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Studies on Synergism in the Extraction of Fe (III) with Anthranilic Acid in the Presence of Heterocyclic Amines using Spectrophotometric Technique

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ABSTRACT: Using *Solvent Extraction Technique* extraction of Fe(III) with anthranilic acid in the presence of pyridine and b-picoline into Benzene have been studied with the help of Spectrophotometric method. Mathematical computation based on equilibrium treatment for Distribution Coefficient has been employed on the experimental data. The effect of various variables such as pH of the aqueous phase, metal ion concentration, anthranilic acid concentration, and base concentration have been reported. Synergistic effect of pyridine and b-picoline in the extraction of Fe(III) with anthranilic acid have been established and the effectiveness of pyridine and b-picoline as synergist has been compared. The most probable composition of the synergistic adduct as [Fe(OH)$_2$A.2B] where A = anthranilate ion and B = pyridine or b-picoline, has been postulated on the basis of Slope analysis method.

Keywords: Solvent extraction, Synergism, Spectrophotometric method, Anthranilic acid, Pyridine, b-picoline, Slope analysis method

1. INTRODUCTION

Solvent extraction, as one of the most useful separation techniques, can be safely extended from microlevels to macro-concentrations. In solvent extraction, certain combinations of two extractants under well-defined conditions, results in the extraction of a metal ion species with a significantly enhanced efficiency compared to the additive effect observed when they are used separately. This phenomenon is referred to as Synergism. Carboxylic acids either singly [1-6] or in conjunction with some synergists such as organophosphorus compounds and nitrogen containing bases, have been used for the effective separations of metal ions by the Solvent extraction [7, 8]. Anthranilates of some metals show poor extraction into non-polar solvents like Chloroform and Benzene. However, the addition of neutral donors such as heterocyclic nitrogen containing organic bases to such systems increases the extraction significantly [9]. The present study embodies work on the extraction of Fe (III) in aqueous phase, with anthranilic acid in the presence of pyridine and $\beta - picoline$ into benzene. The heterocyclic organic base ligand 'B' is added to the species formed between the metal ion, M^{n+} and the anthranilic acid, HA. The synergistic adduct $[MA_n.B_x]_O$ moves across the aqueous-organic phase boundary into organic phase until an equilibrium is established such as:

$$M_{aq}^{n+} + nHA_{Org} + xB_{Org} \rightleftharpoons [MA_n.B_x]_{Org} + nH_{aq}^{+}$$

According to 'Distribution Law':

$$K_d = \frac{C_O}{C_W}$$

Where K_d = Distribution Coefficient, C_O = Concentration of the solute in the organic phase, C_W = Concentration of the solute in the aqueous phase.

If any type of interaction takes place between the extractable species and component in either of the two phases, instead of K_d, the Distribution ratio D is used.

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$$\text{Therefore, } D = \frac{[M]_{org}}{[M]_{aq}} = \frac{[MA_n.B_x]_{Org}}{[M^{n+}]_{aq}}$$

The concentration of Fe(III) in the aqueous phase has been determined spectrophotometrically using Sulphosalicylic acid as the colorimetric reagent and 495 nm as the working wavelength. The concentration of Fe(III) in the organic phase was determined by difference.

The equilibrium constant for extraction is given by:

$$K_{ex} = \frac{[MA_n.xB]_{org}.[H^+]^n_{aq}}{[M^{n+}]_{aq}.[HA]^n_{aq}.[B]^x_{aq}}$$

Hence,

$$\log D = \log K_{ex} + n\ log\ [HA]_{aq} + x \log\ [B]_{aq} + npH$$

Assuming that the metals do not form any other complex with excess of ligand, the mass balance for base is given by:

$$[B]_{total} = [B]_{org} + [B]_{aq} + x[MA_n.xB]_{org} + [BH^+]_{aq}$$

Hence, B_{aq} is given by,

$$[B]_{aq} = \frac{[B]_{total} - x.[MA_n.xB]_{org}}{P_B + 1 + \frac{[H^+]}{K_{BH^+}}}$$

Where P_B and K_{BH^+} are the partition coefficient and ionization constant of B and BH^+, respectively.

$$P_B = \frac{[B]_{org}}{[B]_{aq}}$$

and

$$K_{BH^+} = \frac{[H^+].[B]_{aq}}{[BH^+]_{aq}}$$

K_{BH^+} and pB values have been taken from literature [10, 11]. K_{BH^+} values for pyridine and $\beta - picoline$ are $10^{-5.15}$ and $10^{-5.63}$, respectively. The average values of partition coefficient for pyridine and $\beta - picoline$ are 1.95 and 1.85, respectively. The effect of change in variables such as pH of the aqueous phase, metal ion concentration, anthranilic acid concentration, and base concentration, keeping other variables constant, have been studied for each of the two systems – (i) Fe (III), anthranilic acid, pyridine, and (ii) Fe(III), anthranilic acid, $\beta - picoline$ by measuring Distribution ratio, D. From the plots log D vs pH, log D vs log $[ANTHRA]_{TOTAL}$, log D vs log $[B]_{TOTAL}$, using Slope analysis method, the most probable composition of the synergistic adduct has been postulated as $[Fe(OH)_2A.2B]$ where B = pyridine or $\beta - picoline$. The equilibrium constant for extraction, K_{ex} of Fe(III) with pyridine and $\beta - picoline$ have been calculated as 5.05 and 7.15, respectively. This suggests the effectiveness of pyridine and $\beta - picoline$ as synergist in the sequence as:

$$\beta - picoline > pyridine$$

2. MATERIALS AND METHOD

Chemicals used in the investigation were of AR/GR grade. Fe (III) solutions of various concentrations as per requirement, were obtained by dissolving Ferric chloride in doubly distilled water and standardization using EDTA titrations. Anthranilic acid was recrystallised and freshly prepared solutions of anthranilic acid were used throughout the investigations. Standardization of anthranilic acid solution was carried out using acid-base titration.

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In each set equal volumes (10 ml) of aqueous phase and benzene were shaken in separating funnel at room temperature (30 ± 2^oC) for 5 minutes to achieve complete equilibration. The aqueous phase contained Fe (III), anthranilic acid, and organic base pyridine or β-picoline. The ionic strength of the aqueous phase was maintained constant at 0.1M by adding Sodium perchlorate. The pH of the aqueous phase was adjusted with perchloric acid or sodium hydroxide. After equilibration, the two phases were separated and pH of the aqueous phase was determined. The pH measurements of aqueous phase before and after equilibration were made on an expanded scale pH-meter (ECIL, India). The concentration of Fe(III) in the aqueous phase after equilibration was determined spectrophotometrically by Calibration curve method using BECKMAN DU-6 SPECTROPHOTOMETER, Sulphosalicylic acid as the colorimetric reagent and 495 nm as the working wavelength. The concentration of Fe(III) in the organic phase was determined by difference.

The distribution ratio (D) and the percent extraction (%E) of the metal ion has been calculated as:

$$D = \frac{Concentration\ of\ metal\ ion\ in\ the\ organic\ phase}{Concentration\ of\ metal\ ion\ in\ the\ aqueous\ phase}$$

$$\%E = \frac{Concentration\ of\ metal\ ion\ in\ the\ organic\ phase}{Total\ metal\ ion\ concentration} \times 100$$

Analytical Procedure

In order to investigate the effect of pH of the aqueous phase on the extraction of Fe (III), the extraction studies were carried out at constant metal ion ($1.0 \times 10^{-3}M$), anthranilic acid (0.05M), and pyridine (0.25M) or β-picoline (0.5M) concentrations in the pH range 0.5-3.2.

In order to investigate the effect of metal ion concentration on the extraction of Fe (III), the extraction studies were carried out at constant anthranilic acid (0.05M) and pyridine (0.25M) or β-picoline (0.5M) concentrations and pH of the aqueous phase = 3.2, and varying the Fe (III) concentrations in the range $5.0 \times 10^{-5} - 1.0 \times 10^{-3}M$.

In order to investigate the effect of acid concentration on the extraction of Fe (III), the extraction studies were carried out at constant metal ion ($1.0 \times 10^{-3}M$) and pyridine (0.25M) or β-picoline (0.5M) concentrations and pH of the aqueous phase = 3.2, and varying the anthranilic concentration between 0.003-0.035M.

In order to investigate the effect of base concentration on the extraction of Fe(III), the extraction studies were carried out at constant metal ion ($1.0 \times 10^{-3}M$), anthranilic acid (0.025M/0.015M) concentrations and pH of the aqueous phase = 3.2, and varying the pyridine or β-picoline concentrations between 0.01-0.25M.

3. RESULTS AND DISCUSSION

3.1 EFFECT OF pH OF THE AQUEOUS PHASE

The percent extraction of Fe (III) with anthranilic acid is found to increase with pH of the aqueous phase and becomes almost quantitative at pH 3.2, in both the cases of pyridine and β-picoline as synergists.

3.2 EFFECT OF METAL ION CONCENTRATION

The effect of metal ion concentration in the range $5.0 \times 10^{-5} - 1.0 \times 10^{-3}$M on the extraction of Fe (III) is found to be practically insignificant ($\pm 3\%$). This rule out the possibility of extraction of any polymeric species in the abovementioned concentration range. A concentration of 1.0×10^{-3}M for Fe (III) was chosen to carry out the present investigation.

3.3 EFFECT OF ACID CONCENTRATION

The experimental data [TABLE-1] of distribution ratio for Fe (III) between aqueous phase and benzene as a function of anthranilic acid concentration at constant Fe (III) concentration, constant pH and constant pyridine concentration (0.25M) or β-picoline concentration (0.5M), respectively were obtained. In both cases of pyridine and β-picoline as synergists, the extraction of Fe (III) increases with the increase in concentration of anthranilic acid in aqueous phase before equilibration.

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TABLE 1. Distribution ratio for Fe (III) between aqueous phase and benzene as a function of anthranilic acid concentration at constant pH and base concentration.

$[ANTH]_T$	log $[ANTH]_T$	D	log D
(a) *Pyridine*			
$[Fe]_T = 1.0 \times 10^{-3} M$, $[pyridine]_T$ = 0.25M, pH = 3.2			
5.000×10^{-3}	-2.301	6.586	0.819
1.101×10^{-2}	-1.958	12.882	1.109
1.901×10^{-2}	-1.721	19.952	1.299
2.600×10^{-2}	-1.585	27.461	1.439
3.303×10^{-2}	-1.481	35.424	1.549
(b) β − *picoline*			
$[Fe]_T = 1.0 \times 10^{-3} M$, [β-picoline]$_T$ = 0.5M, pH = 3.2			
3.006×10^{-3}	-2.522	6.046	0.781
6.814×10^{-3}	-2.166	15.342	1.186
8.924×10^{-3}	-2.049	20.657	1.315
1.200×10^{-2}	-1.920	24.618	1.391
1.428×10^{-2}	-1.845	36.054	1.557

Plots of log D vs log of total anthranilic acid concentration has been drawn from the extraction data. The plots $\log D$ vs $\log[ANTHRA]_{TOTAL}$ with pyridine and β-picoline give straight line with slope near one. This suggests that in each case of pyridine and β-picoline there is utilisation of one acid molecule per metal atom releasing one proton during formation of extracting species [Figure. 1,2].

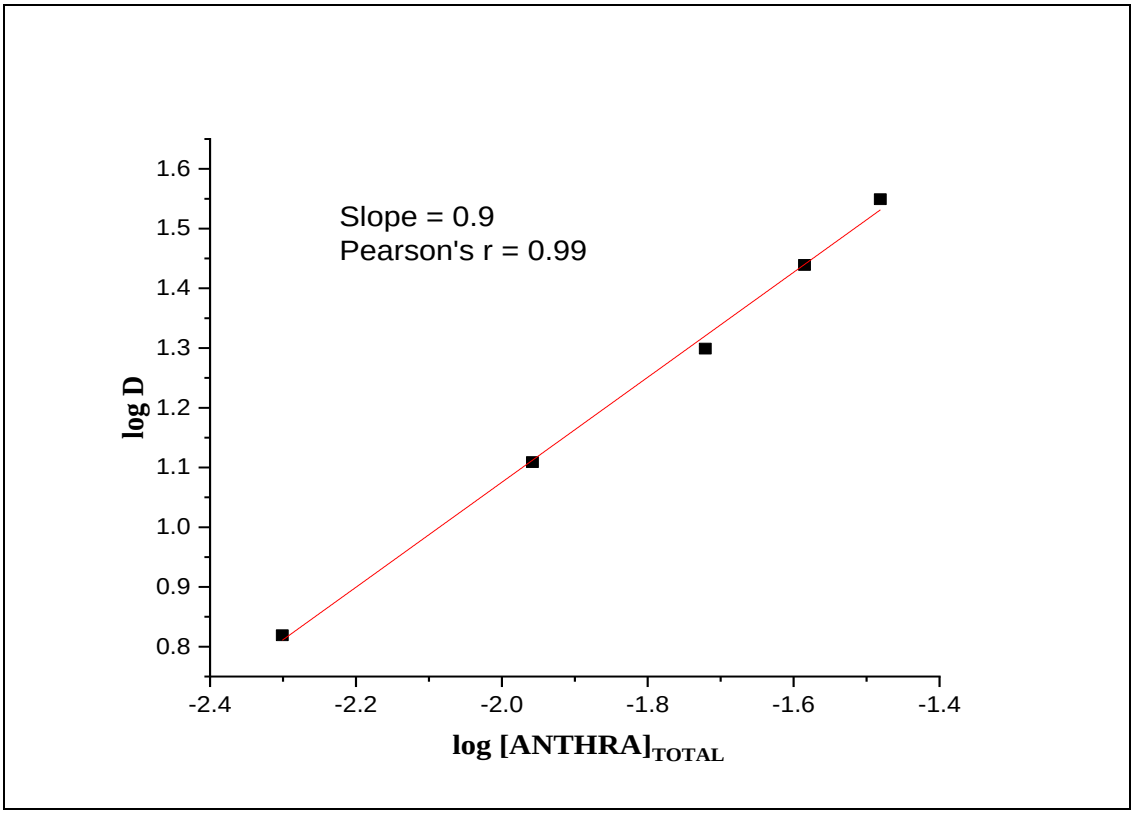

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FIGURE 1: Variation of Distribution ratio for Fe (III) as a function of anthranilic acid concentration at constant pH = 3.2 and at constant pyridine concentration (0.25M)

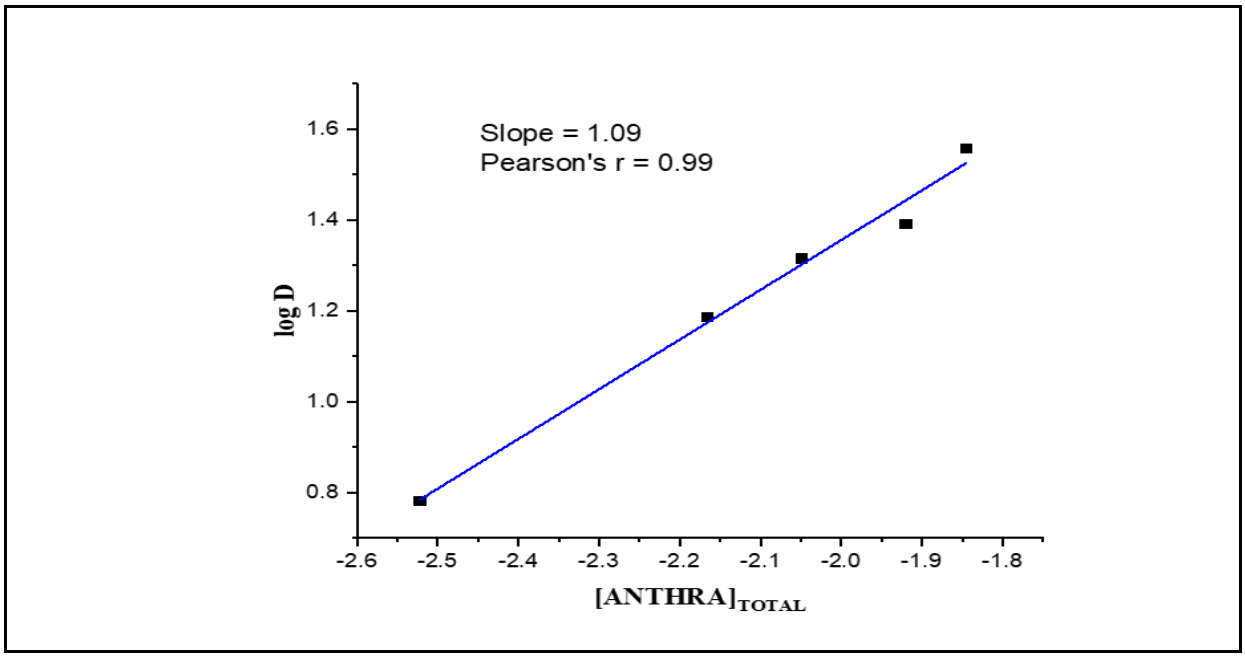

FIGURE 2: Variation of Distribution ratio for Fe (III) as a function of anthranilic acid concentration at constant pH = 3.2 and at constant β-picoline concentration (0.5M)

3.4 EFFECT OF BASE CONCENTRATION

From the extraction studies, distribution ratio D for Fe (III) between aqueous phase and benzene as function of base concentration at constant pH, total metal ion concentration and acid concentration, has been calculated and reported in [TABLE 2]. The log-log plot of distribution ratio versus base concentration has been drawn at constant pH and acid concentration for each of pyridine and β-picoline [Figure 3,4].

TABLE 2. Distribution ratio for Fe (III) between aqueous phase and benzene as a function of base concentration at constant pH and anthranilic acid concentration.

$[BASE]_T$	log $[BASE]_T$	D	log D
(a) *Pyridine*			
$[Fe]_T = 1.0 \times 10^{-3} M$, $[anthranilic\ acid]_T$ = 0.025M, pH = 3.2			
5.000×10^{-2}	-1.301	1.000	0.000
7.500×10^{-2}	-1.125	2.636	0.421
1.250×10^{-1}	-0.903	8.749	0.942
1.450×10^{-1}	-0.839	10.116	1.005
1.750×10^{-1}	-0.757	17.179	1.235
2.500×10^{-1}	-0.602	35.399	1.549
(b) $\beta -$ *picoline*			
$[Fe]_T = 1.0 \times 10^{-3} M$, $[anthranilic\ acid]_T$ = 0.015M, pH = 3.2			
1.000×10^{-2}	-2.000	1.426	-0.154
4.000×10^{-2}	-1.397	6.397	0.806
7.014×10^{-2}	-1.154	18.030	1.256
1.000×10^{-1}	-1.000	22.542	1.353
1.300×10^{-1}	-0.886	43.351	1.637

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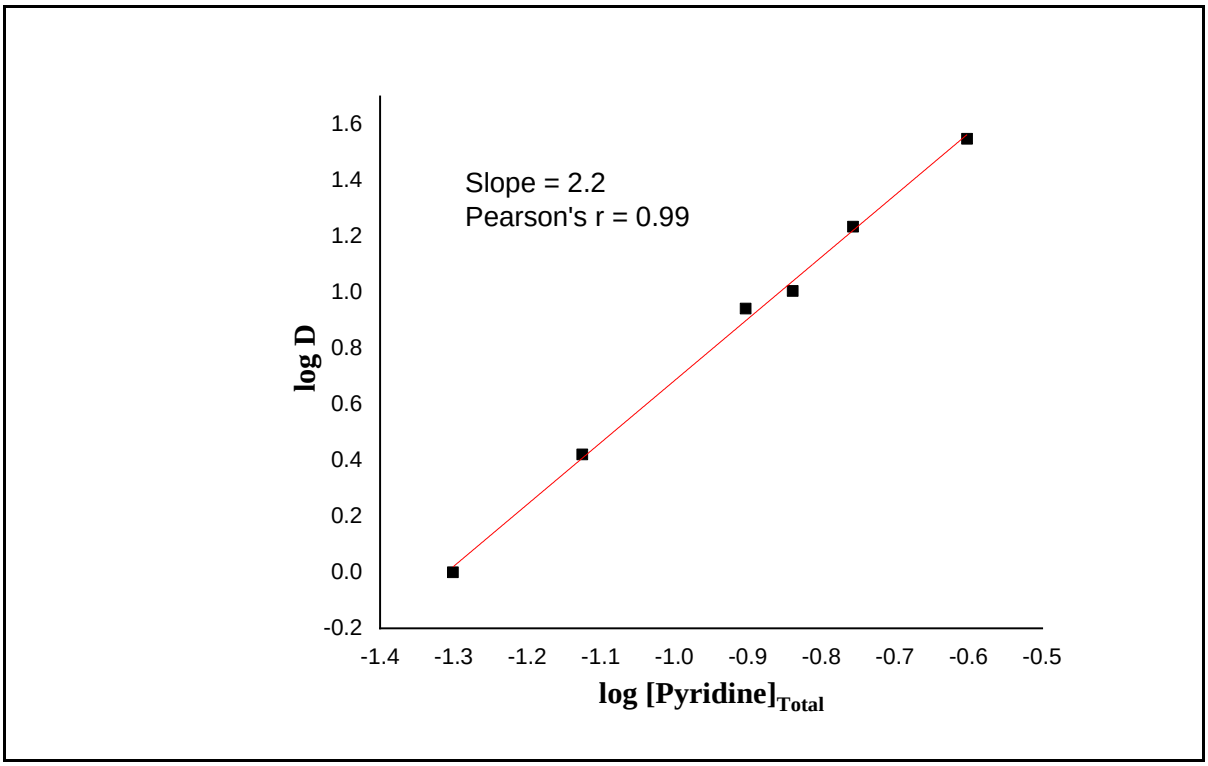

FIGURE 3: Variation of Distribution ratio for Fe (III) as a function of Pyridine concentration at constant pH (3.2) and Anthranilic acid concentration (0.025M)

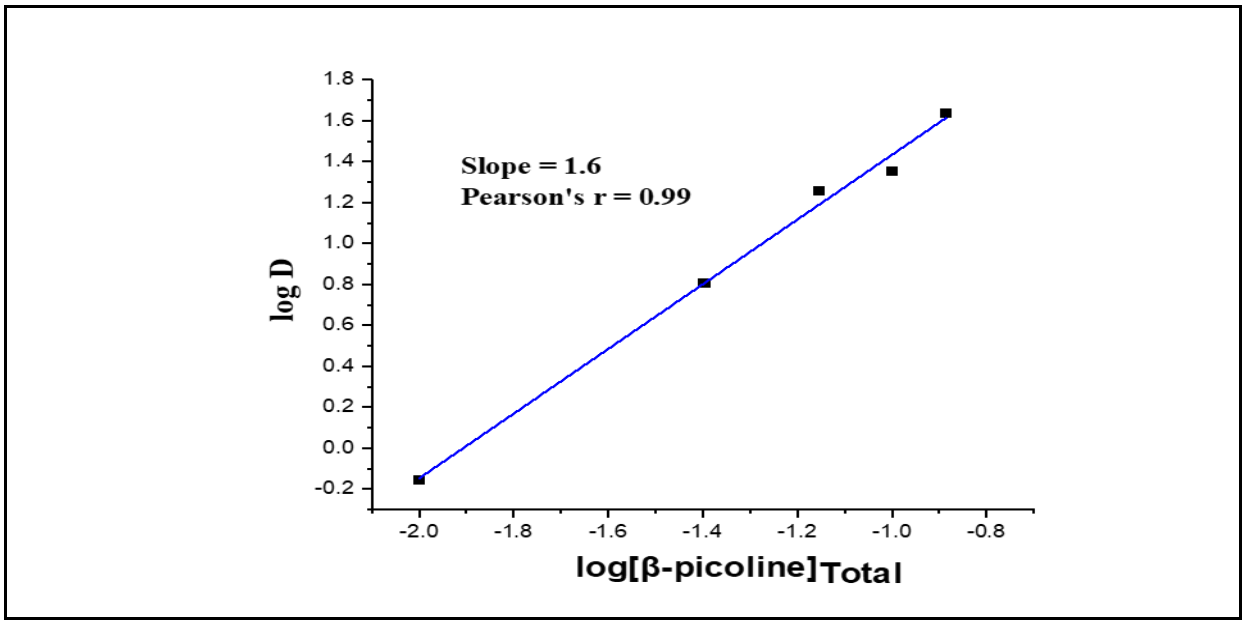

FIGURE 4: Variation of Distribution ratio for Fe (III) as a function of β-picoline concentration at constant pH (3.2) and Anthranilic acid concentration (0.015M)

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It is observed that with the increase in the concentration of organic bases the extraction of Fe (III) increases. The plot of log D versus log $[Base]_{Total}$ with both the bases gives straight line with slope near two [Figure 3,4]. This suggests the incorporation of two molecules of pyridine or β-picoline per metal atom in the extracting species.

3.5 CALCULATION OF EXTRACTION CONSTANTS (K_{ex})

Using the equation:

$$[B]_{aq} = \frac{[B]_{total} - x.[MA_n.xB]_{org}}{P_B + 1 + \frac{[H^+]}{K_{BH^+}}}$$

$[B]_{aq}$ is calculated. Putting the values of $logD$, log $[HA]_{aq}$, log $[B]_{aq}$, pH and n = 1, x = 2 in the following equation

$$\log D = \log K_{ex} + n\ log\ [HA]_{aq} + x \log\ [B]_{aq} + npH$$

Extraction constant, K_{ex} is computed [TABLE – 3, 4].

TABLE 3. Variation of $[\text{pyridine}]_{aq}$ for Fe (III) at constant anthranilic acid concentration (0.025M) and pH = 3.2

$[Fe\}_{Total}$	$[B]_{Total}$	log D	log $[B]_{aq}$	log K_{ex}	Mean of log K_{ex}
1.0×10^{-3}M	5.000×10^{-2}	0.000	-3.274	5.00	**5.05**
	7.500×10^{-2}	0.421	-3.097	5.01	
	1.250×10^{-1}	0.942	-2.874	5.09	
	1.450×10^{-1}	1.005	-2.808	5.02	
	1.750×10^{-1}	1.235	-2.726	5.08	
	2.500×10^{-1}	1.549	-2.569	5.09	

TABLE 4. Variation of $[\beta - \text{picoline}]_{aq}$ for Fe (III) at constant anthranilic acid concentration (0.015M) and pH = 3.2

$[Fe\}_{Total}$	$[B]_{Total}$	log D	log $[B]_{aq}$	log K_{ex}	Mean of log K_{ex}
1.0×10^{-3}M	1.000×10^{-2}	-0.154	-4.489	7.75	**7.15**
	4.000×10^{-2}	0.806	-3.851	7.13	
	7.014×10^{-2}	1.256	-3.602	7.08	
	1.000×10^{-1}	1.353	-3.442	6.86	
	1.300×10^{-1}	1.637	-3.327	6.92	

Computed values of log K_{ex} for pyridine and β-picoline are 5.05 and 7.15, respectively. This suggests β-picoline as better synergist in comparison to pyridine. This, also, suggests that the stability of the extracting complex of β-picoline is higher than those of pyridine.

Plots for log D vs log$[B]_{aq}$ are drawn using data from TABLE. 3 and TABLE. 4 for pyridine [Figure 5] and β-picoline [Figure 6]. The plot gives straight line with a slope nearly two in each case of pyridine and β-picoline

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which again suggests incorporation of two base molecules per metal atom during the formation of extracting species.

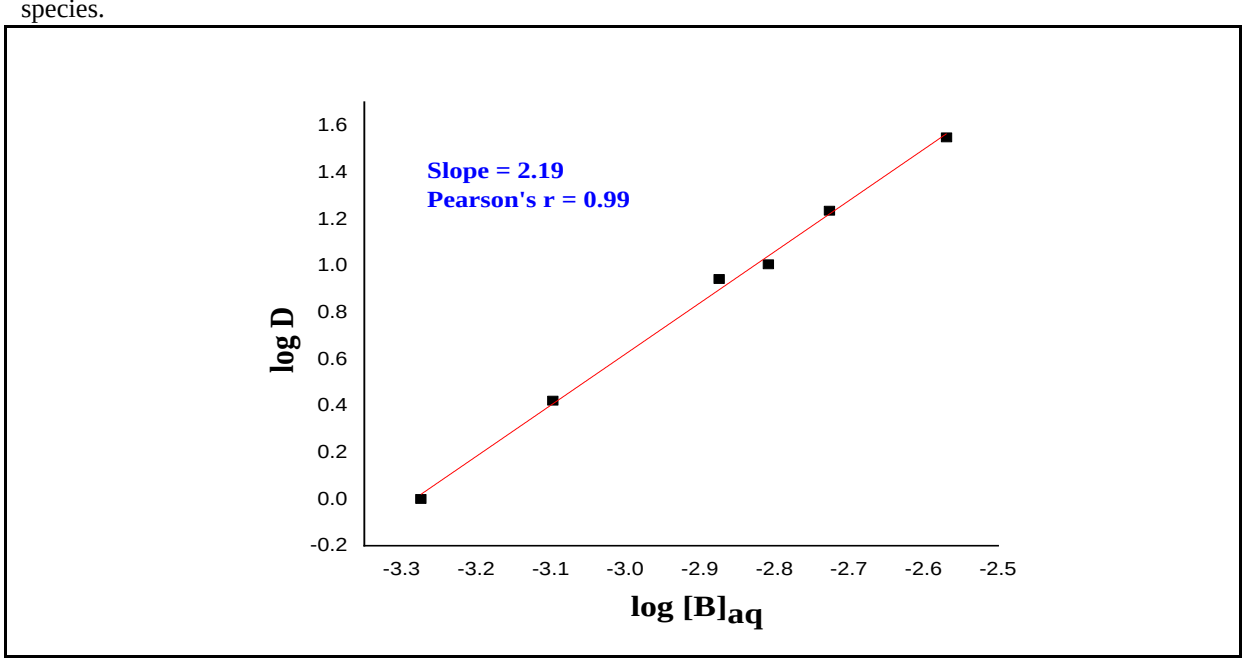

FIGURE 5: Plot of logD vs log$[B]_{aq}$ for variation of pyridine concentration at constant Fe (III) concentration, pH, and anthranilic acid concentration

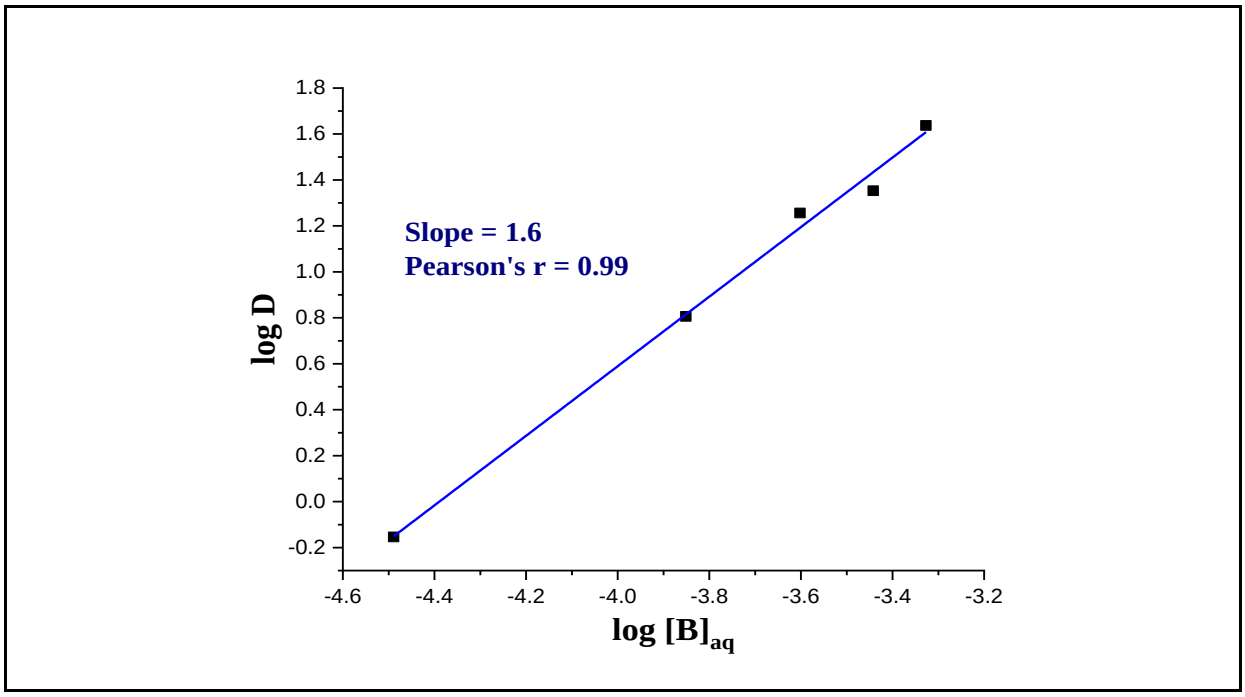

FIGURE 6: Plot of logD vs log$[B]_{aq}$ for variation of β-picoline concentration at constant Fe (III) concentration, pH, and anthranilic acid concentration

3.6 EXTRACTED SPECIES

On the basis of the extraction data, assuming coordination number of Fe (III) to be six the representation of extracting species may be proposed as

$$Fe(OH)_2A.2B$$

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4. CONCLUSION

The reported extracting species in the present investigation is $[\mathbf{Fe(OH)_2A.2B}]$ which contains hydroxyl group in the coordination sphere of the metal ion. Considering the pH of aqueous phase, it is expected. Comparing the value of extraction constant (K_{ex}), the effectiveness of pyridine and β-picoline as synergist is in the sequence as

$$\beta - picoline > pyridine$$

5. ACKNOWLEDGEMENT

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6. CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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