

# Synthesis, Characterization and Antimicrobial Activity of Fe (III) Complexes with Tartaric Acid/Succinic Acid and Heterocyclic Amines

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## Abstract

The synthesis and characterization of mixed ligand complexes of Fe (III) with tartaric acid/succinic acid and heterocyclic amines. These complexes were characterized on the basis of elemental analysis, conductometric, magnetic measurements, UV-vis, IR spectral studies. All the complexes were found electrolytic and paramagnetic in nature with high spin octahedral structure. The synthesized complexes showed moderate to strong antimicrobial activity.

## Keywords

Mixed Ligand Complex, Electronic Spectra, Antimicrobial Activity, Heterocyclic Amines, Tartaric Acid, Succinic Acid

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## 1. Introduction

Tartaric acid is a muscle toxin, which acts by inhibiting the production of malic acid and in high doses causes paralysis and death (Gold and Zahm 1943). It is used in the medical fields (Addington et al., 1998) and acting as a preservative after fermentation (Salgado et al., 2010). Salts of tartaric acid (Rochelle salt) used as mild laxative and tartaric dihydrazides utilized as agrochemicals (Kinoshita et al., 1997). Derivatives of tartaric acid are frequently used, widely available and inexpensive acid resolving agents for the separation of racemic mixtures via diastereoisomeric salt (Jacues et al., 1981), (Ilmarinen et al., 2001) or supramolecular compound formations (Nemak et al., 1996 and Casba et al., 2000).

Succinic acid, derived from fermentation of agricultural carbohydrates, has a specialty chemical market in industries producing food and pharmaceutical products, surfactants and detergents, green solvents and biodegradable plastics, and ingredients to stimulate animal and plant growth (Zeikus et

al., 1999). Succinic acid (Suc) is involved in the citric acid or tricarboxylic acid (TCA) cycle and the glyoxalate cycle. It is synthesized in almost all microbial, plant, and animal cells (Song and Lee 2006). Suc is a bidentate ligand. In coordination chemistry, it forms strong complexes with many metal ions (Gandham and Rao 2012).

Recently, we studied electronic properties of N<sub>2</sub>O<sub>4</sub> Schiff base ligand containing metal complexes of Cd(II), Pd(II), Hg(II) and Zr(IV) (kudrat et al., 2013, Anarul et al., 2014 and Laila Arjuman Banu et al., 2015) also synthesis and characterization of mixed ligand coordinating Co(II), Cu(II), Ni(II), Cr(III) and Fe(III) complexes with amino acid and heterocyclic amines (Bashar et al., 2014, Bashar et al., 2014, Bashar et al., 2015 and Kudrat et al., 2015). In this study, we described a systematic study of preparation and characterization of mixed ligand Fe(III) metal complexes with tartaric acid or succinic acid and heterocyclic amines also their functions as antimicrobial actions.

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## 2. Experimental

### 2.1. Materials

All chemicals were commercial products and were used as supplied. Melting points of all metal complexes were measured by an electro thermal melting point apparatus model no. AZ6512. The SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance was used for magnetic moment measurements. Infrared spectra (KBr) were recorded in a SIMADZU FTIR- 8400 (Japan) spectrophotometer in the range 4000 – 400  $\text{cm}^{-1}$ .

### 2.2. General Method for the Preparation of the Complexes

General method for preparation of the Fe(III) complexes of  $[\text{Fe}(\text{Tar})\text{L}_4]\text{Cl}$  and  $[\text{Fe}(\text{Succ})\text{L}_4]\text{Cl}$ ; Where, L = Pyridine, Quinoline, and Iso-quinoline and 2-Picoline, Tar=Tartaric acid and Succ = Succinic acid.

An ethanolic solution of Fe(III) chloride (1 m mole) and deprotonated tartaric acid (1 m mole) or succinic acid (1 m mol ) were mixed with constant stirring but no precipitate was observed. Then 25 ml of an ethanolic solution of L (4 m

mole) was added to the resulting mixture and heat on a magnetic regulator hot plate with constant stirring. The volume of the solution was reduced to one half and allowed to cool. The precipitate formed and were filtered, washed several times with ethanol and then dried in a desiccator over anhydrous  $\text{CaCl}_2$ .

## 3. Results and Discussion

All the complexes are stable at room temperature. The Fe(III) complexes are insoluble in common organic solvents but are soluble in DMSO, DMF and  $\text{CHCl}_3$ .

### 3.1. Elemental Analysis and Conductivity Measurement

The molar conductance of  $10^{-3}\text{M}$  solution of the complexes in DMF was measured at  $28^\circ\text{C}$ . The molar conductance values are in the range 132.55 to 166.30  $\Omega^{-1}\text{cm}^2\text{mole}^{-1}$  (Table 1). The molar conductance values (Table 1) indicate that the compounds are 1:1 electrolytic in nature. Some physical and chemical properties are shown in table 1. The obtained elemental data were in good agreement with the calculated value (Table 2).

Table 1. Physical properties of Fe(III) complexes.

Complexes	Colour	Melting point or decomposition temperature ( $\pm 5^\circ\text{C}$ )	Molar conductance ( $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ )	$\mu_{\text{eff}}$ (B.M.)
$[\text{Fe}(\text{Tar})(\text{Py})_4]\text{Cl}$	Chocolate	284	142.05	5.58
$[\text{Fe}(\text{Tar})(\text{Q})_4]\text{Cl}$	Grey	178	164.86	5.78
$[\text{Fe}(\text{Tar})(\text{IQ})_4]\text{Cl}$	Redish brown	212	136.60	5.92
$[\text{Fe}(\text{Tar})(2-\text{Pic})_4]\text{Cl}$	Brown	265	128.24	5.75
$[\text{Fe}(\text{Succ})(\text{Py})_4]\text{Cl}$	Ash	186	166.30	5.81
$[\text{Fe}(\text{Succ})(\text{Q})_4]\text{Cl}$	Green	230	155.95	5.88
$[\text{Fe}(\text{Succ})(\text{IQ})_4]\text{Cl}$	Grey	235	132.55	5.99
$[\text{Fe}(\text{Succ})(2-\text{Pic})_4]\text{Cl}$	Light Brown	248	147.52	5.97

Where Py = Pyridine, Q= Quinoline, IQ= Iso-quinoline, 2-Pic= 2-Picoline, Tar=Tartaric acid and Succ = Succinic acid

Table 2. Data of the elemental analysis of the complexes.

Complexes	Molecular Weight		Metal %		Carbon %		Hydrogen %		Nitrogen %	
	Cal	Found	Cal	Found	Cal	Found	Cal	Found	Cal	Found
$[\text{Fe}(\text{Tar})(\text{Py})_4]\text{Cl}$	555.78	555.86	10.05	10.11	51.87	51.92	4.35	4.41	10.08	10.18
$[\text{Fe}(\text{Tar})(\text{Q})_4]\text{Cl}$	756.02	756.12	7.39	7.44	63.55	63.68	4.27	4.33	7.41	7.43
$[\text{Fe}(\text{Tar})(\text{IQ})_4]\text{Cl}$	756.02	756.09	7.39	7.52	63.55	63.58	4.27	4.32	7.41	7.50
$[\text{Fe}(\text{Tar})(2-\text{Pic})_4]\text{Cl}$	611.89	611.93	9.13	9.21	54.96	54.97	5.27	5.29	9.16	9.27
$[\text{Fe}(\text{Succ})(\text{Py})_4]\text{Cl}$	523.78	523.88	10.66	10.76	55.04	55.15	4.62	4.74	10.70	10.80
$[\text{Fe}(\text{Succ})(\text{Q})_4]\text{Cl}$	724.02	724.17	7.72	7.83	66.36	66.49	4.46	4.55	7.74	7.79
$[\text{Fe}(\text{Succ})(\text{IQ})_4]\text{Cl}$	724.02	724.14	7.72	7.85	66.36	66.46	4.46	4.53	7.74	7.84
$[\text{Fe}(\text{Succ})(2-\text{Pic})_4]\text{Cl}$	579.89	579.94	9.63	9.71	58.00	58.22	5.56	5.64	9.66	9.81

Where Py = Pyridine, Q= Quinoline, IQ= Iso-quinoline, 2-Pic= 2-Picoline, Tar=Tartaric acid and Succ = Succinic acid.

### 3.2. IR Spectral Studies

The IR spectrum of the complexes showed characteristic bands at around 1466  $\text{cm}^{-1}$ , 2910  $\text{cm}^{-1}$ , 1615  $\text{cm}^{-1}$ , 1303  $\text{cm}^{-1}$ , 552  $\text{cm}^{-1}$ , and 486  $\text{cm}^{-1}$  corresponding to the  $\nu(\text{C}=\text{N})$ ,  $\nu(\text{CH})$ ,  $\nu(\text{C}=\text{O})$ ,  $\nu(\text{C}-\text{O})$ ,  $\nu(\text{M}-\text{O})$ ,  $\nu(\text{M}-\text{N})$  stretching frequencies,

respectively (Table 3). The ligand Tartaric acid co-ordinates to the Fe(III) ion through the carboxylic acid group as the  $\nu(\text{M}-\text{O})$  and  $\nu(\text{C}-\text{O})$  bands of the complex present in IR spectra. The free hetero amine ligand shows  $\nu(\text{C}=\text{N})$  band at 1815  $\text{cm}^{-1}$ . The lowering of the stretching frequency suggested that the heterocyclic amine bonded to metal

through the nitrogen atom. The bands of the complexes at  $412\text{ cm}^{-1}$  is undoubtedly to the (M - N) modes. In case of first four complexes, the presence of IR band at  $3400\text{ cm}^{-1}$

indicated that the OH group did not make bond to the metal ion.

**Table 3.** Selected IR spectral data of the complexes.

Complexes	$\nu(\text{C}=\text{N})\text{ cm}^{-1}$	$\nu(\text{C}-\text{H})\text{ cm}^{-1}$	$\nu(\text{C}=\text{O})\text{ cm}^{-1}$	$\nu(\text{C}-\text{O})\text{ cm}^{-1}$	$\nu(\text{M}-\text{O})\text{ cm}^{-1}$	$\nu(\text{M}-\text{N})\text{ cm}^{-1}$
[Fe(Tar)(Py) <sub>4</sub> ]Cl	1466	2910	1615	1303	552	486
[Fe(Tar)(Q) <sub>4</sub> ]Cl	1532	2912	1608	1364	509	492
[Fe(Tar)(IQ) <sub>4</sub> ]Cl	1408	2893	1567	1289	521	521
[Fe(Tar)(2-Pic) <sub>4</sub> ]Cl	1543	2870	1669	1337	520	520
[Fe(Succ)(Py) <sub>4</sub> ]Cl	1530	2920	1656	1306	501	491
[Fe(Succ)(Q) <sub>4</sub> ]Cl	1497	3010	1666	1390	495	516
[Fe(Succ)(IQ) <sub>4</sub> ]Cl	1440	2850	1689	1360	533	489
[Fe(Succ)(2-Pic) <sub>4</sub> ]Cl	1563	2851	1703	1413	481	504

Where, Py = Pyridine, Q= Quinoline, IQ= Iso-quinoline, 2-Pic= 2-Picoline, Tar=Tartaric acid and Succ = Succinic acid

### 3.3. Magnetic Moment and Electronic Spectra

The observed values of effective magnetic moments ( $\mu_{\text{eff}}$ ) of the complexes at room temperature are given in table 1. The magnetic moment of Fe (III) complexes varies from 5.58 to 5.99 B.M. corresponding to five unpaired electrons which indicated that all the complexes are in high spin octahedral structure.

All the compounds under investigation are found to be paramagnetic and their electronic spectra are also given in table 4. The electronic spectra of the Fe(III) complexes gave three bands at (423-437) nm, (346-358) nm, and (305-315) nm, which are caused by  ${}^6\text{A}_{1g} \rightarrow {}^4\text{E}_g(\text{G})$ ,  ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}(\text{D})$  and  ${}^6\text{A}_{1g} \rightarrow {}^4\text{E}_g(\text{D})$  and charge transfer band respectively suggesting the octahedral stereo-chemistry.

**Table 4.** Selected electronic spectral data for the complexes in nm.

Complexes	Band I	Band II	Band III	Band IV
[Fe(Tar)(Py) <sub>4</sub> ]Cl	433	352	312	230
[Fe(Tar)(Q) <sub>4</sub> ]Cl	435	351	309	231
[Fe(Tar)(IQ) <sub>4</sub> ]Cl	429	349	307	229
[Fe(Tar)(2-Pic) <sub>4</sub> ]Cl	430	346	305	230
[Fe(Succ)(Py) <sub>4</sub> ]Cl	425	358	310	234
[Fe(Succ)(Q) <sub>4</sub> ]Cl	434	351	315	225
[Fe(Succ)(IQ) <sub>4</sub> ]Cl	437	350	308	233
[Fe(Succ)(2-Pic) <sub>4</sub> ]Cl	423	353	311	237

Where, Py = Pyridine, Q= Quinoline, IQ= Iso-quinoline, 2-Pic= 2-Picoline, Tar=Tartaric acid and Succ = Succinic acid

## 4. Antibacterial and Antifungal Screening

Metal complexes play an important role in regulating biological activities. The disk diffusion method was employed for the in vitro study of antibacterial effects against one Gram positive and two Gram negative bacteria. On the other hand antifungal activities of the metal complexes against two pathogenic fungi *Candida albicans* (Human Pathogens) and *Aspergillus species* (Plant Pathogens) were

measured. The results revealed that the complexes are more microbial toxic than the free metal ions or ligands (Md. Kudrat-E-Zahan *et al.*, 2015). Fe(III) complexes showed moderate to strong antimicrobial activity as shown in table 5 and 6.

**Table 5.** Antibacterial activity of the synthesized complexes.

Compounds	Zone of inhibition, diameter in mm		
	<i>Escherichia coli</i>	<i>Shigella sonnei</i>	<i>Bacillus subtilis</i>
[Fe(Tar)(Q) <sub>4</sub> ] Cl	26	25	40
[Fe(Succ)(Q) <sub>4</sub> ] Cl	23	18	19
[Fe(Succ)(IQ) <sub>4</sub> ]Cl	17	11	16
[Fe(Tar)(IQ) <sub>4</sub> ]Cl	23	20	31
Kanamycin disc	20	20	22

Where, Q= Quinoline, IQ= Iso-quinoline, Tar=Tartaric acid and Succ = Succinic acid

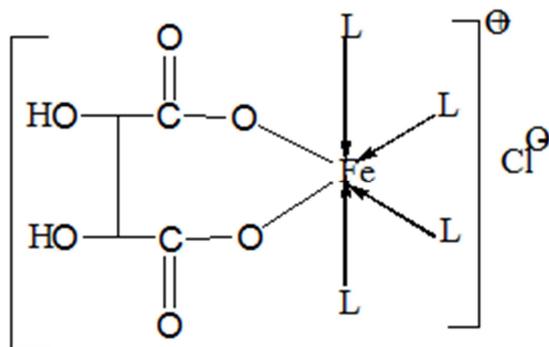
**Table 6.** Antifungal activity the synthesized complexes.

Compounds	Zone of inhibition, diameter in mm	
	<i>Candida albicans</i>	<i>Aspergillus species</i>
[Fe(Tar)(Q) <sub>4</sub> ] Cl	25	23
[Fe(Succ)(Q) <sub>4</sub> ] Cl	22	20
[Fe(Succ)(IQ) <sub>4</sub> ]Cl	17	19
[Fe(Tar)(IQ) <sub>4</sub> ]Cl	21	13
Nystatin	20	20

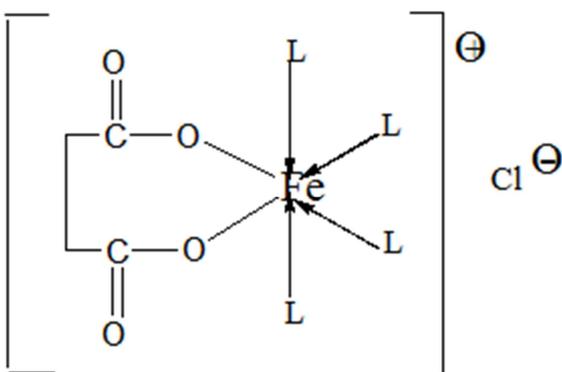
Where, Q= Quinoline, IQ= Iso-quinoline, Tar=Tartaric acid and Succ = Succinic acid

## 5. Conclusion

Elemental analysis correspond to metal: ligand stoichiometry for Fe(III) complexes are 1:1:4. Magnetic susceptibility measurement indicated the paramagnetic nature of the complexes. The IR spectral data shows the ligand coordinate with the metal through O and N atoms. The electronic spectral data are in conformity with the transitions of octahedral Fe(III) complexes. Based on these facts structure of complex have been proposed as shown in Figure 1 and 2.



**Figure 1.** Proposed structure of  $[\text{Fe}(\text{tar})\text{L}_4]\text{Cl}$  complexes Where, L = Pyridine, Quinoline, Isoquinoline and 2-Picoline.



**Figure 2.** Proposed structure of  $[\text{Fe}(\text{Succ})\text{L}_4]\text{Cl}$  complexes Where, L = Pyridine, Quinoline, Iso-quinoline and 2-Picoline.

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