

DETERMINATION OF THE LOWER LIMIT OF COPPER II ION IN ENVIRONMENTAL OBJECTS USING AN IMMOBILIZED REAGENT

Yulchieva Sevara Tojimatovna

Doctor of Philosophy in Chemical Sciences, Senior Lecturer at the Department of Medical Chemistry, Andijan Medical Institute mail: yulchiyevasevara779@gmail.com

ORCID iD: 0009-0006-0357-0908

Annotation. This paper describes the quantitative analysis of copper and the analysis of the results obtained using sorption spectroscopy methods. In addition, the quantitative detection limit was described in the analysis of the immobilization of the organic reagent in the SMA-1 fiber sorbent and its IR spectral results. Immobilization properties of indigo reagent with copper ions were shown. A method for the determination of copper ions in water has been proposed.

Keywords: immobilized reagent, organic reagent, sorption spectrophotometric methods of analysis, IR spectroscopy.

Introduction. The possibility of immobilization of the INDIGO reagent on fibrous sorbents has been investigated. The optimal conditions for the immobilization and complexation of copper (II) ions with the immobilized reagent have been determined. A sensitive layer of an optical sensor for the determination of copper (II) in various water samples has been developed.

As you know [1], industrial enterprises and transport intensively pollutes the environment with heavy and toxic metals (TM). Metallurgical enterprises annually eject to the surface of the earth a huge amount of various mutagens, carcinogens, fossil fuels. Almost all metals can be found in coal and oil ash in concentrations that are economically justified to extract elements from it. One of the widespread types of anthropogenic pollutants is the entry of TM into the soil [2]. The urgency and importance of the problem raised in the work is the intake of ecotoxins in the human body according to the scheme: soil - plant - man - environment [3].

Experimental part.

Reagents and equipment. Standard solution copper was prepared by dissolving the corresponding salt copper ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in bidistilled water, titer which was $T = 1.0 \text{ mg / ml}$ [4]. Organic reagent indigo was prepared by dissolving the required sample of the drug in bidistilled water, the concentration of which was equal to $1 \cdot 10^{-3} \text{ mol / l}$. Buffer solutions were prepared from the corresponding salts and acids of chemically pure grade. [4]. Metal salts and other reagents were of chemically pure grade. or ch.d. and were not subjected to additional purification, and their diluted solutions were prepared by diluting the initial ones with bidistillate before starting the experiment. The acidity and basicity of the solutions were adjusted with acetate-ammonia buffer solutions, The pH of solutions was measured on an I-130 ion meter and a pH meter pH / mV / TEMP Meter P25 Eco Met Korean made. The absorption spectra were measured on an SF-46, KFK-2 spectrophotometer, and the reflection spectrum on dual-beam recording spectrophotometer UV-Vis SPECORD M-40.

Fibrous material was used as a carrier. material synthesized by the method [5] Polyacrylonitrile sorbent (CMA-1) was used in the form of a disk with a diameter of 2 cm and a mass of 20-30 mg. It was washed with a mixture of 50 ml of 0.1 M HCl and 10 ml of acetone, then immersed for 10 minutes. in 10 ml $1 \cdot 10^{-3} \text{ M}$ solution organic reagent, after which the immobilized sorbent was stored in Petri dishes in a wet state. The content of the reagent in the sorbent was determined spectrophotometrically by the change optical density of the initial

reagent solution at 440 nm before and after immobilization. Characteristics of the immobilized reagent was determined in a static mode. The carriers were placed in a glass with the test solution and kept for a certain time with slight weak stirring. The analytical signal was change in diffuse reflectance

INDIGO discs with copper ions at 750 nm (Table 1).

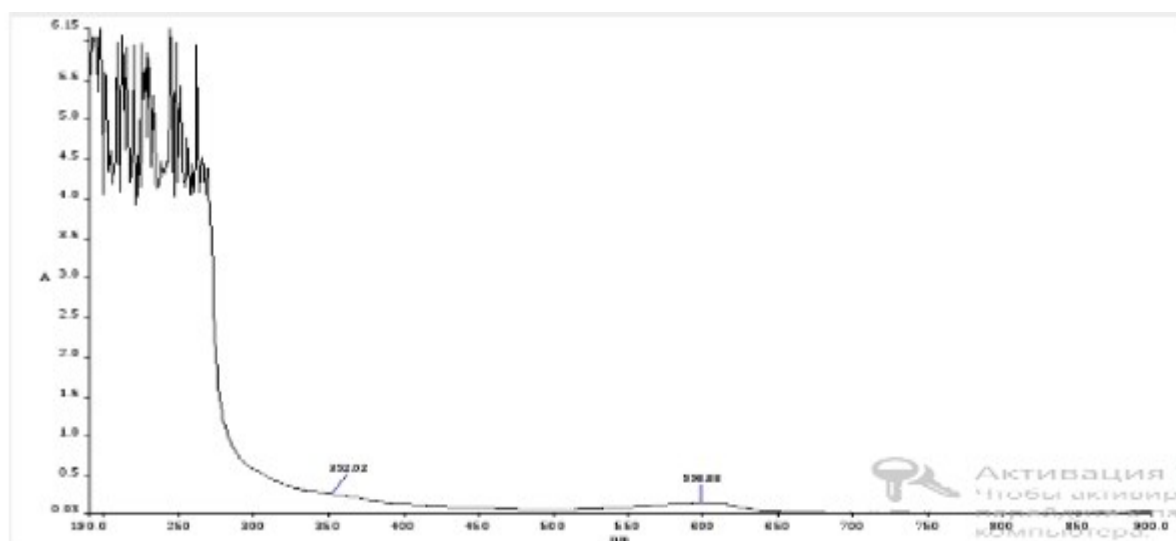
Table. 1

lR,nm	lMeR, nm	Me:R	pH	Immobilization time, min.	Concentration of reagent on carrier, M
590	750	1:1	8	10	1,0×10

We used two types of sorbent, initial (beige) and immobilized with reagents (colored). Color change due to complexation on the solid phase is detected both by an instrumental method and visually

Results and its discussion. We poured 800ml of distilled water on 100 g of indigo leaf and kept it in hot water at 70 -80 C for 7-10 minutes. Then the liquid part of the crushed leaves was separated. We kept the aqueous mass in the open air for 24 hours and a precipitate formed. We filtered and dried the sediment. We then gave him an IQ test. [6]

Figure 1. IR spectrum of indigo reagent.



From the results of IR spectra of indigo obtained under normal conditions - -C-C- ($400\text{--}900\text{ cm}^{-1}$), C-O-C ($900\text{--}1200\text{ cm}^{-1}$), C-N ($500\text{--}1400\text{ cm}^{-1}$), -C=O ($1500\text{--}1800\text{ cm}^{-1}$), ($2100\text{--}2300\text{ cm}^{-1}$), -OH ($3600\pm 50\text{ cm}^{-1}$)) functional groups SMA-1 - orange fiber, obtained by modifying hexamethylenediamine to PAN fiber .

Dependence of the analytical signal on the wavelength.

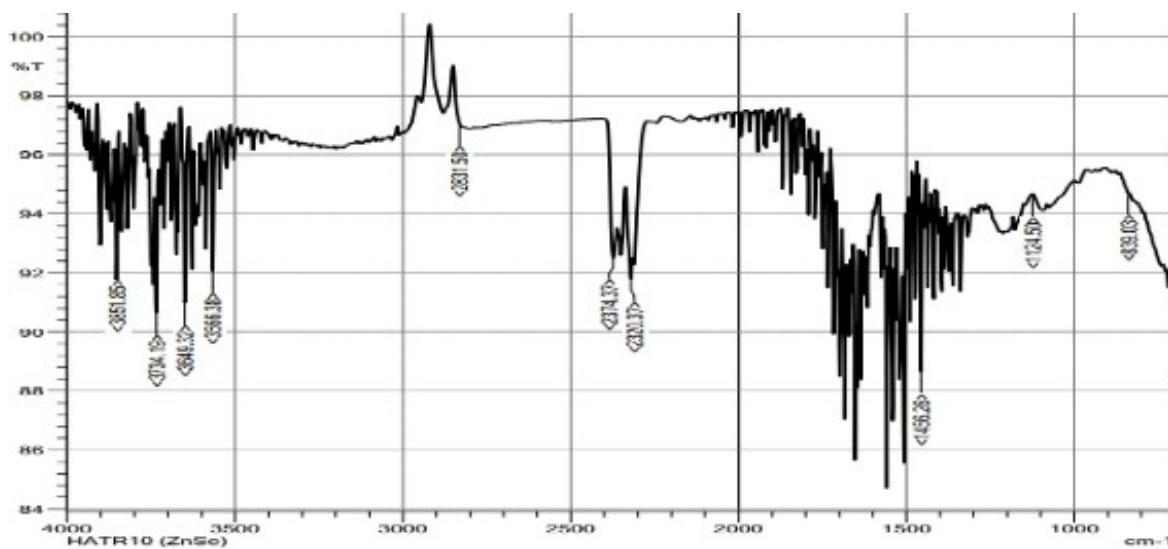
Table. 2

lR,nm	315	364	400	440	490	540	590	670	750	870	980
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A indigo	0,09	0,11	0,11	0,15	0,25	0,40	0,49	0,25	0,09	-	-
Acompl	0.05	0,06	0,07	0.08	0.10	0.14	0.18	0.25	0.29	0.17	-

It is known that a substance absorbs electromagnetic radiation of a certain wavelength, therefore, in order to study the conditions of interaction and determine the quantitative characteristics of the spectrophotometric determination of copper, it is first necessary to select the optimal conditions. To select the optimal wavelength of the complex with the reagent and the copper ion, the optical density of the solution and its complex was measured at different wavelengths. The data obtained are presented in Table 2

Figure 2. Select the maximum filter.



As can be seen from the table, the spectra of the and Fig 2 copper complex and the indigo reagent are opposite to each other (160 nm). According to the Sendal value, we can conclude that the copper detection method is very sensitive.

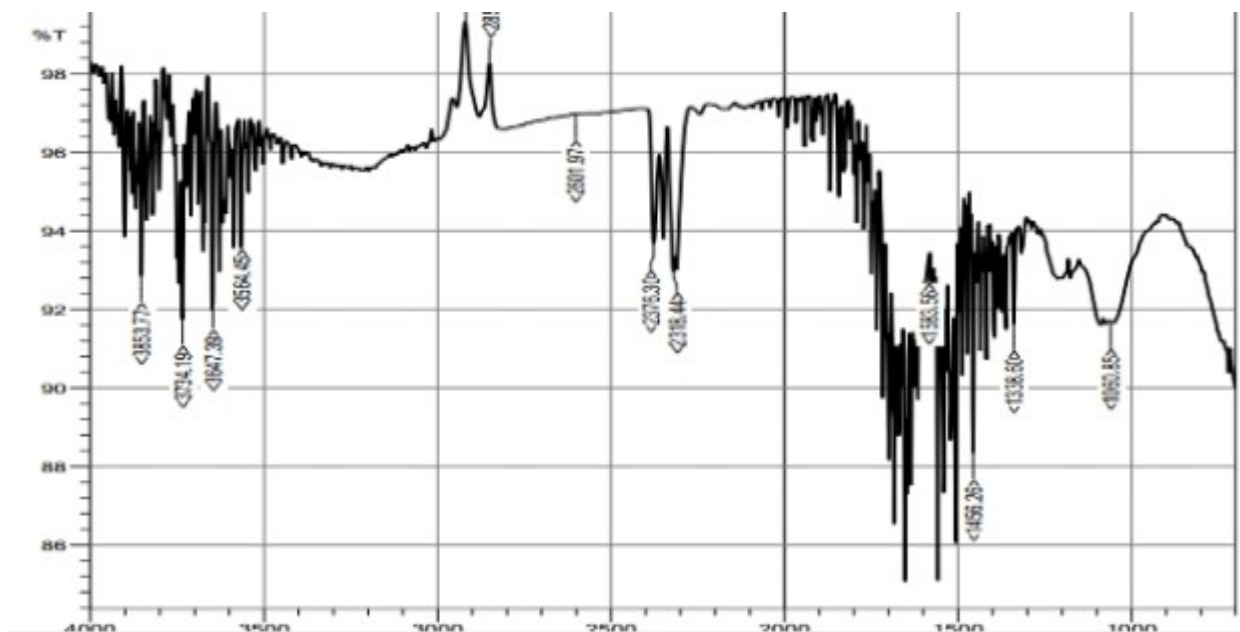
Spectrophotometric characterization of copper ions with selected reagents

Table .3

Complex color	pH	MeR	HR	$\Delta\lambda$	C_{Cu}^{+2} mkg	Ekaj	Mkg / cm ² according to the Sendal value
biue	8,0	750	590	160	30	30319	0,0021

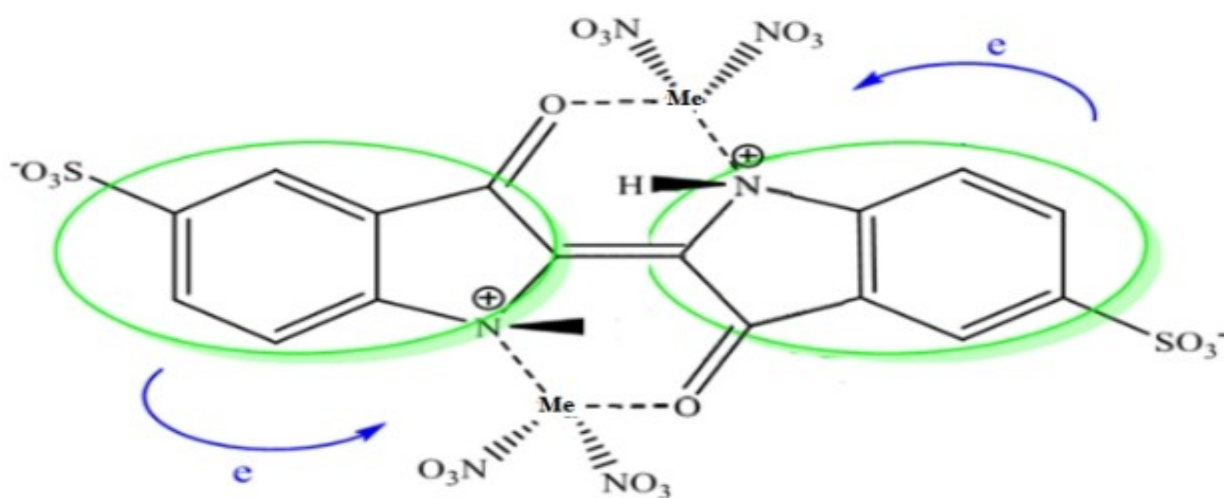
Comparing the analysis results obtained in solution and on immobilized sorbent, it was shown that the pH level of the complex during immobilization moves to a more acidic region and the detection limit decreases by one order of magnitude (Table 3).

Figure.3. Light absorption spectrum of indigo and its copper complex.



From the data in Figure 3 we can see that when comparing the IR spectra of the copper and R2 reagent complexes, the complex spectrum contains a band at 601 cm^{-1} , which is absent in the IR spectrum of the reagent, indicating that the O-Water bond is formed through functional active groups, according to [4].

Figure 4. Approximate structure of complex formation of an organic reagent immobilized with copper (II) ion.



The molar amount of indigo reagent and copper metal was determined using the method of isomoly series. Experimental results (Table 4 and Figure 4) show that the molar ratio of the composition of the copper complex with indigo ($\text{Me}:\text{R}$) = 2: 1. Determination of molar ratio of

copper complex with indigo reagent by Asmus method; The constant amount of copper is 30.0 mcg, 10 ml of universal buffer solution, variable reagent solution with pH = 8.0 is prepared in a flask with a volume of 25 ml and filled with distilled water. The solution was mixed and the light absorption was measured in KFK-2, 1 = 1.0 cm empty test solutions were used for comparison with the light filter. The results are presented in Table 5.

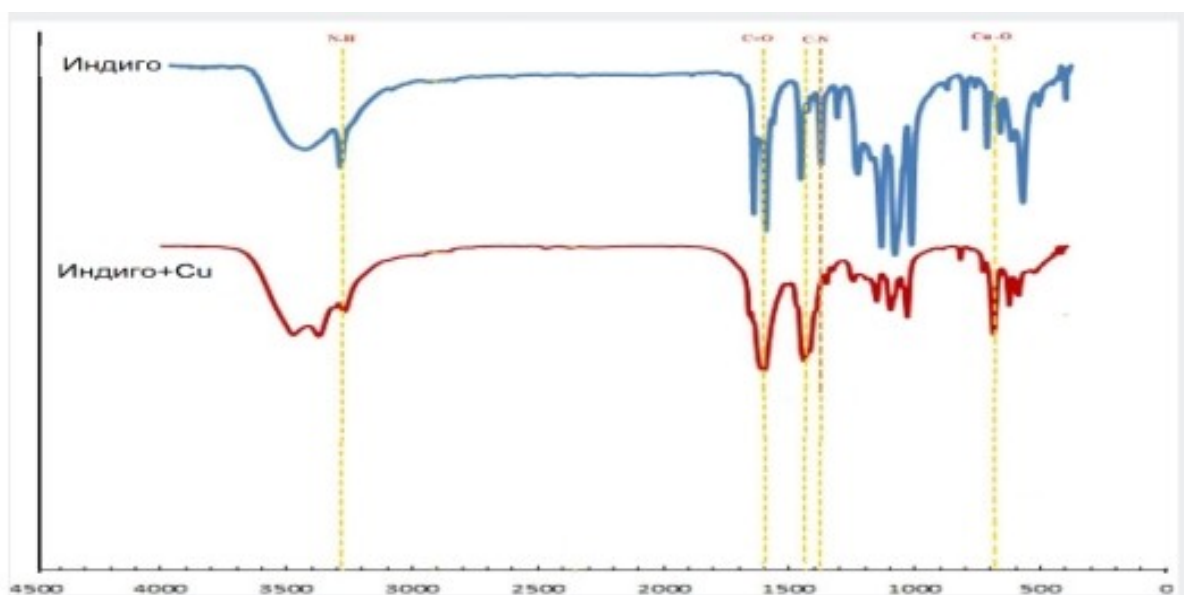
Optical plotnost at postoyannoy kontsentratsii rhenium.

Table .4

№	Reagent ml	$\bar{A}$	$\frac{1}{Am}$	$\frac{1}{V}$	$\frac{1}{\bar{V}}$	$\frac{1}{\bar{V}}$
1.	0,4	0,090	11,11	2,500	6,250	15,625
2.	0,8	0,130	7,69	1,250	1,562	1,953
3.	1,0	0,155	6,45	1,000	1,000	1,000
4.	1,4	0,180	5,55	0,714	0,510	0,364
5.	1,6	0,200	5,00	0,625	0,391	0,244
6.	1,8	0,210	4,76	0,555	0,308	0,171
7.	2,0	0,215	4,65	0,500	0,250	0,125

The results of Table 4 show that in this case the composition of the complex is 2: 1 in the molar ratio of Cu; R, the molar ratio of copper to the reagent obtained by two methods shows the same results. Study of the structure of the copper complex using indigo spectroscopy. The structure of the copper-derived complex using Indigo was proved by IR spectroscopy. IR spectroscopy was recorded on a Nicolet IR 200 (USA) spectrometer using Thermo Scientific spectrometer in the frequency range 500-4000 cm^{-1} using KBr.

Figure 5..IR spectra of indigo (1) and complex connection of medi with indigo .



The changes in the oscillations in Figure 5 and the emergence of new ranges can be seen, in particular, in the indigo spectra of 3700–3800 cm⁻¹ as well as clearly visible groups in the ranges of 1600 cm⁻¹ and 1400 cm⁻¹. The frequencies associated with the oscillations of the copper oxygen bonds increase in our 800cm⁻¹ red content spectrum, which means that the bonds in the group are strengthened and weakened by the remaining mass during the formation of the mixture. According to the study of IR spectroscopy and molar ratios of the metal, the reagent of the copper-colored complex compound with indigo reagent has a ratio (2: 1).

Accuracy and reproducibility of indigo reactive copper detection method. (n=3, p=0,95)

Table 5.

Added Cu ²⁺ , mkg	A	Found Cu ²⁺ , mkg	S	x
5,00	0,047	4,99	0,0692	0,172
10,00	0,088	10,08	0,1250	0,310
20,00	0,170	20,12	0,0692	0,172
30,00	0,249	29,81	0,1200	0,298
40,00	0,341	41,24	0,1880	0,467
50,00	0,411	49,79	0,1200	0,298
60,00	0,492	59,65	0,1250	0,310
70,00	0,575	70,01	0,1360	0,330

The data obtained confirm the accuracy and repeatability of the method developed for the detection of copper with a standard detection level not exceeding 0.188.

Conclusion. Indigo dye was isolated from local plant raw materials, physical and chemical properties of indigo dye were studied by IR spectroscopy and compared with the literature. The structure and composition of analytical active and functionally active groups of natural dye indigo were studied. Optimal conditions for obtaining the copper complex of indigo were determined. The structure of the copper complex was analyzed using indigo spectroscopy. In addition, the quantitative detection limit is SMA-1. analysis of the immobilization of indigo reagents in a fibrous sorbent and their IR spectral results. The immobilizing properties of indigo reagent for a particular fiber have been demonstrated. A method for the determination of copper ions in water has been proposed.

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