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Research Article

Simultaneous Spectrophotometric Determination of Aluminium (III) and Iron(III) using 5-BHMBHBH

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ABSTRACT

Al(III) and Fe(III) react with 5-Bromo-2-hydroxy-3-methoxybenzaldehyde-4-hydroxybenzoichydrazone(5-BHMBHBH) at pH 4.0 to give yellow and brown coloured water soluble complexes. Al(III) shows maximum absorbance at 390 nm while Fe(III) shows maximum absorbance at 390 nm. It is also noticed that Al(III) shows no absorbance beyond 480 nm while Fe(III) shows considerable absorbance. The reagent has negligible absorbance at both 390nm and 480 nm.The absorbance of Fe(III) at 390 nm is 1.816 times that of its absorbance at 480 nm. Thus 390 nm and 480 nm are selected for carrying out simultaneous spectrophotometric determination of Al(III) and Fe(III). The molar absorptivities for Al(III) at 390 am is 2.66 ± 0.004 $\times 10^4$ lit mol^{-1} cm^{-1} .The molar absorptivities for Fe(III) at 390 nm and 480 nm are 3.233 ± 0.002 $\times 10^4$ lit mol^{-1} cm^{-1} and1.947 $\times 10^4$ lit mol^{-1} cm^{-1} respectively. Hence, simultaneous spectrophotometric determination of Al(III) and Fe(III) in synthetic mixtures carried out. The method developed is highly sensitive, selective, precise and rapid for the direct spectrophotometric determination of Al(III) and Fe(III). The method is applied for the determination of Al(III) and Fe(III) in alloy samples.

Key words: Al(III), Fe(III), 5-BHMBHBH, Spectrophotometry and Simultaneous spectrophotometry.

INTRODUCTION

Aluminium (III) and iron(III) are associated with each other in many alloys of industrial importance and in environment. There is a necessity for methods with high sensitivity and selectivity for the simultaneous determination of Al (III) and Fe(III) as they play important roles in biology and industry[1]. In addition, these two elements are present to gether in real samples. Therefore it is highly important to determine them in presence of one another.

Several reports have been published on the simultaneous determination of aluminium and iron, including the expensive instrumental technique of X-ray fluorescence with radio isotopic excitation[2]. Chromatographic techniques such as high performance liquid chromatography (HPLC)[3-4] and atomic absorption spectrometry[5-6] have been applied for simultaneous determination of these two ions in a mixture in the presence of many ions, although in some cases sensitivity is relatively low and a preconcentration step is needed. Inductively coupled plasma atomic emission spectrometry (ICP-AES)[7],inductively coupled plasma mass spectrometry (ICP-MS) and ICP-AES[8], combined on line HPLC and high resolution ICP-MS[9] have been also applied for simultaneous determination of aluminium and iron. Recently simultaneous determination of aluminium and iron was reported in high salt content matrices by adsorptive stripping voltametry[10]. This method requires 90^0C temperature and long analysis time.

Despite the success of the above mentioned methods, there is still great interest in the application of spectrophotometric methods in determination of metal ions, due to both the rapidity and simplicity of the technique. We have reported sensitive spectrophotometric procedures for the determination of Al(III)[11] and Fe(III)[12] using 5-BHMBHBH.

Now we report a sensitive and selective method for the simultaneous determination of Al(III) and Fe(III). The proposed method is easily applied for their simultaneous determination in standard reference samples.

EXPERIMENTAL

Apparatus:

The absorbance and pH measurements were made on a Perkin Elmer (LAMDA 25) UV-Visible spectrophotometer (Model UV-160A) controlled by a computer fitted with 1cm path length quartz cells and an ELICO digital pH meter of (Model LI 613) respectively.

Reagents

5-bromo-2-hydroxy-3-methoxybenzaldehyde-p-hydroxy benzoic hydrazone (5-BHMBHBH) was synthesized in the laboratory by condensing 5-Bromo-2- hydroxy-3-methoxybenzaldehyde and p-hydroxybenzoic hydrazide. A 0.01M DMF solution of the reagent is used in the studies.

All the reagents used were of analytical reagent grade unless otherwise stated. Stock solutions of Al(III) and Fe (III) (1.0×10^{-2}M) were prepared in double distilled water and standardized[13,14] . A buffer solution of pH 4.0 is used.

General Procedure

A 10 ml volumetric flask containing 5 ml of buffer solution pH 4.0, 1.5 ml of DMF and 0.5 ml of 5-BHMBHBH (1.0×10^{-2}M)solution and known aliquot of the synthetic mixture containing Al(III) and Fe (III) are added. The absorbance of the solution is measured at 390 nm and 480 nm against the reagent blank.

RESULTS AND DISCUSSION

Al(III) and Fe(III) are two important metal ions that finds use in industry. They are present together in some alloy samples. Hence, their simultaneous determination is of importance. This increases the selectivity of the method. No simultaneous direct spectrophotometric method is reported for determination of Al(III) and Fe(III) in the literature.

Aluminium (III) reacts with 5-BHMBHBH in the pH range of 2.0 to 8.0 to form yellow coloured water soluble complex. The colour intensity is maximum in the pH range 2.0–7.0. The complex shows λ_{max} at 390 nm. Iron(III) reacts with 5-BHMBHBH giving brown coloured water soluble complex in the pH range of 2.0 to 8.0 .The colour intensity is the maximum in the pH range 3.5–7.5. The complex shows absorbance maximum at 390nm.

Thus, it is clear that both Al (III) and Fe (III) react with 5-BHMBHBH forming water soluble coloured complexes and have maximum absorbance at pH 4.0. It is, therefore, possible that Al (III) and Fe (III) can be determined simultaneously using 5- BHMBHBH spectrophotometrically. The absorption spectra of the following solutions are recorded for selecting the wavelengths for absorbance measurements. **(a)** The absorption spectrum of 5-BHMBHBH (5.0×10^{-3} M) at pH 4.0 is recorded by following the general procedure and presented in fig. 1a, **(b)**The absorption spectrum of Al (III)-5-BHMBHBH complex (2.0×10^{-5} M) is recorded by following the general procedure and presented in fig. 1b. **(c)** The absorption spectrum of Fe (III) – 5-BHMBHBH complex (2.0×10^{-5} M) is recorded by following the general procedure and presented in fig.1c.

From the spectra in fig.1, it is clear that Al(III) shows absorbance maximum at 390 nm and Fe(III) also shows absorbance maximum at 390 nm, but at 480 nm Al(III) shows zero absorption. From experiments it is found that the absorbance of solutions containing mixture of Al(III) and Fe(III) at 390 nm and 480 nm is equal to the sum of the absorbance values due to individual Al (III) and Fe (III) species at these wavelengths. This suggests that it is possible to carry out a simultaneous spectrophotometric determination of Al (III) and Fe(III) using 5-BHMBHBH by measuring the absorbance of the solution mixtures at 390 nm and 480 nm.

The absorbance of the solution mixtures containing Al (III) and Fe (III) complex species is recorded at 390 nm and 480 nm at pH 4.0. From the absorbance value at 480 nm (where Al(III) has zero absorbance) the amount of Fe(III) is computed from the predetermined calibration plot. The amount of Al(III) is calculated from the absorbance values measured at 390 nm. It is obtained after subtracting the absorbance due to Fe(III) at this wavelength . The absorbance of Fe(III) at 390 nm is 1.816 times that of its absorbance at 390 nm. Therefore, the amount of Al(III) is computed from the absorbance measured at 390 nm. The following equation is employed to calculate the amounts of Al(III) at 390 nm.

$$\text{Al(III)} = \frac{A_{390} - \left(A_{480} \times 1.816\right)}{{}_{390}\varepsilon^{Al}} \times 26.98 \times 10^{3} \quad .. \ 1$$

where A_{390} and A_{480} are the absorbance values of the solution mixtures at 390 and 480 nm respectively. ${}_{390}\in^{Al}$ is the molar absorptivity of Al(III) at 390nm.The factor 1.816 is to convert the absorbance at 480 nm to equalize to the absorbance contribution of Fe(III) at 390 nm. 26.98×10^{3} is the factor to convert the concentrations of Al(III) into micro grams.

The simultaneous spectrophotometric determination of Al(III) and Fe(III) in synthetic mixtures is carried out by following the general procedure. The results are presented in table-

Applications

The method is applied for the determination of Al(III) and Fe(III) in alloy samples. The alloy samples are brought into solution by adopting by the procedure reported literature and the percentages of Al(III) and Fe(III) present are determined by the present method. The certified composition of alloy samples are given underneath the table containing the results and are presented in table-2.

CONCLUSION

A survey of literature reports no method for simultaneous direct spectrophotometric determination of Al(III) and Fe(III). Hence, the present method is first report of direct spectrophotometric determination of Al(III) and Fe(III). The method is rapid, precise, sensitive and highly selective for the determination of Al(III) and Fe(III) in alloy samples.

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Table 1
Simultaneous determination of Al(III) and Fe(III) in synthetic mixtures

Amount taken (μg/ml)		Amount found* (μg/ml)		Relative error (%)	
Al(III)	Fe(III)	Al(III)	Fe(III)	Al(III)	Fe(III)
0.323	0.558	0.322	0.553	0.92	-0.89
0.323	0.279	0.318	0.284	-0.61	0.56
0.323	1.117	0.326	1.119	-0.30	-0.51
0.323	1.396	0.320	1.390	0.61	-0.45
0.323	1.675	0.323	1.671	0.30	0.25
0.377	0.837	0.380	0.840	-0.53	0.56
0.431	0.837	0.427	0.842	0.23	-0.85
0.647	0.837	0.640	0.847	0.46	0.28
0.755	0.837	0.759	0.831	-0.52	-0.71

* Average of five determinations

Table -2
Simultaneous determination of Al(III) and Fe (III) in different samples

Alloy sample	Composition (%)				Relative error(%)	
	Certified		Found*			
	Al(III)	Fe(III)	Al(III)	Fe(III)	Al(III)	Fe(III)
[a]SRM 679	11.1	9.05	10.94	9.15	-1.44	1.10
[b]CRM851-1	2.01	19.9	2.00	20.29	-0.49	1.95

* Average of five determinations
[a]Composition: Ca:0.1628%,Mg:0.7552%, K:2.433%, Si:24.34%, Na:0.1304%,Ti: 0.577%
[b]Composition: SiO_2:13.53%,TiO_2:0.42%,CaO:40.1%,MgO:3.73%,Cr_2O_3:0.80%, MnO:7.96%,V_2O_5: 0.24%,P_2O_5:3.06%,S:0.37%,F:0.1%

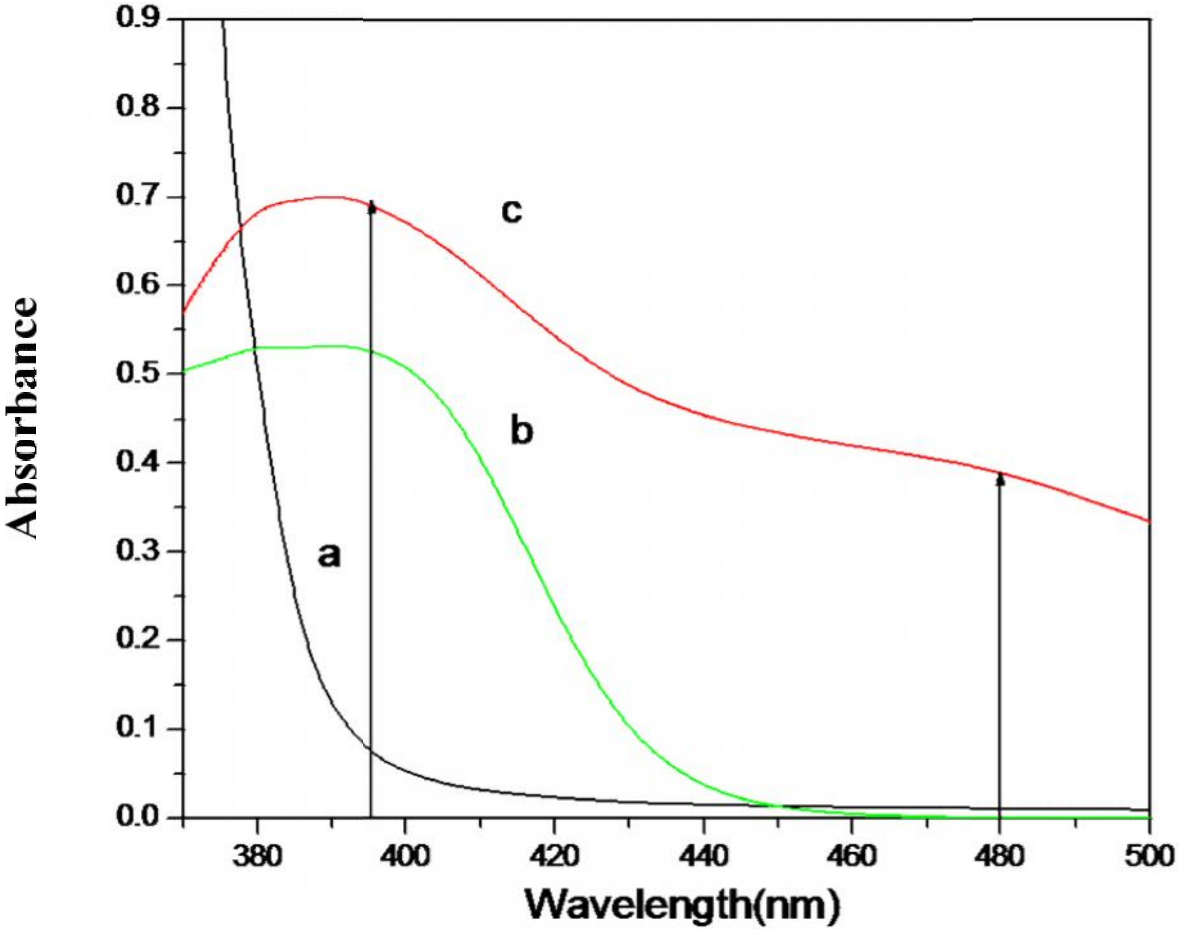

Fig. 1 Absorbance spectra of

A. 5-BHMBHBH system Vs buffer blank
B. Al(III)-5-BHMBHBH system Vs reagent blank
C. Fe(III)-5-BHMBHBH Vs reagent blank
[Al(III)]= [Fe(III)]=2.0 × 10–5 M; pH 4.0
[5-BHMBHBH] = 5 × 10–3 M

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