

## Micellar Aided Chromogenic Reagents for Simultaneous Spectrophotometric Determination of Aluminium and Iron

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**Abstract:** Simple and novel spectrophotometric method is described for simultaneous determination of aluminium and iron. The method is based on the metal ions - complexes formed by aluminium and iron with mixed chromogenic reagents of 1, 10 phenanthroline and 8-hydroxyquinoline (oxine). The derivatives have absorption maxima at 400 nm and 510 nm respectively in the presence of cetyltrimethyl ammonium bromide (CTAB) as micellar media (surfactant) maintained at pH 5. Reaction conditions were optimized and the linear dynamic ranges for determination of aluminum and iron were found be 0.8 – 12.0 µg/mL and 0.6 – 8.0 µg/mL respectively. The standard deviation (S.D) and coefficient of variation (CV) for the simultaneous determination was 0.018 and 0.45% for aluminium and 0.03 and 1.5% for iron (II). The recoveries were between 102 % and 106 %, 101.2 % and 104 % for aluminium and iron respectively at 95 % confidence level ( $p \geq 0.05$ ). The proposed method was successfully applied to the assay of aluminium and iron in rock minerals, lubricating oil and water samples. The results and paired t-test for the analyzed samples were found to be in satisfactory agreement (shows no significant difference) with those acquired by the flame atomic absorption spectrophotometric (FAAS) technique.

**Keywords:** Aluminum, Chromogenic reagents, Iron, Micellar media, spectrophotometry.

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### I. Introduction

The determination of trace amounts of aluminium and iron is important in industry, food products, potable water, metallurgy, environmental and biochemical materials. These metals have important roles in human health, environment and industrials <sup>[1]</sup>. Aluminium is a non-essential element to which humans are frequently exposed <sup>[2]</sup>. Aluminium is widespread throughout nature, air, water, plants and consequently in all the foods because of its uses. The metal enters the human system mainly through foods, drugs, cosmetics, drinking water and beverages <sup>[2]</sup>.

Aluminium, present in most industrial effluents discharges and in domestic wastes and wastewater, is also found in over the counter medicines, such as antacids and buffered aspirins, is used as a food additive, and is found in a number of topically applied consumer products such as antiperspirants, first aid antibiotic and antiseptics <sup>[3]</sup>. All these findings cause alarming concerns in public health, demanding accurate determination of this metal ion at traces and sub-trace levels. Iron, is a trace essential element for human bodies. Lack of this essential element can induce some diseases such as anaemia, while it is harmful and deleterious when taken in large quantity. Aluminium and iron are usually present together in both natural and artificial materials and samples <sup>[4]</sup>, hence the need for analytical techniques capable of single or simultaneous multi-component quantitative determination of the metals in the presence of each other.

Several techniques, such as ion chromatography, liquid-liquid extraction with atomic absorption spectrophotometry, atomic fluorescence spectrophotometry, X-ray fluorescence spectrophotometry, graphite furnace atomic absorption spectrophotometry, inductively coupled plasma atomic emission spectrophotometry <sup>[5]</sup>, derivative spectrophotometry and chemometrics <sup>[6]</sup> have been applied for the simultaneous determination of aluminium and iron ions in different samples. All these techniques require costly instrumentation for quantitation of these chemical species. A few UV-Vis spectrophotometric applications have been developed for the simultaneous determination of aluminium and iron using chromogenic reagents. UV-Vis spectrophotometry, one of the most widely used analytical methods, has been an attractive method for its rapidity, simplicity, low cost and broad applications.

Mixed chromogenic reagents, mixtures of organic compounds chromogenic and complexing agents which do not undergo any reaction or alteration with each other and are specific or selective towards different metallic ions whether ordinarily or at controlled conditions such temperature, pH or solvent, have been proposed for spectrophotometric multi-component analysis (MCA) of metal ions <sup>[7]</sup>. The limitation on the use of such systems usually arises from the necessity for finding compromise conditions with respect to the existence of different metal complexes <sup>[8]</sup>. In spite of this, the use of mixed reagent systems can be considered as an alternative means to extend the applicability of spectrophotometric multi-component analysis (MCA) of metal ions.

The organized molecular assemblies such as micelles are used in spectroscopic measurements due to their possible effects on the systems of interest. In the field of metal ion complex, at a concentration above critical micelle concentration (CMC) micelles form a ternary complex with advantageous properties, such as hyperchromic and bathochromic shifts, that can modify the sensitivity of the method by affecting the interferences and matrix effects<sup>[9]</sup>. The ability of micellar systems to solubilize slightly soluble or even insoluble complexes has been used to enhance the analytical merit of given methods<sup>[10]</sup>. Most organic solvents that are used as extraction solvents can be classified as toxic and environmental pollutants, and some have been listed as carcinogenic by the US Environmental Protection Agency (USEPA)<sup>[11]</sup>. Application of micellar systems avoids the solvent extraction steps which are necessary following the formation of slightly soluble complexes in the absence of micelles.

In this present work, a micellar aided mixed chromogenic reagents of 8-hydroxyquinoline and 1, 10 phenanthroline is hereby reported for the first time for the simultaneous determination of aluminium and iron by UV-Vis spectrophotometry.

## II. Materials And Methods

### 2.1. Apparatus

A UV-VIS spectrophotometer (Spectronic D20+) and a Metro-Ohm pH meter were used for the measurements of absorbance and pH, respectively. A Perkin Elmer A Analyst 200 model atomic absorption spectrophotometer with deuterium arc background correction was used for comparing the results.

### 2.2. Reagents and Solutions

All chemicals used were of analytical grade. Doubly distilled deionized water was used in the preparations of solutions and used throughout. Stock solutions were kept in polypropylene bottles containing 1 mL concentrated nitric acid.

Cetyltrimethyl ammonium bromide (CTAB) solution (0.1 and 1%). Appropriate amounts of pure Cetyltrimethyl ammonium bromide salt (Merck) were dissolved in 100 ml standard flask and made up to mark with doubly distilled deionized water.

Ascorbic acid solution (0.1M). 0.9905g of ascorbic acid salt was dissolved in doubly distilled deionized water in a 50 ml standard flask and made up to mark. This solution was prepared daily and refrigerated when not been used immediately. This served as reducing agent and was used to reduce all iron (III) species to iron (II).

1, 10 phenanthroline solution (0.05M). Prepared by dissolving 0.2937g in doubly distilled deionized water in a 25 ml standard flask. This solution was refrigerated when not been used.

8-hydroxyquinoline solution (0.07M). This was prepared by dissolving 1.0g of purified 8-hydroxyquinoline salt (Sigma-Aldrich) in 2M acetic acid first, then transferred into a 100 ml standard flask and made up to mark with the acetic acid solution.

Sodium acetate buffer, 0.1M (pH 5). 28.82 ml of 1M acetic acid and 273.3 ml of 0.3M of sodium acetate were mixed in a 1000ml standard flask and made up to mark.

Aluminium Standard solution (1000 µg/mL). Stock aluminium containing 1000 µg/mL Al(III) was prepared by dissolving appropriate amounts of pure analytical grade of  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (Merck) in doubly distilled water. More dilute standard solutions were prepared from this stock solution, as and when required.

Iron Standard solution (1000 µg/mL). Stock iron solutions containing 1000 µg/mL Fe (III) was prepared by dissolving appropriate amounts of pure salt ( $\text{Fe}(\text{NO}_3)_3 \cdot 10\text{H}_2\text{O}$ ) in 100 ml of doubly distilled deionized water. The working solutions were prepared just before use by dilution of the standard solution with redistilled deionized water.

Mixed chromogenic reagents (1:1). Prepared by mixing appropriate volumes of 1, 10 phenanthroline and 8-hydroxyquinoline solutions and was sonicated for 15 minutes.

Other solutions – Solutions of inorganic ions and complexing agents were prepared from their Analar grade.

### 2.3. Optimization of Analytical variables

Analytical variables and factors such as pH, temperature, amount and concentrations of micellar media and excess mixed chromogenic reagents. Stability studies, reproducibility and recovery studies were carried to determine the robustness of the present method.

### 2.4. Procedure

In 25.0 mL volumetric flasks, appropriate volumes of Al (III) and Fe (III) solutions, 2.0 mL of buffer solution (pH = 5), 1.0 mL of 0.1M ascorbic acid, 2.0 mL of cetyltrimethyl ammonium bromide (CTAB) and 2.0 mL of mixed chromogenic reagents were added, and the solution was made up to the mark with deionized-distilled water. The final concentration of Al (III) and Fe (II) should be between 0.60-8.00 and 0.8-12 µg/mL respectively, in order to have absorbance within the linear calibration ranges. For each measurement, about 5.0 mL of the above solution was transferred to a spectrophotometric cell and the absorbance were recorded after 2 minutes at 510 nm for iron and 15 minutes at 400 nm for aluminium upon addition of the mixed reagents to the binary solution. Absorbances were recorded against a reagent blank for aluminium and iron respectively. The method of standard addition was used to determine the quantities of Al and Fe (II) in samples aliquots. Determinations of aluminium and iron concentrations in unknown samples were also done using concurrently prepared calibration graphs.

## III. Results And Discussion

Studies had shown that 8-hydroxyquinoline (oxine) could react with aluminium ion ( $\text{Al}^{3+}$ ) and that 1, 10 phenanthroline reacts with iron (II) to form coloured complexes, that could be monitored spectrophotometrically [12][13]. The absorption spectra of both complexes in micellar media show some level of resolution, and the wavelengths of absorption maxima of the two complexes are sufficiently discrete occurring at regions of relatively little mutual spectral interference (Fig.1).

### 3.1. Absorption Spectra

The absorption spectra of Al - 8-hydroxyquinoline complex in a 2M acetic acid medium and Fe (II) - 1, 10-phenanthroline in micellar system were recorded. The absorption spectra are symmetric curves with maxima occurring at 400 nm and 510 nm respectively (Fig.1 and 2). The reaction mechanisms for these complexes have been reported in the literature [14][15]. In all instances, measurements were made at 400 nm and 510 nm against reagents blank.

### 3.2 Optimization of Analytical Variables

Various factors and conditions were optimized using the method of one at a time. These conditions were determined (Table 1) and were used to validate the applicability of the proposed method in the determination of iron and aluminium in real and complex environmental samples.

Effect of pH – The influence of pH values on the spectra of each complex at constant concentrations (2.0 and 4.0 g/mL of  $\text{Fe}^{2+}$  and  $\text{Al}^{3+}$  ions respectively) was investigated. There were no significant changes in the absorbance of the Fe (II) - 1, 10 phenanthroline complex in the pH range of 3-8, and for the Al(III) - 8-hydroxyquinoline complex in the pH range of 3-7 (Fig. 3). In order to achieve higher sensitivity and stability, pH 5 (acetate buffer) was selected as an optimum pH for simultaneous determination of aluminium and iron.

Effect of amount and concentration of micellar media - various concentrations and volumes of cetyltrimethyl ammonium bromide (CTAB) surfactant were investigated on the absorbance profile of the complexes. From the results obtained 2 mL of 1% CTAB micellar solution gave the maximum absorbance (Fig. 4).

Effect of excess Mixed Chromogenic Reagent - The effect of the different amounts of mixed chromogenic reagent on the absorbance of the complexes was studied in the range of 0.5.0 to 5.0 mL of 0.07 M 8-hydroxyquinoline and 0.05M 1, 10 phenanthroline mixed in equal proportion (1:1). From the result obtained (Fig. 5), 2 mL of the mixed reagent was considered suitable to ensure complete complexation.

Effect of Temperature – Absorbance were recorded at different temperatures, 20 - 70°C for the mixed complexes using standard procedure. The absorbances at the dual wavelengths were unaltered between the temperature range of 25 - 45 °C (Fig. 6). Therefore all measurements were carried out at room temperature of 25±5 °C.

Effect of Time (Stability studies) - The stabilities of the complexes were studied differently and the mixture of the metals with the chromogenic mixed reagent. The Fe (II) - 1, 10 phenanthroline complex develops its colour immediately with maximum absorption within 5-10 minutes, after which it became quite stable up to 48 hours in the micelle medium. The Al (III) – 8-hydroxyquinoline complex attained maximum absorption after 15-20 minutes and found to be stable for a period of 72 hours.

Effect of foreign ions - For interference study, Cu (II), Cu (I), Ni (II) and Fe (III), Sulphates, Phosphates ions were added to the mixture prior to the determination. Cu (II) caused slight interference at a

concentration of 100 µg/mL during Fe (II) determination Cu (I) did not interfere even at a concentration of 1000 µg/mL. However, in the determination of the Al ions, Fe (III) ion, prepared from ferric chloride (FeCl<sub>3</sub>.5H<sub>2</sub>O) gave high interference at a concentration as low as 10 µg/mL, and this interference by Fe (III) is, however, avoided by reducing the Fe (III) species to Fe (II) by ascorbic acid which is a good reducing agent. No interference was however observed due to the presence of sulphate, phosphate and fluoride ions.

#### **Effect of Iron on Aluminium determination.**

The effect of iron on aluminium determination was determined analyzing binary mixtures of iron and aluminium solutions (aluminium solution concentration is left fixed while iron solutions concentration was varied). The results (Table 2) show that the level of deviation of the aluminium found from that present tend to increase with increase in the concentration of the iron species.

#### **Effect of Aluminium on Iron determination.**

The effect of aluminium on iron determination was determined analyzing binary mixtures of iron and aluminium solutions (iron solution concentration was left fixed while aluminium solutions concentration was varied). The results (Table 3) show that the level of deviation of the iron found from that present tend to increase with increase in the concentration of the aluminium species.

### **3.3 Precision and Accuracy**

In order to check the accuracy of the method, several mixtures prepared using different concentration ratios of Al (III) and Fe (II) were analyzed using the proposed method. The metal ions were determined in five replicate mixtures, the standard deviation (SD) and coefficient of variation (CV) were found to be 0.018 and 0.45% for Al (III) and 0.03 and 1.5% for Fe (II) as shown in Table 6. The good agreement between amount present and amount found for different compositions of the binary mixture as shown in Table 4 and 5 are indicative of satisfactory accuracy and the precision of the method is satisfactory as well.

### **3.4 Recovery studies of the proposed method**

#### **3.4.1 Recovery of Aluminium and Iron from prepared binary mixtures of aluminium and iron.**

Mixtures of varying concentration of the analyte results (Table 4) showed that good recoveries were achieved in all mixture solutions. The various mixtures, recoveries were within the ranges of 97.6 -98.9% and 95-105% for Al and Fe respectively. The mean recoveries were 96.50 and 100.03% for Al and Fe respectively. These recoveries further indicate the validity of the proposed method.

#### **3.4.2 Recovery in spiked water samples**

Tap water samples were spiked with known concentration of aluminium and iron solutions. The recovery test results (Table 5) show recoveries in all spiked samples to range between 102-106% and 101.5-104% for aluminium and iron respectively. The recoveries were all above 100% which could be due to the presence of some levels or residues of chemical and coagulants such as alum and ferric chloride used in water purification. The recovery results did not show any significant difference ( $p > 0.05$ ) between the concentrations of analytes in spiked samples and the added analyte concentrations.

### **3.5 Reproducibility of the Proposed Method**

Employing the optimal conditions, simultaneous determination of Al<sup>3+</sup> and Fe<sup>2+</sup> was carried out in binary mixtures containing 4.0 µg Al<sup>3+</sup>/mL and 2.0 µg Fe<sup>2+</sup>/mL of the ions. To check the reproducibility of the method, five replicate experiments were performed. The mean concentrations of Al<sup>3+</sup> and Fe<sup>2+</sup> determined were 4.004 and 1.996 µg/mL respectively. Standard deviation and the coefficient of variation (CV) were 0.018 and 0.45%, 0.030 and 1.50% for Al<sup>3+</sup> and Fe<sup>2+</sup> ions, respectively. The low coefficient of variation and standard deviations amongst replicate determinations indicated no significant variation and hence shows that the method has very good reproducibility (Table 6). Since the mean concentrations of Al and Fe determined gave close approximation to the concentration of the Al and Fe (4.0 and 2.0 µg/mL) present in the prepared binary mixtures.

### **3.6 Applications of the proposed method**

The reliability and the applicability of the proposed method were validated by applying the method in the determination of aluminium and iron in a series of real samples - mineral ore, lubricating oil (spent) and lagoon water samples were analyzed in triplicate. The results were compared to the results obtained for the same samples by the AAS method, which employs a different principle from the UV-VIS spectrophotometric method. The results obtained are in close agreement (Table 7). The results obtained from the two methods were

subjected to the statistical test of significance. The paired t-test showed no significant difference ( $p \geq 0.05$ ) exist between the results obtained from the two methods.

Mineral ore and the spent lubricating oil samples with complex matrices were analyzed for aluminium and iron using the method of standard addition. The method of standard addition has been frequently employed in the determination of analytes in complex environmental sample<sup>[15]</sup>. This was to avoid the case of interference from the sample matrices.

#### IV. Figures And Tables

**Table 1. Selected analytical parameters obtained from optimization.**

Parameter	Studied range	Selected value
Wavelength (nm)	300-700	400 (Al) and 510 (Fe)
pH	3-8	5
Time/minute	0-72	5 (Fe) and 15 (Al)
Mixed reagent (mL)	1-5	2
Micellar solution (CTAB)		
Concn (%)	0.1-2.0	1
Vol (mL)	1-3	2
Linear range( $\mu\text{g/mL}$ )	0.1-12	0.6-8 (Fe) and 1-12 (Al)
Mean recovery (%)	-	100.03 (Fe) and 96.5(Al)
Standard deviation	-	0.030 (Fe) and 0.018 (Al)
Coefficient of variation	-	1.5 (Fe) and 0.45 (Al)
Regression coefficient ( $R^2$ )	-	0.9972 (Fe) and 0.998(Al)
Interferences		Cu (II) and Fe (III), $\text{SO}_4^{2-}$ , $\text{PO}_4^{3-}$

**Table 2. Effect of Iron on Aluminium Determination (Aqueous standards, 1.00  $\mu\text{g Al/mL}$ ).**

Iron added ( $\mu\text{g/mL}$ )	Aluminium found ( $\mu\text{g/mL}$ )	Deviation, %
0	1.00	0
1.0	1.02	+2
3.0	1.03	+3
5.0	1.06	+6

**Table 3. Effect of Aluminium on Iron Determination (aqueous standards, 1.00  $\mu\text{g Fe/mL}$ ).**

Aluminium added ( $\mu\text{g/mL}$ )	Iron found ( $\mu\text{g/mL}$ )	Deviation, %
0	1	0
1.0	1.008	+0.8
5.0	1.023	+2.3
10.0	1.024	+2.4

**Table 4. Recovery Studies of the proposed method for prepared mixture of the metal ions**

Amount present ( $\mu\text{g/mL}$ )		Found ( $\mu\text{g/mL}$ )		Recovery (%)	
Al	Fe	Al	Fe	Al	Fe
5.00	3.00	4.88	2.85	97.6	95.0
1.00	1.00	0.98	1.05	98.0	105.0
2.50	6.00	2.47	6.01	98.8	100.1
Mean Recovery				96.5	100.1

**Table 5. Result of Recovery Studies of the proposed method for spiked samples**

Sample	Amount added ( $\mu\text{g/mL}$ )		Amount recovered ( $\mu\text{g/mL}$ )		Recovery (%)	
	Al	Fe	Al	Fe	Al	Fe
1*	1.00	1.00	1.06	1.04	106	104
2**	2.50	2.00	2.55	2.03	102	101.5

\*Spiked Al (1.00  $\mu\text{g/mL}$ ) and Fe (1.00  $\mu\text{g/mL}$ ). \*\*Spiked Al (2.50  $\mu\text{g/mL}$ ) and Fe (2.00  $\mu\text{g/mL}$ )

**Table 6. Reproducibility of the proposed method.**

Amount present ( $\mu\text{g/mL}$ )		Found ( $\mu\text{g/mL}$ )	
Al	Fe	Al	Fe
4.00	2.00	4.01	1.98
4.00	2.00	3.98	2.00
4.00	2.00	4.02	1.97
4.00	2.00	3.99	2.02
4.00	2.00	4.02	2.01
<b>Mean (found)</b>		<b>4.004</b>	<b>1.996</b>
<b>Standard Deviation</b>		<b>0.018</b>	<b>0.030</b>
<b>RSD/CV (%)</b>		<b>0.45</b>	<b>1.50</b>

**Table 7. Aluminium and Iron in real samples by the proposed method and comparison with other method.**

Sample	Found ( $\mu\text{g/mL}$ ) <sup>‡</sup>			
	Proposed Method		AAS	
	Al	Fe	Al	Fe
Lagoon water sample	$1.06 \pm 0.013$	$3.42 \pm 0.052$	$0.996 \pm 0.021$	$3.592 \pm 0.01$
Lube Oil sample	$3.2 \pm 0.016$	$5.02 \pm 0.023$	$3.31 \pm 0.018$	$4.98 \pm 0.030$
Rock sample <sup>#</sup>	$2.16^{\pm} \pm 0.028$	$12.22^{\pm} \pm 0.182$	$2.15 \pm 0.022$	$12.02 \pm 0.23$

<sup>‡</sup> Mean value of three replicate determinations      <sup>#</sup> Measured in  $\mu\text{g/g}$  (mg/kg)

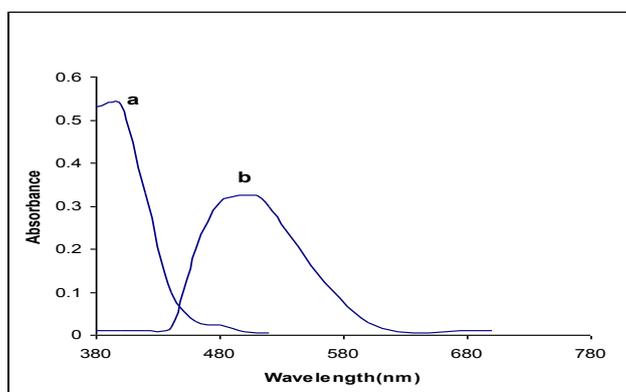


Fig. 1. Absorption spectra of (a)  $4.0 \mu\text{g mL}^{-1}$  of Al(III), (b)  $2.0 \mu\text{g mL}^{-1}$  of Fe(II) with mixed reagents at pH 5.

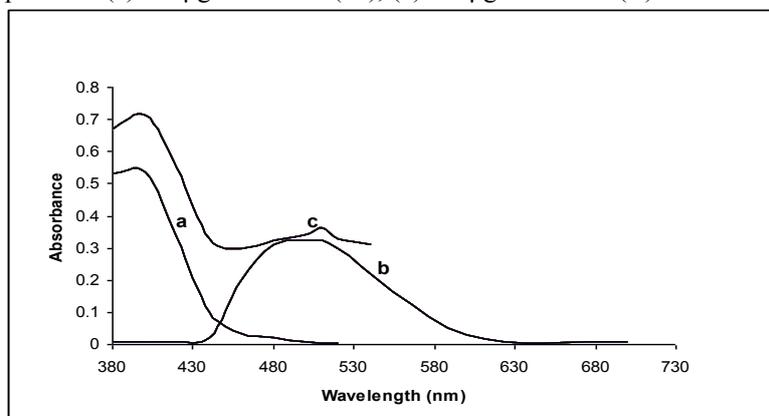


Fig. 2. Absorption spectra of (a)  $4.0 \mu\text{g mL}^{-1}$  of Al(III), (b)  $2.0 \mu\text{g mL}^{-1}$  of Fe(II), (c) mixture of  $4.0 \mu\text{g mL}^{-1}$  Al(III) and  $2.0 \mu\text{g mL}^{-1}$  Fe(II) with mixed reagents at pH 5.

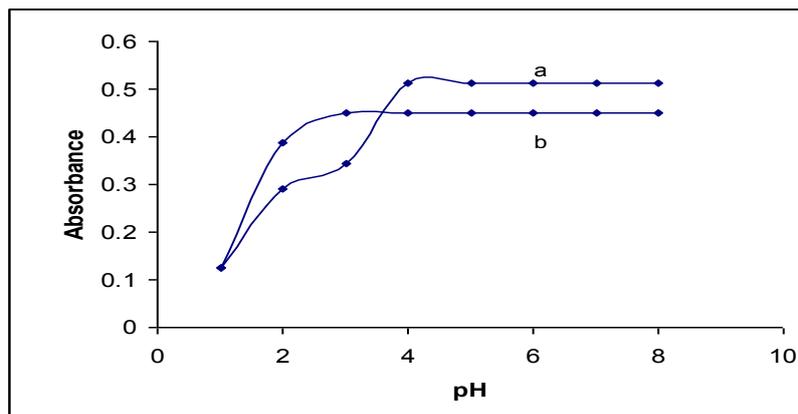


Fig. 3. Effect of pH on the absorbance of (a) Fe<sup>+2</sup>-mixed reagents and (b) Al<sup>+3</sup>- mixed reagents complexes.

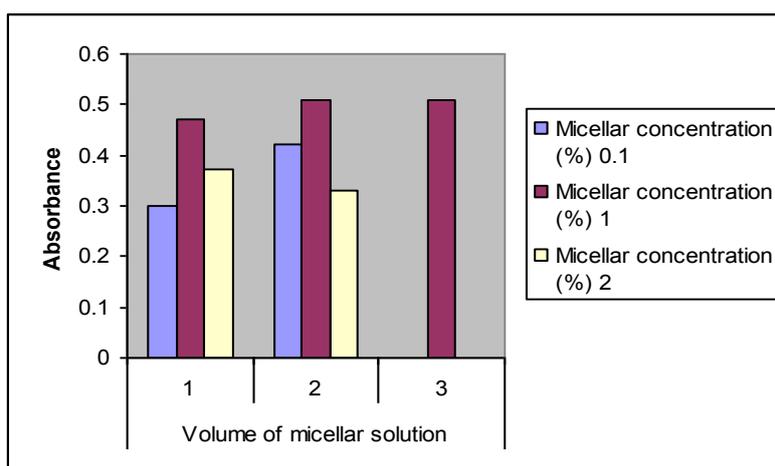


Fig. 4. Effect of amounts of micellar solution on derivatives.

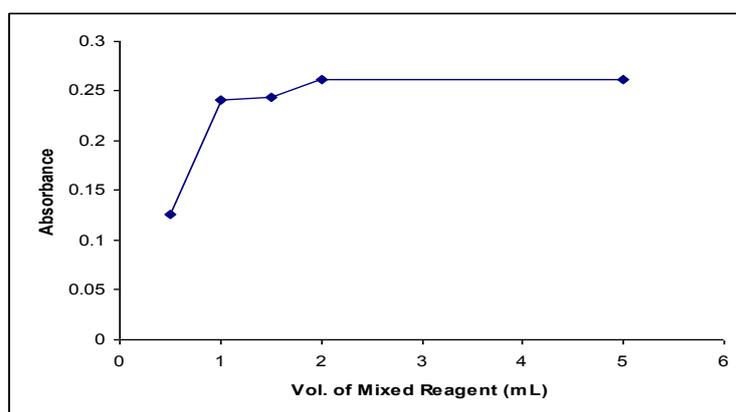


Fig. 5. Effect of amount of mixed reagent (0.069M oxine and 0.05 M phenanthroline) on absorbance of derivatives.

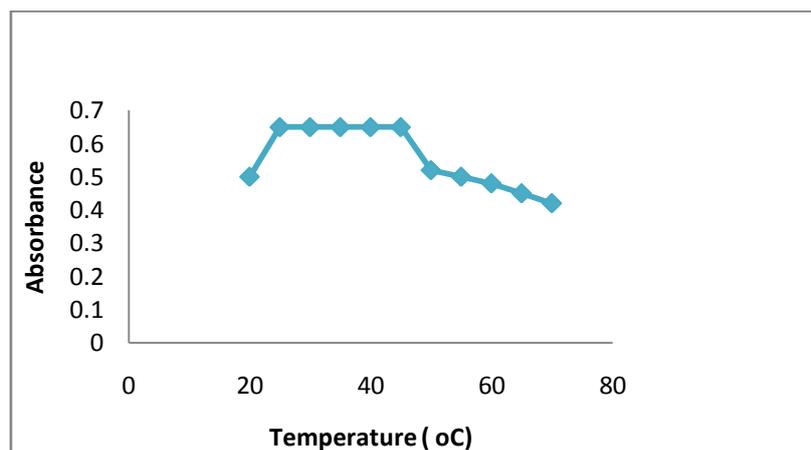


Fig. 6. Effect of temperature on the absorbance of Al and Fe derivatives.

### V. Conclusions

The determination of aluminium and iron in real samples (mineral ore, lubricating oil and lagoon water samples) and the close agreements between the present method and the atomic absorption spectrophotometric method may indicate the wide applicability and reliability of the method. This shows that the method can be well adapted for analyses of complex environmental and biological samples. The high degree of reproducibility, precision and accuracy, valuable dynamic range, simplicity and relatively short experimental period of the proposed method permit the handling of large number of samples. This research reports the simultaneous spectrophotometric determination of total iron and aluminium with the use of mixed chromogenic reagents of 1, 10 phenanthroline and 8-hydroxyquinoline, thereby increasing the array of methods that are available for the simultaneous determination of aluminium and iron. The proposed method has low cost, simplicity and green process as added advantages. Results obtained in the analysis of samples of rock minerals, spent lubricating oil, lagoon water, prepared mixtures and validation of the results with FAAS technique, which show no significant difference ( $p \geq 0.05$ ) with the proposed method are reported to demonstrate the effectiveness of the method.

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