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Research Article

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Determination of neodymium by fading spectrophotometry

Qing-Zhou Zhai

Research Center for Nanotechnology, Changchun University of Science and Technology, Changchun, Weixing Road, P. R. China

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ABSTRACT

In an 80 ℃ water bath, Nd^{3+} can catalyze decoloration reaction of ponceau S by H_2O_2 oxidation. Based on this, a fading spectrophotometric new method for the determination of neodymium was put forward. The maximum absorption wavelength of the system is 450 nnm and at this wavelength there is a good linear relationship between a concentration of 0.2 ~ 4 μ g / mL of Nd^{3+} and the absorbance difference. The method has been applied to the determination of neodymium in molecular sieve.

Keywords: neodymium, fading spectrophotometry, ponceau S, hydrogen peroxide, molecular sieve.

INTRODUCTION

Neodymium is a light rare earth element and its abundance in the earth's crust is 2.4 ×10^{-3}%. Neodymium metal is mainly used for producing neodymium iron boron permanent magnetic material, but also widely used in electronics, mechanics and other industries. Adding appropriate amount of neodymium in magnesium or aluminum alloy, can improve high temperature performance, air tightness and corrosion resistance of the alloy. These materials are widely used in aerospace materials. In the petroleum chemical industry, the molecular sieve catalyst made by rare earth, has the advantages of high activity, good selectivity, strong ability of anti heavy metal poisoning, therefore it can replace aluminum silicate catalyst for the catalytic cracking process of oil [1].

Spectrophotometric method for the determination of neodymium [2], compared to the inductively coupled plasma (inductively coupled plasma, ICP) - atomic emission spectrometry (atomic emission spectrometry, AES) and other large instrument analysis method [3], has the advantages of cheap instrument, simple operation, rapidity, still is concerned for analysis workers. Although 2, 6-dibromo-4-methyl sulfonazo [4], dibromo-*p*-chloro-chlorophosphonazo [5], 7-iodo-8-hydroxyquinoline-5-sulfonic acid [6] have been put forward for the spectrophotometric determination of Nd, but the selectivity of method is still not ideal and a new spectrophotometric method for the determination of neodymium still needs to be established. Ponceau S（$C_{22}H_{12}N_4Na_4O_{13}S_4$）, was ever used for the determination of cobalt [7]. To the best of our knowledge, no study report on the spectrophotometric determination of neodymium with ponceau S is seen. In this article, hydrogen peroxide and ponceau S oxidation discoloration is studied in a medium of 2.8 × 10^{-2} mol/L hydrochloric acid and neodymium(III) has a catalytic action. Based on this fading reaction, a new method is developed for the spectrophotometric determination of neodymium.

EXPERIMENTAL SECTION

Apparatus and reagents

A 722S spectrophotometer （Shanghai Lingguang Technique Co. Ltd., China） equipped with 1-cm cells was used for all absorbance measurements. The HH-4 digital display constant temperature water-bath boiler (Jintan City Ronghua Instrumental Manufacture Co. Ltd., China) was used for the control of temperature.

The stock standard solution of neodymium (III), 1 mg / mL, was prepared by dissolving 0.2330 g Nd_2O_3 (Specpure) in 5 mL of 2 mol/L hydrochloric acid and then diluted to100 mL with water. The working standard solution of neodymium (III), 10 μg / mL，was prepared by diluting the stock solution with water. Ponceau S solution, 5.0×10^{-3} mol/L, was prepared by dissolving 0.3729 g of ponceau S in 100 mL of water. Hydrochloric acid solution：1.0 mol/L. Hydrogen peroxide solution：15%(V/V). It was placed in a brown bottle and kept in a 4℃ refrigeratory for storage avoiding light. Unless otherwise stated，all of the chemicals used were of analytical reagent grade, and doubly deionized was used throughout the study.

Standard procedure

To two 25-ml calibrated flasks, 0.8 mL of ponceau S solution, 1.5 mL of hydrogen peroxide solution, 0.7 ml of hydrochloric acid solution were in turn added, respectively. To one of them, an appropriate amount of Nd(III) standard solution (Catalytic reaction, for condition experiment 50 μg Nd^{3+} was used) was added and its absorbance is recorded as *A*. In another calibrated flask, no Nd(III) standard solution (Non-catalytic reaction) was added and its absorbance is recorded as A_0. The above solutions were diluted to the mark with water and shaken well. They were heated in a boiling water bath at 80℃ for 15 min and rapidly taken out and cooled for 10 min by running water. Using water as reference, at 450 nm absorbance was measured with 1 cm cell. The absorbance difference value of reagent blank and the solution containing Nd(III) could be obtained, $\Delta A = A_0 - A$.

Procedure for the analysis of molecular sieve sample

0.2000 g of molecular sieve was accurately weighed and placed in a 50-mL beaker. 10 mL of HF（1+1, V/V）solution was added to the beaker. The contents was heated for dissolution and evaporated to near dryness. It was dissolved with a definite amount of water, transferred to a 10-mL volumetric flask and diluted to the mark with water. An appropriate amount of the aliquot of the above resulting solution was taken out, placed in a 25-mL volumetric flask and determined neodymium contents using the recommended procedure. At the same time recovery experiment of the sample was made out and contrast determination was done with the dibromo-*p*-chloro-arsenazo spectrophotometry [8].

RESULTS AND DISCUSSION

Absorption spectra

Absorption spectra are shown as Fig. 1. Under the present experimental condition, the maximum absorption wavelengths of the catalytic reaction (Curve *b*) and non-catalytic reaction (Curve *a*) systems are both at 450 nm. At this wavelength, the absorbance difference was a maximum. In the experiment, 544 nm was selected as the determination wavelength.

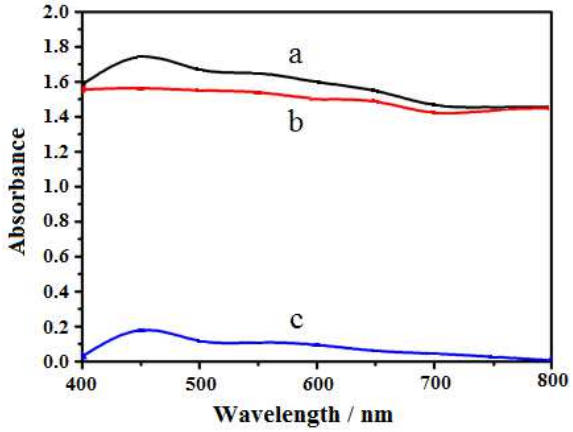

Fig.1: Absorption spectra

(a) ponceau S + H_2O_2— non-catalytic reaction A_0; (b) ponceau S + H_2O_2 + Nd^{3+} — catalytic reaction A; (c) net catalytic reaction $\Delta A = A_0 — A$. [Nd^{3+}] = 1.7 ×10^{-5} mol/L; [ponceau S] = 1.6 × 10^{-4} mol/L; [H_2O_2] = 0.50 mol/L; [HCl] = 2.8 × 10^{-2} mol/L; heating temperature = 80 ℃; heating time = 15 min

Effect of hydrochloric acid amount

The variations of ΔA were studied over the range of 0.1 - 2.0 mL of 1.0 mol/L hydrochloric acid were studied. Over the range of 0.1-0.6 mL, ΔA increased with the increase in hydrochloric acid amount. Over the range of 0.6-0.8 mL,

ΔA attained a maximum. Then, with further increase in hydrochloric acid amount, ΔA decreased. In subsequent experiments, the hydrochloric acid solution volume was fixed at 0.7 mL.

Effect of reagent concentration

The effect of reagent concentration on ΔA was studied by varying the reagent concentration under the optimum conditions at a constant neodymium (III) concentration of 1.7×10^{-5} mol/L. The results indicated that the maximum ΔA was attained with 0.7 - 0.9 mL of 5.0×10^{-3} mol/L ponceau S. In this study, 0.8 mL was selected. The results on H_2O_2 amount displayed that 1.4 - 1.6 mL of 15%(V/V) H_2O_2 led to a maximum ΔA. So 5 mL of this H_2O_2 solution was selected.

Effect of heating temperature

The results showed that below 40℃ the reaction did not occur. When temperature was in 40 - 80℃, ΔA increased with the increasing of heating temperature. At 80℃, ΔA reached a maximum. When the heating temperature was 80 - 100℃, ΔA decreased with the increase of the heating temperature. 80℃ water bath heating was chosen and running was used for cooling to terminate the reaction. For the determined 40 - 80℃ data, regression process was made to obtain a linear equation: $\log(\Delta A) = 0.299/T - 1.799$, the correlation coefficient r = 0.9959. According to the slope of the equation, the activation energy of the catalyzed reaction obtained was Ea = 5.725 KJ · mol^{-1}.

Effect of heating time

The results showed that when the heating time was 2 – 15 min, ΔA gradually increased with increasing in heating time and ΔA was linear with time. At 15 min ΔA attained a maximum. When the heating time was greater than 15 min, ΔA reduced with the increase of the heating time. Therefore, the heating time was selected to be 15 min. By plotting ΔA against t, its linear regression equation was: ΔA = 0.0080t (min) + 0.0455, with a correlation coefficient r = 0.9981. The reaction rate constant was $k = 1.33 \times 10^{-4}\ s^{-1}$ and the half-life was 86.6 min.

Stability of system

The experimental results showed that a variation of ΔA was less than 5% within 1.5 h and the system kept stable.

Analytical characteristics-Working curve，sensitivity，precision and accuracy

The experimental results showed that in 25 mL of solution Nd^{3+} mass over the range of 5 ~ 80 μg and ΔA presented a good linear relationship and its regression equation was：ΔA = 0.0027C + 0.0195（C：μg / 25mL），with a correlation coefficient of 0.9992. According to the general procedure, 11 parallel determinations of 2.0 μg / mL of Nd^{3+} were made and the relative standard deviation of method was 0.13%. For 11 determinations of reagent blank, according to 3S / K method (S is the standard deviation of 11 parallel determinations of reagent blank，K is the slope of working curve)，the detection limit of method was calculated to be 0.080 μg / mL.

Selectivity of method

Under the recommended optimum conditions, the effects of various foreign substances on the determination of 60 μg neodymium (III) were studied. Tolerable levels (5% error maximum) of foreign substances on the determination of neodymium are as follows (W/W)：K^+，Na^+，Cl^-，SiO_3^{2-} (1200)；Ca^{2+}，Fe^{3+}，Al^{3+}，SO_4^{2-}，PO_4^{3-}，HAc (200)；Ag^+，Ni^{2+}，Cd^{2+}，Mg^{2+}，B（III）(100)；F^-（80）；Co^{2+}，W（Ⅵ），Mo（Ⅵ）(50)；Br^-(40)；Fe^{2+}，Zn^{2+}，Mn^{2+} (1)；I^-(20)；EDTA(100)；Sr^{2+}, Ba^{2+}，Cr(VI) (1)；Zr^{4+}，Ti^{4+}(0.1)；MnO_4^-(0.05)； La^{3+}，Y^{3+}(0.1)；tartaric acid (50) ； hydroxylamine hydrochloride, triethanolamine（10）； oxalic acid, ascorbic acid, citric acid (5). The selectivity of the procedure is better than those given in the literature [4-6] with respect to Ca^{2+} and Cd^{2+}.

Table 1: Analytical results of samples

Sample	Found (n = 11，%)	Relative standard deviation (%)	Added (μg / g)	Recovered (μg / g)	Recovery (%)	Dibromo-*p*-chloro-arsenazo spectrophotometric contrast method (%)[8]
No.1	0.35	0.48	10.00	9.99	99.9	0.35
No.2	0.23	1.03	10.00	10.03	100.3	0.22

Sample analysis

In order to verify the practicability of this method, this study applied the proposed method for the determination of neodymium in molecular sieves. The determination results are listed in table 1. It can be seen that the analysis results are very satisfactory.

CONCLUSION

This paper presented a new method for the determination of neodymium with Nd^{3+}-H_2O_2-Ponceau S system. At the maximum absorption wavelength of 450 nm, a Nd^{3+} concentration over the range of 0.2 ~ 3.2 μ g / mL has a good linear relation with the absorbance difference. The detection limit of method was 0.080 μ g / mL. The method was used for determination of neodymium in molecular sieve with satisfactory results

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