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Direct determination of iron in titania slag leach liquor using thiocyanate

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A spectrophotometric method has been developed for the direct determination of total iron in titania slag leach liquor containing Fe(II), Fe(III), Ti(IV), Ca(II), Mg(II) etc., using thiocyanate as a complexing agent. The iron complex absorbs at the wavelength 474-479nm. Beer-Lambert's Law obeys within the range of 0.1-0.9mg/ml of iron. Iron determination by the present method, compared with the titrimetric method, shows good agreement.

Keywords: Iron, Titania, Titania slag, Leach liquor, Thiocyanate

IPC Code: G01J3/00, C21D9/00

Introduction

Acidic leach liquor of titania slag containing mainly titanium and iron [Fe(II) & Fe(III)] finds importance in industries for producing titanium dioxide as well as iron oxide pigments. Iron determination from various sources such as ores, minerals, various biological materials are generally carried out by using gravimetric, titrimetric, iodometric, spectrophotometric methods etc[1,4]. Separation of Fe(II) or Fe(III) from solutions containing other elements have been done by complexing with various ligands followed by extracting the same through solvent extraction route. Various other reagents such as O-phenanthroline[5], 5-nitro-6-amino-1-10-phenanthroline[6], 2, 2'-dipyridine[7], 5-bromo-salicylaldehyde thio-semicarbazone (5-BAST)[8] etc. have been used for the determination of iron(II), whereas for the determination of iron(III), piroxicam[9], thiocyanate and EDTA etc[4]. have been used. Thiocyanate is known to form a series of complexes with Fe(III) depending on thiocyanate concentration, whereas Fe(II) does not[4]. Subsequently, thiocyanate has been reported to form complex with iron(II)[10].

Determination of iron from acidic leach liquor of titania slag is generally done by titrimetric method[1]. After complete separation of iron from titanium and other ions, it can be determined by atomic absorption spectrometry[2]. Alternatively, direct determination of iron is possible by using inductively coupled plasma emission spectrometry[3]. Among these processes, inductively coupled plasma emission spectrometry is costly, and atomic absorption spectrometry requires the complete separation of iron from other elements. Besides this, spectrophotometric method is used for analyzing total iron either as Fe(II) or Fe(III) in solutions obtained from grape leaves, multivitamin capsules, human blood, pharmaceutical formulations, plant materials, food stuffs and ores[4-8].

This paper describes a method for direct determination of total iron as iron(III)-thiocyanate complex in the leach liquor containing Fe(II) or Fe(III) with other impurities such as Ti(IV), Ca(II), Mg(II) etc.

Experimental Procedure

Leach liquor was prepared from acid leaching of the titania rich slag using HCl. All chemicals were of analytical grade. A microcomputer based Chemto-250 UV-Visible spectrophotometer equipped with quartz cells was used for all the spectral measurements. An ELICO Li-120 digital pH meter was used for pH adjustments. A stock standard solution of Fe(III) was prepared by dissolving a weighed amount of $FeCl_3$ in water. The iron concentration in the solution was determined by titrimetric method. Sodium thiocyanate solution (2*M)* was prepared with distilled water.

To leach liquor [0.54mg iron, along with Ti(IV), Ca(II), Mg(II) etc.], 3-4 drops of 5% HNO_3 was added to convert all the iron to iron(III). Then 2*M* sodium thiocyanate solution (1-5 ml) was added and total volume was made up to 50 ml. Similar experiment was carried out without thiocyanate. Absorbance spectra were taken for Fe(III) thiocyanate complex at

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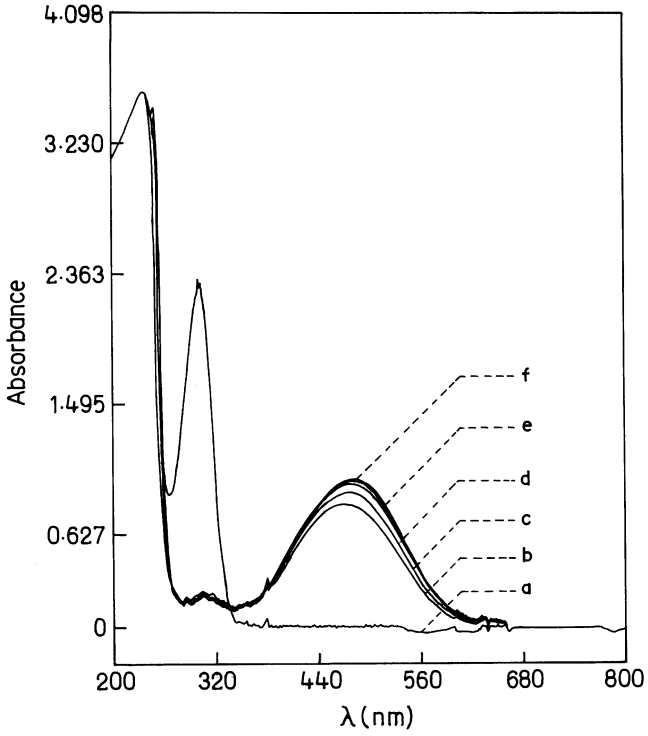

Fig. 1 — Spectra of iron-thiocyanate complex formed in standard $FeCl_3$ solution

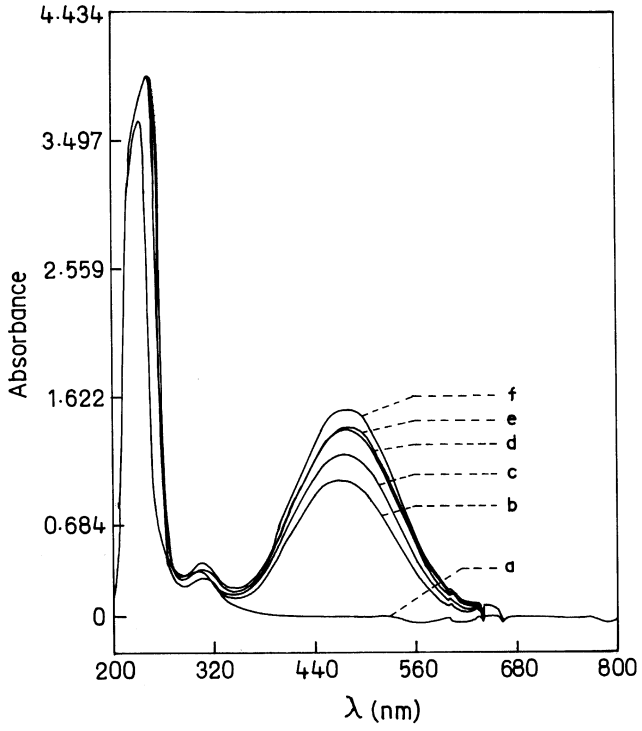

Fig. 2 — Spectra of iron-thiocyanate complex formed in leach liquor

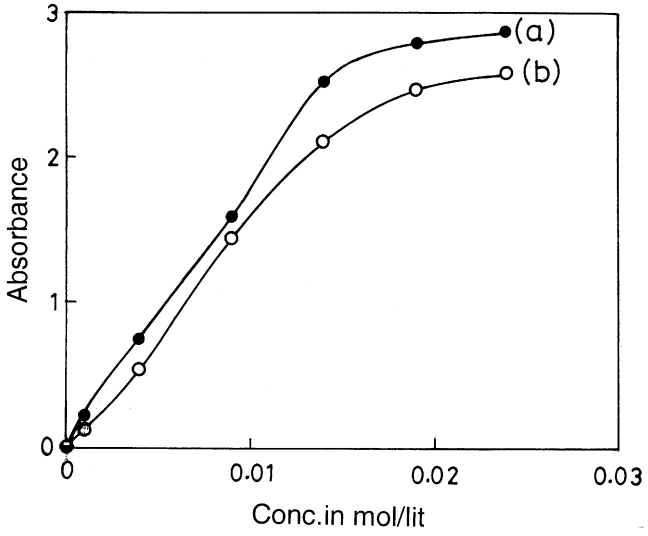

Fig. 3 — Beer-Lambert's plot: (a) Standard $FeCl_3$ solution; and (b) Leach liquor

different thiocyanate concentration and compared with the literature (λ = 474-479 nm) and also with the experimental results obtained from the standard iron (III) solution (vide supra).

Keeping the optimum thiocyanate concentration, iron concentration was changed from 0.541 to 2.164 mg/ml. The absorbance was measured at λ = 479nm and Beer- Lambert's plot for the same was obtained and compared with that of the standard solution. Using above plot, total iron concentration from unknown leach liquor was obtained and compared with that of the titrimetric method.

Results and Discussion

The spectra of iron-thiocyanate complex for standard $FeCl_3$ at various thiocynate concentration (1-5 ml) shows absorption maxima in the range of λ = 474-479 nm (Fig.1). This may be due to formation of a red-brown colour complex (log β = 5.3).

$$Fe^{3+}(aq) + SCN^{-}(aq) = Fe(SCN)^{2+}(aq) \quad \ldots(1)$$

Colourless colourless red-brown complex

Similarly, Fig. 2 (a) shows the absorption spectra of the leach liquor whereas Fig 2(b-f) shows absorption spectra of the thiocyanate complex for leach-liquor at various thiocyanate concentrations (1-6 ml) of 2*M* thiocyanate. From the same, optimum thiocyanate concentration was found to be 0.019 *M* for 0.009 *M* of iron.

Beer-Lambert's plot was obtained by varying the iron concentration both in the standard $FeCl_3$ [Fig. 3(a)] as well as iron present as Fe(III) in leach liquor [Fig-3(b)].

Using Beer-lambert's plot, it is found that Beer's law is obeyed up to Fe(III) concentration of 0.017 *M* for the standard whereas it obeys up to 0.001-0.016 *M* for leach liquor. The results, compared with titrimetric method, show values within the error +0.01. Further increase in thiocyanate concentration results decrease in the absorbance value.

Conclusions

This method can be applied to any synthetic solution or any leach liquor containing iron in addition to other ions, which has lots of industrial application for direct determination of iron in presence of other ions. So in this method, to the leach liquor containing iron (0.001-0.017 *M*), Ti (0.009-0.031 *M*), Ca and Mg (0.004-0.006 *M*), etc., 3-4 drops of 5% HNO_3 is added followed by 0.019 *M* sodium thiocyanate solution. Absorbance value of yellow-orange colour complex was measured at 474 nm. The total iron is calculated by comparing absorbance value of this solution with that of the standard iron solution. The proposed method has an advantage of its simplicity and reliability.

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