

Development of A New Sorption-Spectroscopic Method for The Determination of Fe (Iii) Ions Using Immobilized Methylthymol Blue

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Abstract: The origin of the problem. Iron, one of the most common metals in the environment, plays a key role in many biological and biogeochemical processes, which determines its presence in various oxidation states. The relevance of iron in environmental and industrial chemistry, human physiology, and many other fields has necessitated the development and optimization of precise analytical methods for determination. In the analytical determination of iron in real samples, spectrophotometric and absorption-spectroscopic methods are most frequently used.

Objective of the work: To develop fast, selective, and sensitive sorption-spectroscopic methods for the determination of iron ions using immobilized reagents.

Methodology. Sorption-spectrophotometric hybrid methods were applied. Optimal conditions for immobilization and complex formation were studied depending on time, environment, temperature, and other factors.

Scientific novelty. A sorption-spectrophotometric method has been developed for determining the micronumber of iron ions using immobilized methylthymol blue in the analysis of natural surface waters.

Results obtained. An accelerated method for sorption-spectroscopic determination of iron ions in natural wastewater has been developed using immobilized methylthymol blue and improved metrological indicators. Conditions for the immobilization, complexation, and detection of iron ions have been optimized. It has been established that the optimal buffer at pH 2.5 is a universal buffer solution. The developed method was used in the analysis of drinking water with a determination limit of 0.1 µg/l.

Keywords: Analytical reagent, iron ion Methylthymol blue, immobilization, absorption-spectroscopic.

INTRODUCTION

Iron is one of the trace elements essential for the human body. A deficiency of the element iron in the human body causes various vascular diseases [1]. However, it is worth noting that an excess of iron in the human body causes damage to the liver, pancreas, and myocardium - the development of hemochromatosis [2]. Iron plays an important role in the development of the ecosystem, agriculture, and industry, but it is also a pollutant of water bodies [3]. Heavy metals in drinking water pose a threat to human health. Therefore, it is necessary to determine and control the amount of iron ions in environmental objects.

A simple and fast solid-phase spectrophotometry method has been proposed for determining free iron (III) in environmental and biological samples [4]. The disodium salt of 1-nitroso-2-naphthol-3,6-disulfoacid (Nitroso R salt) and hexadecyltrimethylammonium bromide were used as ligands [5]. A flow-injection spectrophotometric method for determining the amount of dissolved and total iron in tap and natural waters has been developed. In natural water samples, the catalytic effect on the oxidation of N,N-dimethyl-p-phenyldiamine was determined [6]. The spectrophotometric method for determining iron ions using various ligands does not allow for wide

application due to its selectivity and low sensitivity. Iron (III) ion can be determined by atomic absorption spectrometry using immobilized sulfasalicylic acid [7], by immobilizing sulfarsazen ligand on polyacrylonitrile-modified polyethylene-polyamine fiber [8], and in dithizone ligands [9]. Inductively bound plasma mass spectrometry (ICP MS) was applied to determine iron and iodine elements in watermelon seeds [10], iron and many trace elements in the plant, leaves, and seeds [11], and 44 macro- and microelements in *Tanacetum vulgare* flowers [12].

METHODS

0.5585 g of high-purity metallic iron is dissolved in 10 ml of a mixture of hydrochloric and nitric acids (3:1), diluted with 50 ml of water, and boiled until nitrogen oxides and 5585 mg/ml of Fe^{+3} chlorides are removed, forming a standard solution of nitrates [13]. Buffer solutions [14] were prepared based on literature data. To prepare a 0.01 M solution of methylthymol blue, 0.76085 g of the reagent powder is measured on analytical scales and distilled water is added to a 100 ml flask.

RESULTS AND DISCUSSION

A sorbent for the immobilization of the organic reagent

was selected, and the procedure for adding components to the immobilization process was determined. The optimal conditions for the formation of a complex of the immobilized organic reagent Methylthymol blue (MTK) with Fe^{+3} have been established. In the research work, the time dependencies of complex formation, optimal pH values and optimal buffer solutions, the order of adding buffer solutions during complex compound formation, and other features were studied.

To select optimal conditions for the immobilization of organic reagents on fibrous sorbents, 0.2000 g of the analytical scale was taken from several sorbent types and activated with a standard 0.1 M hydrochloric acid solution for 24 hours, then washed with distilled water and stored in a special Petri dish (in a moist state). CMA1, PPD, PPF, and PPA1 fibers were selected for analysis; 0.2000 g of the selected fibrous sorbents were placed in a beaker, 10 ml of a 0.1% solution of our reagent, methylthymol blue, was added, and stirred for 5-10 minutes. The reflection spectrum of immobilized fibers was measured using the X-rite one (China) spectrophotometer and calculated using the Kubelke-Munk function.

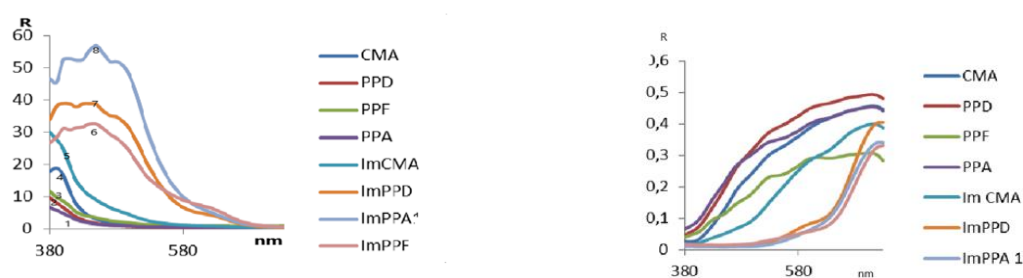


Figure 1. Reflection spectrum of fibers immobilized with methylthymol blue reagent.

From the results obtained, it can be concluded that the complex connection is formed in 1 minute, has a maximum optical density in 7 minutes, and forms a

complete complex. Since the complex compound is stable enough, it can be easily used in analytical processes.

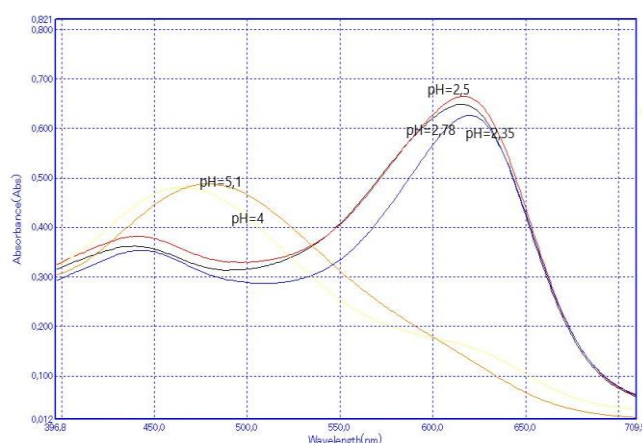


Fig. 2. Dependence of immobilized MTB complex formation with Fe³⁺ ion on environmental pH

The formation of a complex of immobilized MTB with the Fe³⁺ ion depends on the acidity of the medium. As seen in Figure 2, it was established that the optimal environment for complex formation is pH = 2.5, while at pH=4 and above, no complex compound is formed and only the reagent itself (MTB) gave an analytical signal.

CONCLUSION

In this research work, the optimal RPA1-fiber sorbent for immobilizing the methyltimol blue reagent was selected. A sorption-spectroscopic method for determining iron (III) ions with immobilized MTB has been developed. Optimal conditions for complex compound formation were studied: dependencies on time, pH of the medium, buffer solution, and the order of component addition.

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