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Electrochemical Determination of Lead (II) ION Using 4-(6-Phenyl-7h-[1,2,4] Triazolo[3,4-B][1,3,4]Thiadiazin-3-Yl)Aniline Reagent

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KEYWORDS

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ABSTRACT:

The electrochemical properties of the reagent 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline were investigated using cyclic voltammetry and amperometric techniques. An electrochemical method for the determination of lead(II) ions was developed and optimized. The influence of different solvents, including acetic acid, pentyl alcohol, butyl alcohol, and ethyl alcohol, on the determination of lead(II) ions was studied. It was found that the oxidation of lead(II) ions increases with higher acidity, with the highest oxidation observed in acetic acid. The half-wave potential in acetic acid was determined to be 0.3 V. Furthermore, the complex formation reaction between 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline and lead(II) was shown to occur at a rate of 4.8 per second. These findings provide insights into the electrochemical behavior of the reagent and its potential for lead(II) ion detection.

1. Introduction

It is known that lead is one of the heavy and toxic metals, and the creation of rapid and selective methods for their determination is one of the urgent problems. The use of sulfur-containing heteroatomic organic reagents in the determination of lead increases the selectivity of the method. A number of scientific researches are being carried out on the determination of lead. For example, a method of ultrafine microquantity determination of lead in food products using a modified glassy carbon electrode by voltammetric method has been proposed [1,2]. A spectrophotometric analysis method for determining lead from drinking water samples has been developed [3]. In this [4] work, lead ion was determined in the concentration range of 0.01 mg/l to 0.6 mg/l in aqueous solutions using the LIBC method. 5-(4-Aminophenyl)-1,3,4-Oxadiazole-2-thiol reagent with copper and lead ion from technological solutions [5], pyrrole-1-carboxylic acid (Py-CO2) [6], based on a DNA strand and DNA-templated silver nanoclusters (DNA-AgNCs)-mediated amplification strategy [7], based on vertically aligned 2D MoS2 [8] an electrochemical detection method of lead (II) was developed. An electrochemical sensor has been developed using a nanocomposite based on multi-walled carbon nanotubes (MWCNTs) and 2-aminothiophenol polymer modified with silver nanoparticles (AgNPs) for the simultaneous determination of Pb(II) and Cd(II) in food samples [9]. In another study, a sensor based on Schiff base modification with gold nanoparticles was proposed for the determination of Pb(II) in biological samples and medical applications [10]. A layered triple hydroxide (NiCoFe-LTH) hybridized with g-C3N4 was also used as an adsorbent for the extraction of Pb(II) to purify water and various food products from toxic elements [11]. Another electrochemical sensor was developed using CuO nanoparticles modified with polyaniline (PANI) for

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the simultaneous determination of Pb(II) and Cd(II) ions in water samples [12]. PbCrO4 nanoparticles and their integration with MXene Ti3C2Tx were proposed to create a sensitive photoelectrochemical sensor for the detection of cysteine in blood serum, which has potential applications in medical analysis and monitoring toxic substances [13]. An electrochemical sensor using CuO nanoparticles modified with PANI was also developed for the simultaneous detection of Pb(II) and Cd(II) ions in water samples [14]. Additionally, graphene oxide nanocomposites were proposed for electrochemical detection of Pb(II) and Cd(II) in complex matrices [15-17]. The use of metal-organic frameworks (MOFs) in combination with carbon nanoparticles has shown high sensitivity and selectivity for detecting Pb(II) and Cd(II) due to effective ion adsorption on the material's surface [18]. The application of electrothermal microplasma spectrometric methods also demonstrated good results in determining Cd, Zn, Pb, and Mn in water samples [19]. Multifunctional fluorescent bio-probes for the determination of Pb(II) and pesticides were proposed [20]. Recently, sensors using MOFs combined with carbon nanoparticles for detecting Pb(II) and Cd(II) were developed, ensuring high sensitivity and selectivity due to effective ion adsorption [21]. Fluorescent and quantum dots were also used for sensor systems [22]. Multifunctional sensors using two-dimensional materials such as MXene and graphene were applied for the detection of Pb(II) and Cd(II) ions with high sensitivity [23]. Furthermore, a sensor with infrared hydrogel was proposed for non-contact detection of Pb(II), utilizing photothermal conversion and the temperature sensitivity of the hydrogel for non-destructive desorption of Pb(II) [24]. The use of heteroatomic reagents in the determination of lead (II) ion increases the sensitivity of the developed method. Electrochemical methods, including cyclic voltammetric and amperometric methods of analysis, are methods of high sensitivity and reliability of analysis results of metal ions. Therefore, the method of electrochemical analysis was used for the determination of lead ion. Sulfur-containing triazole derivative was used as an organic reagent.

In this paper, we carried out the electrochemical studies of Pb(II) ion on the basis of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent. Cyclic voltammetry and amperometry methods, which are economically convenient, effective and highly accurate, were used.

2. Materials and Methods

Standart solution of lead (II) ion was prepared in a 100.00 ml flask by dissolving 0.0625 g of $PbSO_4 \cdot 5H_2O$ salt in bidistilled water and bringing it up to the mark of the flask with water.

0.1% 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4] thiadiazin-3-yl)aniline reagent was prepared by dissolving a sample in acetone.

The results of determination were carried out by manual patentiostat CS350, chronoamperometry, cyclic voltammetry and amperometric methods. Bidistilled water was used for the experiment. Bidistilled water was obtained using a Heal Force CR-RO30 distiller (China). The relative electrical conductivity of bidistilled water was 0.475 msm/m.

During the research, the effect of solvents such as ethyl alcohol, formic acid, acetic acid, trichloroacetic acid, propanol, butanol, pentanol, and background electrolytes on the electrooxidation of the reagent 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline was studied.

The procedure for determining lead (II) ion with 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent on a manual potentiostat CS350 is as follows: 1.0 ml of a $1 \cdot 10^{-3}$ M solution of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent was placed in the cell, 50 ml of solvent and 2.0 ml of a $1 \cdot 10^{-3}$ M standard solution of lead (II) ion were added. Then analytical signals were obtained by cyclic voltammetric method using manual potentiostat CS350.

3. Results and their Discussion

In electrochemical processes, the direction of analysis is determined by studying the electroactivity of a reagent or metal ion. To do this, the first the electroactivity of the reagent at different voltages and in different environments is studied by cyclic voltammetry, chronoamperometry, and electrochemical impedance spectroscopy.

The electrooxidation of 4-(6-phenyl-7H-[1,2,4]triazolo [3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent under the influence of various background electrolytes was studied

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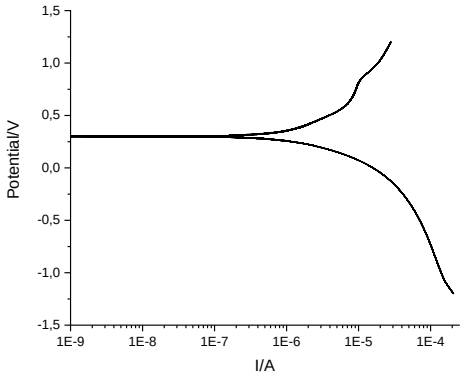
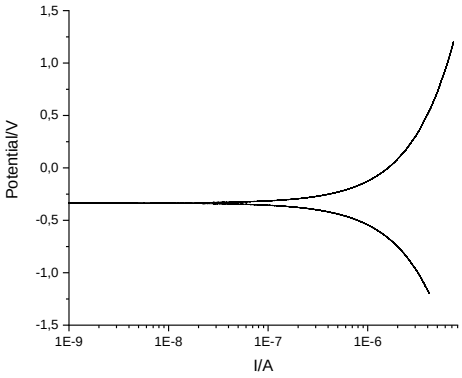
by cyclic voltammetry and chronoamperometric methods.

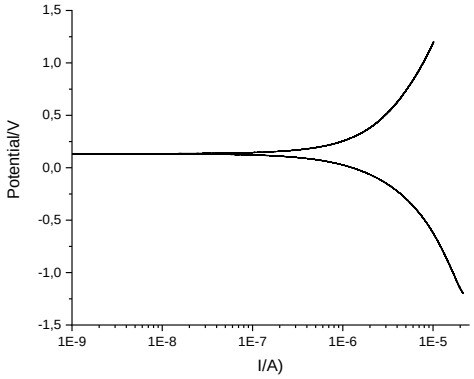
The electrochemical properties of the 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent were studied under the influence of various solvents. Add 1.0 ml of a 0.01% solution of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in acetone to a 50.0 ml cell and add: acetic acid (0.01 M); butanol (0.01 M); pentanol (0.01); ethanol (0.01 M) were added as background electrolytes and 2 platinum and silver chloride reference electrodes were placed, a voltage of 100 mV was applied to the electrode, the solution was rotated 800 times in 1 second, the solution was cooled, and cyclic voltammetry analysis was performed. The results of the analysis are presented in Figure 1.



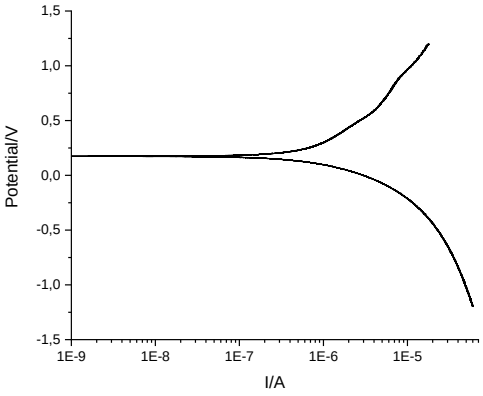
A. Cyclic voltammetry analysis of 6-phenyl-7H- [1,2,4] triazolo[3,4-b][1,3,4]thiadiazin-3-yl) aniline reagent in acetic acid solvent



B. Cyclic voltammetry analysis of 6-phenyl-7H-[1,2,4] triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in pentanol solvent



C. Cyclic voltammetry analysis of 6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in butyl alcohol solvent



D. Cyclic voltammetry analysis of 6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in ethyl alcohol solvent

Figure 1. Cyclic voltammetry analysis of 6-phenyl-7H-[1,2,4] triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent under the influence of various solvents

Figure 1 shows that the CV of the 6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in acetic acid, pentanol, butanol, and ethanol solvents explain the increase in oxidation of the organic reagent with increasing acidity of the solvents. An increase in the basicity of a solvent is explained by its reduction or decomposition, that's why acetic acid was chosen for the determination of lead ion.

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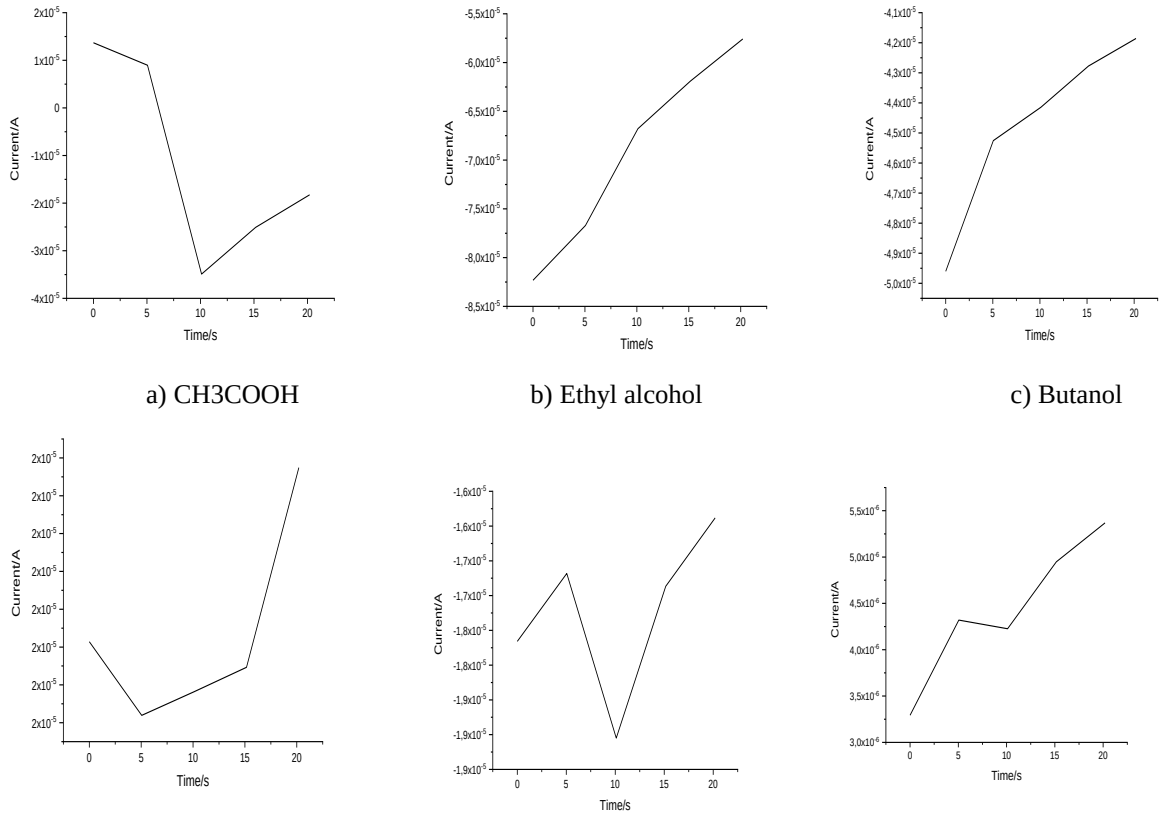
To prove the oxidation of an organic reagent in solution or the formation of a complex with a metal ion, the integral pulse voltammetric analysis method was also used, and voltammograms were obtained in various solvents.



a) CH3COOH b) Ethyl alcohol c) Butanol

d) Pentanol-2 e) Vinegar acid, 1ml 10 µg /ml Pb (II) f) Vinegar acid, 2 ml 10 µg / ml Pb (II)

Figure 2. IPAD analysis of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent in various solvents

Figure 2 shows that the oxidation of the reagent 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline in acetic acid occurs in 5 seconds, while in alcohols, oxidation decreases with increasing number of methylene groups. When lead(II) ion is added to the acetic acid solution, the oxidation takes place in 4.8 seconds, which proves that the complex is formed. Increasing the amount of metal ion appears to increase the time for complex formation.

The complex formation of lead with the reagent 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline was studied amperometrically at a voltage of 100 mV and in an acetic acid environment. 1.0 ml of a standard solution of lead(II) ion was placed in the cell and titrated with a standard solution of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline in acetone. The obtained results are presented in Table 1.

Table 1: Results of amperometric titration of a standard solution of 20.00 µg/ml lead with a solution of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline

№	**Found, µg** ($\bar{X}$ **±ΔX; R=0.95)**	**n**	**S**	**Sr**
1	20.56 ±0.25	4	0.14	0.007
2	20.42 ±0.16	4	0.09	0.004
3	20.44 ±0.19	5	0.10	0.005
4	20.52 ±0.36	5	0.18	0.009
5	20.11 ±0.12	5	0.06	0.003

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The table shows that the entered amount matches the found amount, which proves that there is no error.

The effect of interfering ions on the titration of lead (II) with a solution of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline reagent under optimal conditions was studied using an amperometric titration method. In this case, standard solutions of metal ions of various nature were added and an analytical signal was obtained. The results are presented in Table 2.

Table 2: Effect of interfering ions on the amperometric titration of a 20.00 μg/ml lead standard solution with a 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline solution

Interfering ions; [x]	**Entered quantity [x], μg**	$\frac{[x]}{[Pb]}$	**Founded Pb(II), μg; ($\bar{X}$ ±ΔX; R=0.95)**	**N**	**S**	**Sr**
Zinc	50.0	2.50	20.88 ±0.19	4	0.11	0.005
Nickel	95.0	4.75	20.49 ±0.44	4	0.25	0.012
Tin	200.0	10.00	2052±0.34	4	0.19	0,009
Copper (II)	60.0	3.00	20.43±0.48	4	0.27	0.013
Iron (III)	120.0	6.00	20.57±0.70	4	0.40	0.020
Chromium (III)	250.0	12.50	20.46±0.66	4	0.33	0.019
Selenium (IV)	220.0	11.00	20.38±0.75	5	0.37	0.018
Manganese (II)	260.0	13.00	18.96±0.73	4	0.42	0.022
Cadmium	70.0	3.00	20.39±0.36	5	0.18	0.009
Cobalt	80.0	4.00	20.58±0.78	4	0.22	0.011
Gold (III)	65.0	3.25	19.69 ±0.41	4	0.24	0.012
Silver(I)	75.0	3.75	19.87 ±0.35	4	0.20	0.010
Mercury (II)	150.0	7.50	20.87 ±0.60	4	0.34	0.016

The results show that the titration of lead is not affected by the above ions, which allows the developed ion to be used in the analysis of natural objects.

In conclusion, the electrochemical properties of 4-(6-phenyl-7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)aniline and its promising reagent for the detection of lead (II) ions, good sensitivity and selectivity, and the results obtained indicate the possibility of determining lead (II) ions in wastewater from the Navoy and Almalyk mining metallurgical combines.

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