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Separation/enrichment of Trace Nickel(II) by Potassium Bromide—8-Hydroxyquinoline—Phenolphthalein System

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Abstract. A novel method for the separation/enrichment of trace Ni^{2+} by potassium bromide—8-hydroxyquinoline—phenolphthalein system was studied. The effects of different parameters on the enrichment yield of Ni^{2+} have been investigated. The possible enrichment mechanism of Ni^{2+} was discussed. The results showed that under the optimum conditions, the chelate precipitate of $\text{Ni}(\text{HQ})_2$ which produced by Ni^{2+} and HQ was quantitatively absorbed on the surface of microcrystalline phenolphthalein, while Zn^{2+} , Cd^{2+} , Co^{2+} , Mn^{2+} and Bi^{3+} could not be adsorbed. A new method for the separation/enrichment of trace Ni^{2+} was established. The proposed method has been successfully applied to the separation/enrichment of Ni^{2+} in the samples of synthetic water, and the enrichment yield was 95.2%~99.2%.

1. Introduction

Nickel is one of the essential microelements for humans, which plays an important role in maintaining physiological function of human body. Nickel can accumulate in the organs of human body, ingesting excessive nickel can cause inflammatory disease, cancer, system disorders, neurasthenia, lower fertility, teratogeny and so on. Nickel usually dissolves in nature water in the form of halide, nitrate, sulfate and inorganic complex. Therefore, it is of great importance and significance for environment science and life science to separate/enrich and determinate trace nickel in environmental water samples.

It was impossible for $\text{Ni}(\text{II})$ to be directly determined due to the low concentration of $\text{Ni}(\text{II})$ in environmental samples. In order to achieve accurate results, separation and enrichment step is necessary when the concentration of $\text{Ni}(\text{II})$ is too low to be directly determined. There are many other methods to separate and enrich $\text{Ni}(\text{II})$, such as solvent extraction,¹⁻² solid phase extraction,³⁻⁴ cloud point extraction,⁵⁻⁶ extraction chromatography,⁷ ion exchange,⁸ flotation separation,⁹ electrodialysis separation,¹⁰ liquid membrane separation,¹¹ emulsion liquid membrane separation,¹² chelating resin separation,¹³ ultrasound-assisted extraction.¹⁴

In this paper we have studied the separation/enrichment of trace Ni^{2+} by potassium bromide—8-hydroxyquinoline—phenolphthalein system. By controlling pH 3.0, the chelate precipitate of $\text{Ni}(\text{HQ})_2$ which produced by Ni^{2+} and HQ was quantitatively absorbed on the surface of microcrystalline phenolphthalein, while Zn^{2+} , Cd^{2+} , Co^{2+} , Mn^{2+} and Bi^{3+} could not be adsorbed. Therefore, the quantitative separation of Ni^{2+} from these metal ions could be achieved without any masking agent. A method for the separation/enrichment of Ni^{2+} was established, and it has been successfully applied to



the separation/enrichment of Ni^{2+} in the samples of synthetic water with satisfactory results. This method not only has the advantages of non-organic solvent liquid-solid extraction system,¹⁵⁻¹⁶ but also has characteristics such as simply, rapidness, convenience and economy and so on.

2. Experiment

2.1. Equipment and reagents

A model 722S spectrophotometer (Shanghai No.3 Analysis Equipment Plant) was used for photometric measurements.

8-Hydroxyquinoline (HQ) solution: $0.010 \text{ mol}\cdot\text{L}^{-1}$. Phenolphthalein ethanol solution: 5%. Borax solution: $0.1 \text{ mol}\cdot\text{L}^{-1}$ $1.0\times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ of 4-(2-pyridylazo) resorcinol (PAR) ethanol solution was prepared by dissolving 0.1076g PAR in 500mL of ethanol. A stock of standard solution of Ni^{2+} : $1.000 \text{ g}\cdot\text{L}^{-1}$, a working standard solution was prepared by appropriately diluting the stock standard solution. Buffer solutions of different pH was prepared as reference 17.

All reagents were of analytical reagent grade. Bidistilled water was used throughout.

2.2. Method

$50\mu\text{g}$ of Ni^{2+} , a given amounts of $0.010 \text{ mol}\cdot\text{L}^{-1}$ HQ solution and 5% phenolphthalein ethanol solution were added into a 25 mL ground color comparison tube. Then adjust the pH with buffer solutions and dilute the mixture to 10.00 mL with bidistilled water, then 0.50 g KBr was added. Shaken adequately and they were kept still for a moment. 1.00 mL of supernatant liquid was transferred into another 25 mL ground color comparison tube, then 1.5 mL 4-(2-pyridylazo) resorcinol (PAR) ethanol solution and 3.0 mL borax solution were added. The solution was diluted to the mark and the absorbance was measured at 494 nm against the reagent blank prepared in the same way. The amount of Ni^{2+} remained in the solution was calculated. The enrichment yield of Ni^{2+} (E/%) was calculated according to the determination results.

The determination of the content of trace Ni^{2+} in microcrystalline phenolphthalein phase is as follows: the precipitation by filtration was dissolved in ethanol, and then was transferred into a 25 mL ground color comparison tube and diluted the mixture to 10.00 mL with bidistilled water, shaken adequately and they were kept still for a moment, and the content of Ni^{2+} was determined in the same method above.

Photometric analysis of other metal ions was referring the reference 18.

3. Results and discussions

3.1. Effect of phenolphthalein dosage

The effect of phenolphthalein dosage on the enrichment yield of Ni^{2+} was shown in Figure 1. The results show that the enrichment yield of Ni^{2+} is 42.4% in the absence of phenolphthalein. It is probable that the partially Ni^{2+} can directly react with HQ to form the chelate precipitate of $\text{Ni}(\text{HQ})_2$. It is consistent with the phenomenon of precipitate which can be directly observed without phenolphthalein in experiment.

The enrichment yield of Ni^{2+} increase with the increase of phenolphthalein dosage, when the dosage of phenolphthalein was up to 1.00 mL or more, the enrichment yield of Ni^{2+} is 100%. It is probable that HQ adsorbed onto microcrystalline phenolphthalein (PP) has higher concentration and can react with Ni^{2+} left in the solution to form the chelate precipitate of $\text{Ni}(\text{HQ})_2$, and the chelate can be adsorbed on the surface of microcrystalline PP. This indicates that microcrystalline phenolphthalein can perfectly adsorb trace Ni^{2+} in solution. So, 1.00 mL of phenolphthalein was selected in the subsequent studies.

3.2. Effect of HQ dosage

The effect of HQ dosage on the enrichment yield of Ni^{2+} was studied, which was shown in Figure 2. The enrichment yield of Ni^{2+} was zero without HQ in the solution, or Ni^{2+} was totally left in the solution. It is likely that Ni^{2+} cannot be absorbed directly on the surface of microcrystalline PP when the solution without HQ. With the increase of HQ dosage, the enrichment yield of Ni^{2+} increased. When the dosage of HQ was 1.00 mL, Ni^{2+} can be completely retained on the surface of microcrystalline PP. For the reason that Ni^{2+} can react with HQ to form the chelate precipitate of $\text{Ni}(\text{HQ})_2$, and the chelate precipitate can be adsorbed on the surface of microcrystalline PP. The amount of $\text{Ni}(\text{HQ})_2$ increases when the solution includes more HQ. It leads to the increase of enrichment yield of Ni^{2+} . The enrichment yield of Ni^{2+} maintained 100% with further increasing the dosage of HQ. Hence, 1.00 mL of HQ was chosen for all further studies.

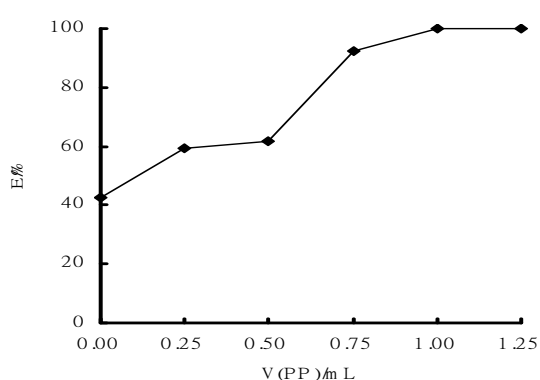


Figure 1. Effect of phenolphthalein dosage on the enrichment yield of Ni^{2+} (Ni^{2+} : $50\mu\text{g}$; HQ ($0.010\text{ mol}\cdot\text{L}^{-1}$): 1.00mL; Phenolphthalein: 5%).

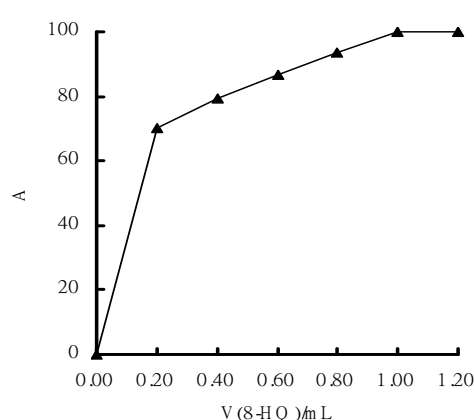
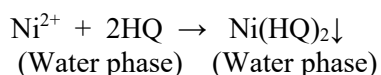


Figure 2. Effect of HQ dosage on the enrichment yield of Ni^{2+} (Ni^{2+} : $50\mu\text{g}$; Phenolphthalein (5%): 1.00 mL; HQ : $0.01\text{ mol}\cdot\text{L}^{-1}$).

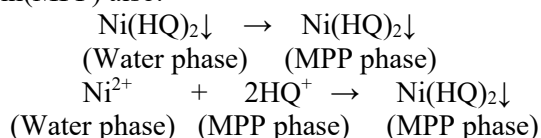
3.3. Enrichment mechanism of Ni^{2+}

Based on the results above, it can be concluded that only in the simultaneous presence of HQ and phenolphthalein in the solution, can Ni^{2+} be completely enriched. Therefore, the enrichment mechanism of Ni^{2+} is as follows:

(1) When the solution includes HQ but not phenolphthalein, partially Ni^{2+} can directly react with HQ to form the chelate precipitate of $\text{Ni}(\text{HQ})_2$, which leads to the decrease of the concentration of Ni^{2+} in the solution.



(2) When the solution includes both HQ and phenolphthalein, the chelate precipitate of $\text{Ni}(\text{HQ})_2$ in water phase was adsorbed on the surface of microcrystalline phenolphthalein. Meanwhile HQ adsorbed onto microcrystalline phenolphthalein reacts with Ni^{2+} left in the solution to form the chelate precipitate of $\text{Ni}(\text{HQ})_2$, and it can be adsorbed on the surface of microcrystalline phenolphthalein(MPP) also.



3.4. Effect of various salts

The effects of various salts including NaCl, KNO₃, (NH₄)₂SO₄ and KBr on the enrichment yield of Ni²⁺ were investigated. The results showed that KBr did not affect the enrichment yield of Ni²⁺, while NaCl, KNO₃, (NH₄)₂SO₄ decreased the enrichment yield of Ni²⁺ in a certain extent. When KBr dosage was 0.50g, 1.0g, 3.0 g, the enrichment yields of Ni²⁺ were 100%. Therefore, 0.50 g KBr was chosen in the further studies.

3.5. Effect of pH

Under the optimum conditions, the effects of pH on the enrichment yield of different metal ions were investigated. The experimental results showed that the enrichment yields of Ni²⁺ were 100% in the pH range 3.0~7.0, while Zn²⁺, Cd²⁺, Co²⁺, Mn²⁺ and Bi³⁺ could not be enriched at pH 3.0, or the enrichment yield of Zn²⁺, Cd²⁺, Co²⁺, Mn²⁺ and Bi³⁺ were zero. Therefore, Ni²⁺ could be separated from Zn²⁺, Cd²⁺, Co²⁺, Mn²⁺ and Bi³⁺ in the solution at pH 3.0

3.6. Separation experiments

By controlling pH 3.0, when the dosage of 5% phenolphthalein solution was 1.00 mL and 0.010 mol·L⁻¹ HQ solution was 1.00 mL respectively, the separations of Ni²⁺ from Zn²⁺, Cd²⁺, Co²⁺, Mn²⁺ and Bi³⁺ in synthesized samples of binary and polybasic system were studied respectively. The results were shown in Table 1 and Table 2.

Table 1 The separation results of binary-mixed ions (pH=3.0)

Mixed ions	Dosage of metal ions(μg)		Content of metal ions in water phase (μg)		Enrichment yield(E/%)	
	Ni	Me	Ni	Me	Ni	Me
Ni ²⁺ -Zn ²⁺	50	100	0.2	101.0	99.6	-1.0
	50	200	0.1	212.0	99.8	-6.0
	50	300	0	327.0	100	-9.0
Ni ²⁺ -Cd ²⁺	50	100	0	105.4	100	-5.4
	50	200	0.2	210.6	99.6	-5.3
	50	300	0.1	317.1	99.8	-5.7
Ni ²⁺ - Co ²⁺	50	100	0.2	97.0	99.6	3.0
	50	200	0.1	193.7	99.8	3.2
	50	300	0.1	307.5	99.8	-2.5
Ni ²⁺ - Mn ²⁺	50	100	0.1	101.4	99.8	-1.4
	50	200	0.2	209.1	99.6	-4.6
	50	300	0	320.6	100	-6.9
Ni ²⁺ - Bi ³⁺	50	100	0	105.6	100	-5.6
	50	200	0.2	189.1	99.6	5.5
	50	300	0.3	291.7	99.4	2.8

Me represents other metal ions except Ni²⁺.

Table 2. Separation results of Ni^{2+} from polybasic-mixed ions (pH=3.0)

Number of the synthesized samples	1	2	3	4
Dosage of Ni^{2+} (μg)	50.0	50.0	100.0	200.0
Dosage of Zn^{2+} , Cd^{2+} , Co^{2+} , Mn^{2+} and Bi^{3+} (μg)	50.0	100.0	200.0	400.0
Ni^{2+} found in solid phase (μg)	49.3	49.0	95.2	198.4
Enrichment yield of Ni^{2+} (E/%)	98.6	98.0	95.2	99.2

4. Conclusion

In this paper, a novel method for the separation/enrichment of trace Ni^{2+} by potassium bromide—8-hydroxyquinoline—phenolphthalein system was reported. The proposed method has been successfully used for the separation/enrichment of trace Ni^{2+} in the samples of synthetic water. It was obvious that this study had certain practical significance and foreground of application on establishing a new methods of separation/enrichment of trace nickel(II).

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