

EXTRACTION OF LEAD (II) IONS USING DITHIZONE METHOD ON WASTEWATER OF KHONDZA

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Abstract. This paper studies methods of lead and zinc metal ions extraction by dithizone from Khondzha wastewater samples. Inorganic metals or ions can act back with dithizone to produce colourful coordination. The dithizone reagent is highly sensitive to the presence of heavy metals such as lead (Pb) and zinc (Zn) as designated in this study. Inorganic metals or ionic can react with dithizone to produce coloured coordination compounds. The resulting dithizoneate may be extracted with an organic solvent such as carbon tetrachloride (CCl₄). This study also involves adjusting the pH value to produce our desired dithizone metal product. The dithizone extraction method used in extracting the Pb element indicates a good performance.

Keywords: dithizone, extraction method, Khondzha, lead and zinc metal ions,

Introduction In addition to the widespread use and economic importance of zinc, lead and related compounds, they are included among the ecotoxicologically dangerous materials. This makes the removal, separation and purification of these metals from various sources an important topic of many fundamental and industrial studies[1].

It is known that liquid-liquid extraction techniques play an important role in the development of processes for the recovery and separation of various metal ions. Undoubtedly, the selectivity and efficiency of these methods are mainly determined by the extracting substances [2]. These heavy metals are non-biodegradable and accumulate in living systems, causing various serious diseases. These metals are widely distributed in the environment and eventually enter the food chain and are released from industrial activities (industrial waste, metal production, incineration plants, electroplating, fuel burning, etc.) and agricultural activities[3]. There are numerous techniques for extracting metals from water, but extracting metals from seawater stands out as particularly challenging due to factors like chloride ions and high salt concentrations. Additionally, this extraction process involves intricate procedures that demand significant manpower, extensive time, and substantial amounts of seawater samples[4]. In this research, the dithizone method was employed for trace metal extraction. Inorganic metals or ions can form colored coordination compounds when they react with dithizone. This dithizone reagent exhibits high sensitivity to heavy metals such as lead (Pb), cadmium (Cd), and zinc (Zn), as highlighted in this study. The resulting dithizoneate can then be extracted using organic solvents like carbon tetrachloride (CCl₄). Furthermore, this study entails pH adjustment measures to facilitate dithizoneate production[5].

Experimental part

Methods *Preparation of Standard Solution Dithizone.* 35 mg dithizone dissolved in 1000 mL of chlorophyll. Solution dithizone shaken until no visible sedimentation under volumetric flask. It then added 7.0 mL of ethanol before being stored in the laboratory refrigerator under 15 ° C. Before

use, incorporate 5.0 mL of HNO_3 into the solution and shake to avoid precipitate the volumetric glass flask[6,7].

There are many types of standard methods that have been used in determining the concentration of heavy metals in seawater samples. However, only a few studies have been able to effectively extract the traceable metal and the results of this study are more pronounced than seawater. Among such studies were carried out by Lim and made using the Cadmium method, the hydronium method. Both methods are an ideal method for assessing the content of heavy metals. However, both methods require a considerable length of time and complicated measures[8]. Additionally, these methods involve high risk reagents besides requiring high costs of either cost or maintenance costs. Dithizone or known as diphenylthiocarbazone or 1,5- diphenylthiocarbazone is a compound containing sulfur (S) and a good ligament. Dithizone is prepared with dithizone's reaction to react with various metals such as lead (Pb) produce colored complexes. With the pH value adjustment, the results for the determination of heavy metals such as lead (Pb) and zinc (Zn) will be easier and faster. For example, when conducting zinc (Zn) metal analysis, by adjusting the pH value to 4.0 - 5.5, zinc (Zn) will react with dithizone to produce zinc.

Results and Discussion

Zinc metal extraction percentage and concentration when stirring 10 mL of 5.0 ppm $\text{Zn}(\text{NO}_3)_2$ and 10.0 ppm $\text{Zn}(\text{NO}_3)_2$ diluted solutions. The data reveals that using the dithizone extraction method with distilled water for the 10.0 ppm $\text{Zn}(\text{NO}_3)_2$ solution yielded the highest trace metal concentration at 0.615 mg/L, with an extraction percentage of 30.8%. In contrast, extraction using seawater resulted in the lowest trace metal concentration for the 10.0 ppm $\text{Zn}(\text{NO}_3)_2$ solution, measuring 0.349 mg/L with a recovery extraction percentage of 17.5%. This discrepancy is attributed to the pH conditions being less suitable for zinc metal in seawater. The experimental pH was adjusted to a relatively low range of 2.5 - 3.0 by adding acetic acid, which was necessary for zinc production. Additionally, much of the zinc metal was lost during solvent evaporation via spinning evaporation and drying prior to analysis using ICP-OES. Zinc (with a boiling point of 1040.15 K) is more volatile compared to lead (2022.15 K) and cadmium (1180.15 K) under standard atmospheric pressure, leading to its easier loss during heating[9,10].

The incident observed is likely due to the pH level during titration, which was approximately 3.0, corresponding to the behavior of metals. pH plays a crucial role in the recovery of trace metals, with an extraction efficiency reaching 99.2%. Furthermore, contamination from cadmium metal can be identified through this analysis, with extraction percentages of 84.3% for 5.0 ppm and 70.7% for 10.0 ppm standard metal solutions in water blank samples. An air blank refers to a sample without added oceans. The contamination is suspected to originate from instrument contamination, possibly from the rotating evaporator or from the beaker during the solvent drying process[11].

Depicted the extraction percentage and concentration of extracted lead (Pb) from 5.0 ppm and 10.0 ppm standard $\text{Pb}(\text{NO}_3)_2$ solutions in 10.0 mL. The extraction percentage and concentration of extracted lead using artificial seawater samples were higher compared to using natural seawater samples. Specifically, for lead (Pb) elements, the effectiveness of metal recovery reached 66.2% with a 5.0 ppm standard metal solution and 62.6% with a 10.0 ppm standard metal solution using artificial seawater samples[12]. The lower extraction percentages observed in natural seawater samples are attributed to significant ion interference from chloride ions and salt matrices in the

water, which impacts the efficiency of the extraction method. Overall, the dithizone extraction method employed for extracting lead demonstrates good performance[13].

Conclusion

Research indicates that straightforward extraction methods like dithizone can effectively recover trace metals from seawater samples for analysis using ICP-OES. This method requires a relatively small volume of seawater sample, typically ranging from 180 to 220 mL, for each concentration of standard metal solution (e.g., 5.0 mg or 10.0 mg). The study also demonstrates the potential for simultaneous analysis of multiple elements (such as Pb, and Zn) by spiking samples with various standard solutions. This approach is advantageous for analyzing large quantities of trace metals because it enables the measurement of multiple elements using a minimal sample volume, thereby simplifying sample collection, storage, and analysis. By paying close attention to the pH value during analysis, researchers can enhance the accuracy of trace metal concentration readings obtained from experiments. Using the dithizone method, the concentrations of extracted metals were found to be 0.038 mg/L for Pb and 0.473 mg/L for Zn. These results demonstrate the effectiveness of the dithizone method in extracting and quantifying trace metals from seawater samples.

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