds of copper, zinc sulfate salts and industrial microelement secondary raw materials with citric acid. The following are the results of the IR spectral analysis of complex forms of microelement salts and microelement secondary raw materials with citric acid.

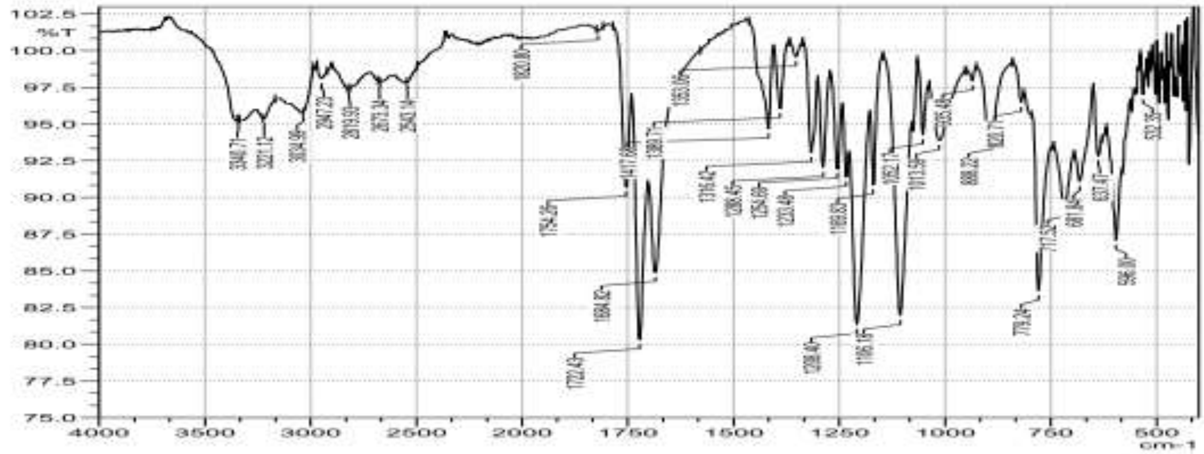

Figure 1. IR absorption spectrum of citric acid salt

The following frequencies were observed in the IR-absorption spectrum of the citric acid salt: 3341, 3221, 3035, 2947, 2820, 2673, 2543, 1821, 1754, 1722, 1685, 1418, 1390, 1353, 1316, 1288, 1254, 1233, 1208, 1170, 1106, 1052, 1014, 935, 888, 821, 779, 718, 682, 637, 596, 532 см$^{-1}$. The IR absorption of citric acid salt in the crystalline state has specific spectra: 3341-ν (OH), 1685 - ν(CO) cm^{-1}.

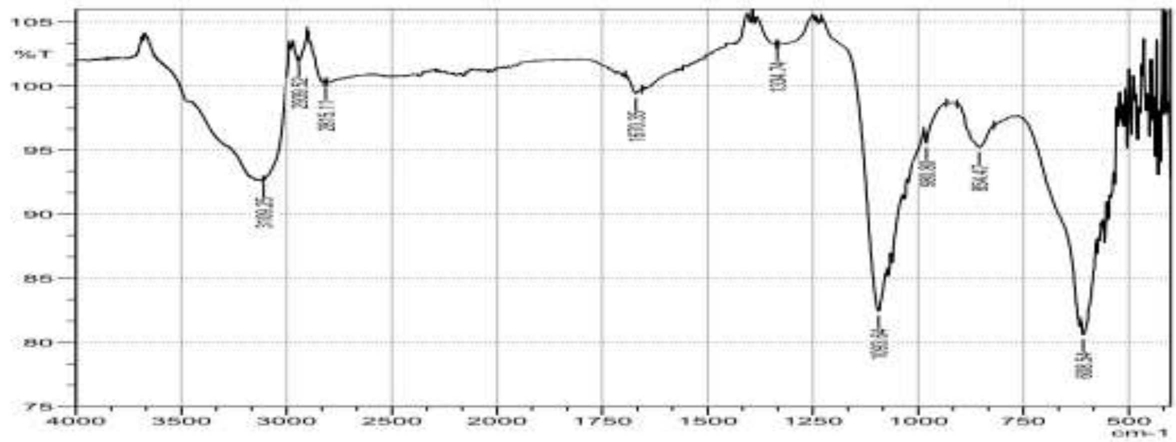

Figure 2. IR absorption spectrum of copper sulfate salt

Intensive absorption lines 1094 and 609 cm-1 in the IR spectrum belong to the sulfate ion. The following frequencies were observed in the IR-absorption spectrum of the copper sulfate salt: 3109, 2940, 2815, 1670, 1335, 1094, 981, 855, 609 cm^{-1}.

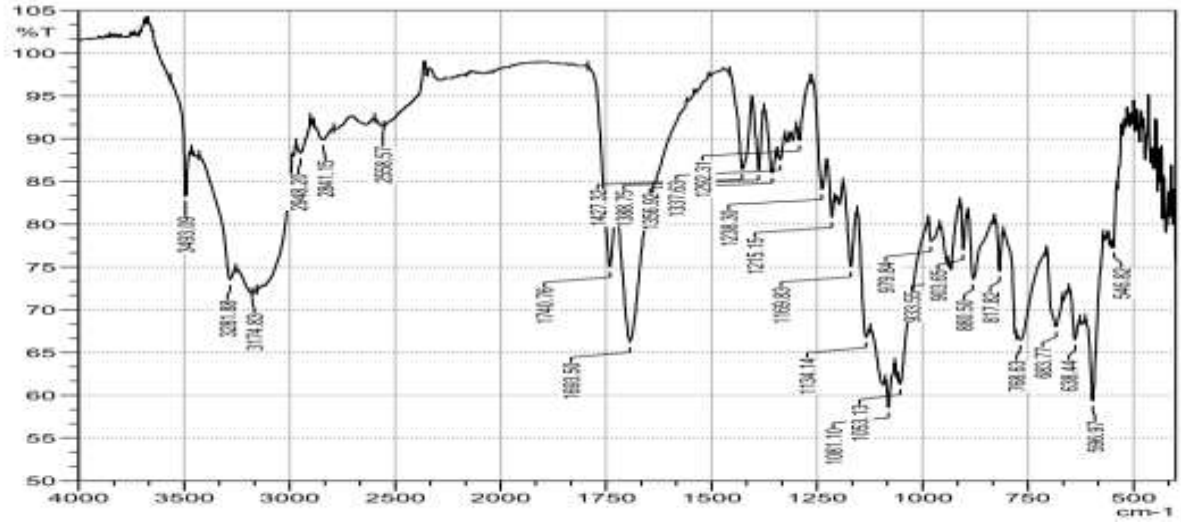

Figure 3. IR absorption spectrum of copper citrate complex

The following frequencies were detected in the IR-absorption spectrum of the copper citrate salt: 3493, 3282, 3175, 2948, 2841, 2559, 1741, 1693, 1427, 1388, 1356, 1338, 1292, 1238, 1215, 1170, 1134, 1081, 1053, 980, 934, 904, 881, 818, 769, 684, 638, 597 ва 547 cm^{-1}. Based on the IR-absorption spectrum, citric acid coordinates with the copper ion through oxygen instead of the hydrogen atom in the carboxyl group. It was found that the frequencies in the IR-absorption spectrum of the copper citrate salt differed from the IR-absorption frequencies of the original citric acid and copper sulfate compounds. it can be observed that new frequencies such as 3493, 3282, 3175, 1081 and 904 cm-1 are formed in the copper citrate salt.

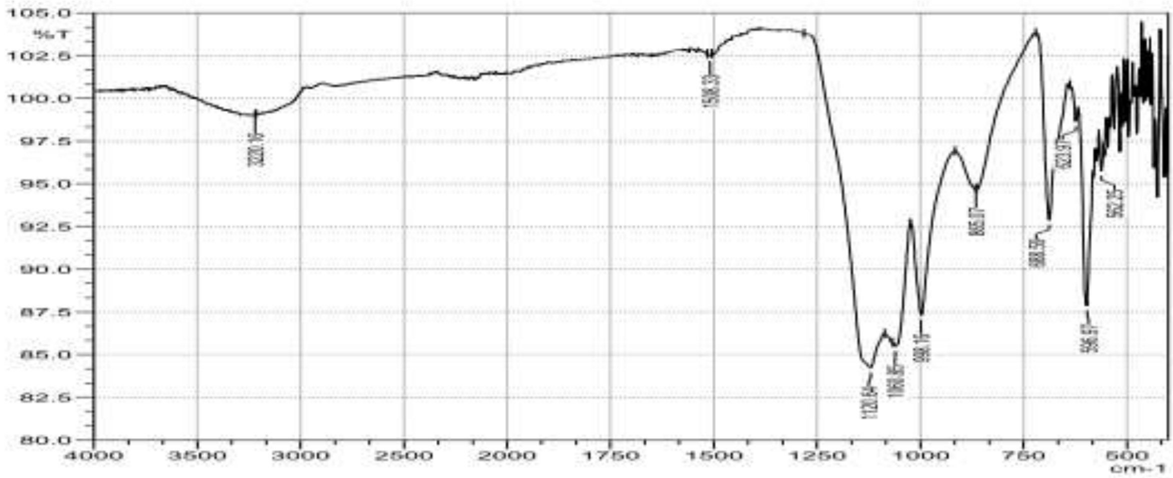

Figure 4. IR absorption spectrum of zinc sulfate salt

The following frequencies were detected in the IR absorption spectrum of zinc sulfate salt: 3220, 1508, 1121, 1061, 998, 865, 689, 624, 597, 562 cm^{-1}.

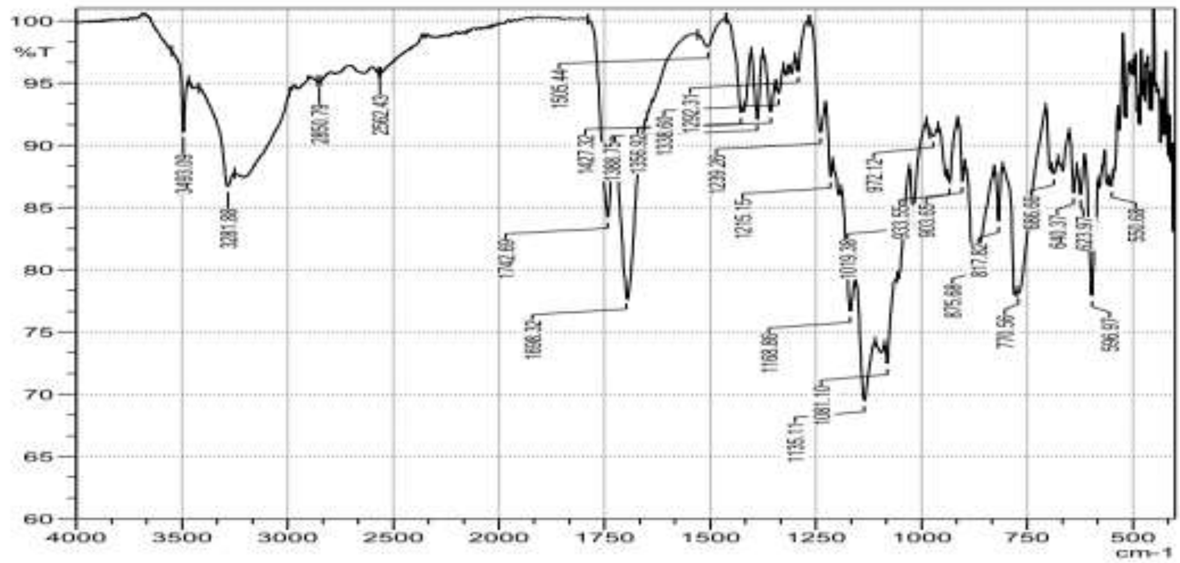

Figure 5. IR absorption spectrum of zinc citrate salt

The following frequencies were detected in the IR-absorption spectrum of zinc citrate salt: 3493, 3282, 2851, 2562, 1742, 1698, 1427, 1389, 1357, 1339, 1292, 1239, 1215, 1169, 1135, 1081, 1019, 972, 933, 904, 876, 817, 771, 687, 640, 624, 597 ва 551cm^{-1}. According to the IR-absorption spectrum, citric acid coordinates with the zinc ion through oxygen instead of the hydrogen atom in the carboxyl group. It was found that the frequencies in the IR-absorption spectra of the complexes varied relative to the IR-absorption frequencies of the original citric acid and zinc sulfate compounds. It can be observed that new frequencies 3493, 3282, 1339, 972 and 904 cm^{-1} are formed in the zinc citrate salt.

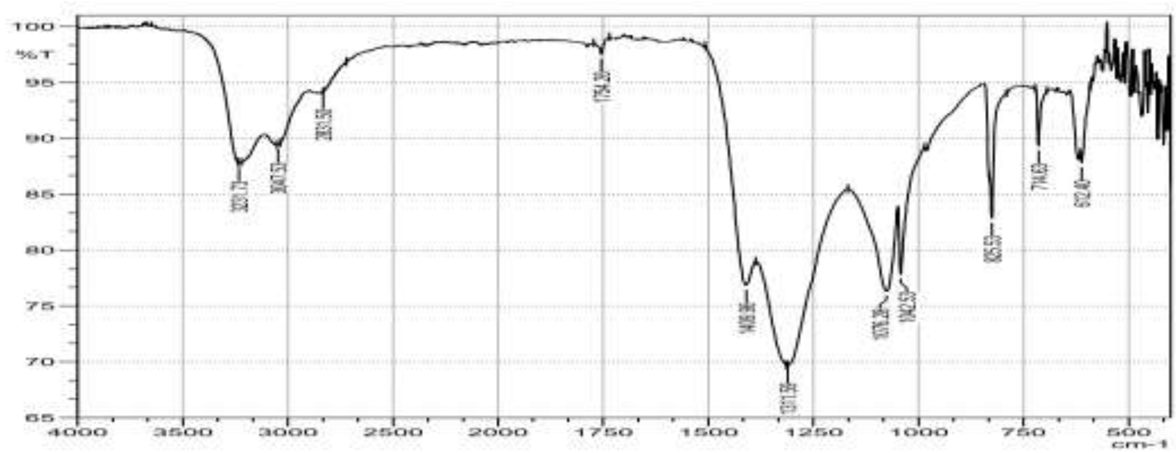

Figure 6. IE-absorption spectrum of MECSRM

The following frequencies were detected in the IR-absorption spectrum of MECSRM: 3232, 3048, 2832, 1754, 1410, 1312, 1076, 1043, 826, 715 ва 612 cm^{-1}.

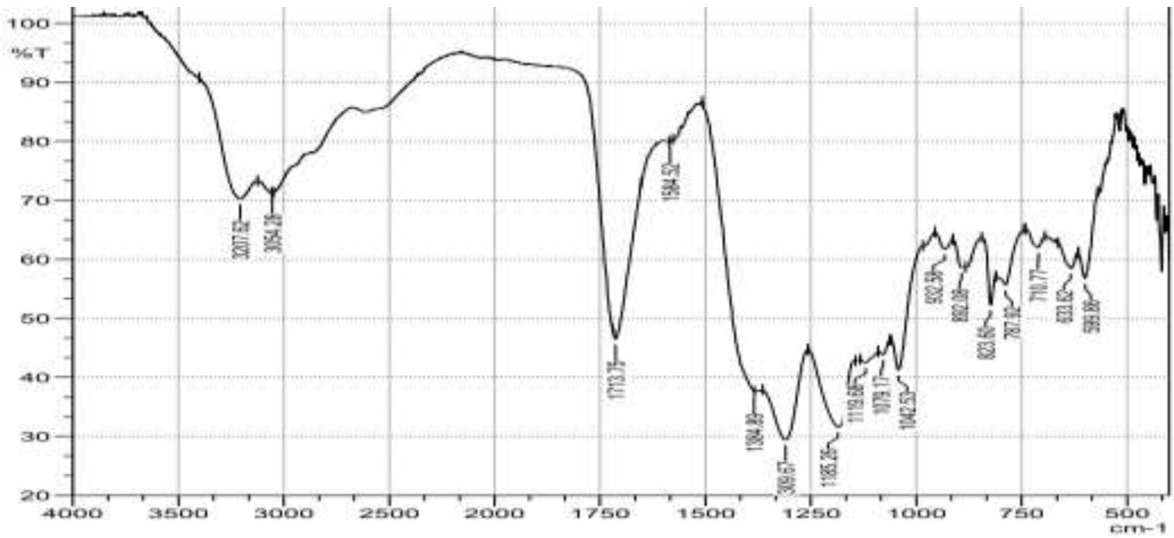

Figure 7. IR-absorption spectrum of MECSRM in complex form

The following frequencies were observed in the IR-absorption spectrum of the complex form MECSRM: 3208, 3054, 1714, 1585, 1385, 1310, 1185, 1120, 1079, 1042, 933, 892, 824, 788, 711, 634, ва 600 см$^{-1}$. Based on the IR-absorption spectrum, citric acid coordinates with the ions of copper and zinc and other salts in the MECSRM via oxygen instead of the hydrogen atom in the carboxyl group.

The results of IR spectral analysis revealed that microelement salts and secondary raw materials combine with citric acid to form new bonds. The microelement salts in the form of these obtained citrates can be applied to plants in agriculture. It is also advisable to apply complex form microelements to plants with the addition of macroelements in order to reduce the overuse of labor and equipment in the field.

IV. CONCLUSION

Complex compounds with monoethanolamine and citric acid salts were obtained in order to increase the ability of microelements to be assimilated by plants. Microelementt salts were carried out with monoethanolamine in a ratio of 1: 2 mol, copper sulfate and citric acid in a ratio of 0.65: 1 mol, and zinc sulfate and citric acid in a ratio of 0.75: 1 mol. The results of IR spectral analysis of microelement salts and microelement secondary raw materials converted into complex forms were also studied. The results showed that microelement salts and secondary raw materials combined with citric acid to form new bonds. It can be said that it is necessary to obtain complexes of microelement salts (Su, Zn, Mo) and on their basis to develop technologies of microelement fertilizers and their application in agriculture.

When microelementts are applied directly to plants, it is found that their level of plant absorption is low, ie 35-45%, and the water-soluble form is 15-20%. Complex plant microelementt salts and microelement secondary raw materials in plant-assimilated form increased by 85-90%. It has been proved that microelement salts and microelement secondary raw materials can be converted into complex forms in the presence of monoethanolamine or citric acid salts to obtain new varieties of microelement fertilizers.

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