DETERMINATION OF FERROUS IRON IN CEMENT SAMPLE- A COLORIMETRIC STUDY

[1]M.Shanti, [2]O.Sujana, [3] M.Sreevalli
[1] Asst Prof, [2]Asst Prof , 3Asst Prof
[1]Basic Sciences,
[1]G.Narayanamma Institute of technology &Science, Hyderabad, India

Abstract : A simple, rapid and sensitive spectrophotometric method was developed for the determination of trace amounts of iron using as a chromogenic reagent. The proposed method was based on the reaction of iron with ammonium thiocyanate forming a complex having a maximum absorption at 450 nm. Iron was estimated colorimetrically , the results were found to be reproducible and the method was economical and less time consuming. The method was successfully applied to cement sample and results were found to be accurate and precise as the more sophisticated colorimeter was commonly used for ferrous determination.

IndexTerms - Cement, ferrous iron, ammonium thiocyanate,colorimeter.

I. INTRODUCTION

Cement, as a binding material, is a very important building material[1,2]. Almost every construction work requires cement. Therefore, the composition of cement is a matter of great interest to engineers[3,4,5]. For understanding cement composition, one must know the functionality of Cement ingredients[6,7]. By altering the amount of an ingredient during cement production, one can achieve the desired cement quality[8,9]. Ability to calculate compositions of cements in terms of the amounts of the main compounds present provided a valuable new tool for explaining, or predicting, differences in engineering performance among Portland cements[10].

1.1Composition of Cement

There are seven major ingredients of cement.The general percentage of these ingredients in cement is given below in Table 1.1:

Table 1.1

Ingredient	Percentage in cement
Lime	60-65
Silica	17-25
Alumina	3-8
Magnesia	1-3
Iron oxide	0.5-6
Calcium Sulfate	0.1-0.5
Sulfur Trioxide	1-3

Iron oxide: Chemical formula is Fe_2O_3.

- Iron oxide imparts color to cement.
- It acts as a flux.
- At a very high temperature, it imparts into the chemical reaction with calcium and aluminum to form tricalcium alumino-ferrite.
- Tricalcium alumino-ferrite imparts hardness and strength to cement

Colorometric measurements were made using a colorimeter and samples were analysed at 450 nm .The basis of spectrophotometric methods is the simple relationship between the absorption of radiation by the solution and the concentration of species in the solution. When monochromatic light passes through a transparent medium (coloured solution) the rate of decrease in intensity with the concentration and thickness of the medium is directly proportional to the intensity of the light .In order to determine a species or analyte in the solution spectrophotometrically, it is usually converted into a colored complex. Ammonium thiocyanate yields a blood red colour with ferrous iron and the colour produced is stable in nitric acid medium. The sample of cement was analysed with certain colouring reagent and optical density of the coloured compound is measured at 450 nm since

the maximum absorption was at 450nm , hence all measurements were made at 450 nm. Its optical density is measured in a colorimeter and the concentration of ferrous iron is found from the standard calibration curve.

II.Methodology:

Dissolve the given ferrous ammonium sulphate (0.7022 gms) in 100 ml of water and add 5 ml of 1 : 5 H_2SO_4 followed by dil. $KMnO_4$ solution through burette until light-pink colour appears. Dilute the solution to 1 L such that 1 ml of solution contains. 0.1 mg of Fe^{+2}. From the above solution, take separately 1,2,3,4,5 ml into separate 100 ml standard volumetric flasks. Add 1 ml of nitric acid and 5 ml of 40% ammonium thiocyanate solution to all the above samples to get blood red colour and make up the solutions to the mark by adding distilled water. Before recording the optical densities values for the above prepared solutions filter selection is done to know the maximum absorption in table 2.1. Now measure the optical densities of all the solutions using colorimeter table 2.2 and plot a graph by taking amount of ferrous iron on x-axis and optical density on the y-axis. The curve obtained is called standard calibration curve in figure 2.1

2.1Dissolution of sample

Weight out 0.1 gm of the cement sample into a beaker. Add 5 ml of water and stir with a stirrer. Add few drops of concen HCl through the walls of the beaker and stir again. Heat the mixture until the moisture is evaporated. Then add 20 ml of distilled water and dissolve the content and make up the solution with distilled water to 100 ml. Shake well for uniform concentration.

2.2Development of Colour

Pipette out 10 ml of above solution, into 100 ml std. flask add 1 ml of con HNO_3 and then add 5 ml of 40% NH_4SCN with a Burette. Make up the solution upto the mark with distilled water. Now take the sample solution into colorimeter tube and measure the O.D. (optical density) using the photo colorimeter. From the O.D. we can measure the concentration of Fe^{2+} from the calibration curve. The calibration curve is as follows in figure 2.1

Table 2.1 Filter selection:

Filter Number	Absorbance
45	0.52
47	0.41
51	0.34
52	0.29
54	0.21
57	0.12
67	0.01

Table 2.2 Absorbance of solutions with various concen of Ferrous(Fe^{+2})

S.No	Concentration of Fe^{+2} in mg	Absorbance
1	0.2	0.18
2	0.4	0.39
3	0.6	0.58
4	0.8	0.81
5	1	0.98

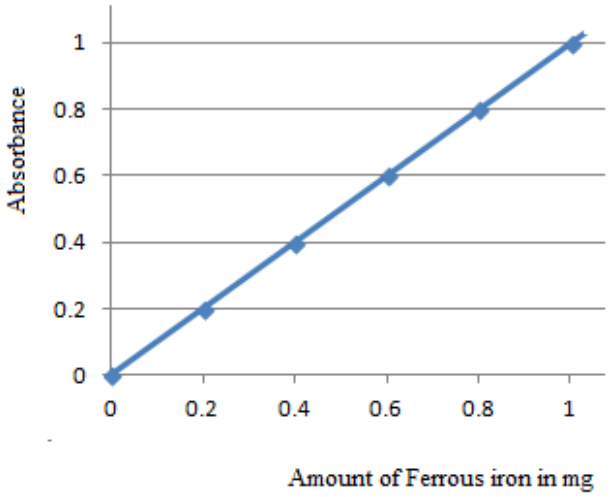

Figure 2.1 CALIBRATION CURVE

III CONCLUSION

In the present study an attempt was made to develop a simple and accurate procedure for ferrous iron analysis. Analysis of ferrous in cement was successfully performed colorimetrically and an attempt was made to study the concentrations of ferrous in cement sample by using the reagent ammonium thiocyanate .The results were found to be accurate and reproducible .From the result obtained in this research work, it can be deduced that the level of iron in selected cement sample was found in the specified range .

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