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Synthesis and Characterization of Chalcone and their Fe(III) metal complexes

Thakare AP[1], Dahane AS[2], Rathod SB[3] and Mandlik PR[1]

[1]P. G. Department of Chemistry, Shri Shivaji Science College, Amravati, MS, India
[2]Koradi Art, Science and Commerce College, Mahadula, Nagpur, MS, India
[3]Balasaheb Jadhao Arts, Commerce and Science College, Ale, MS, India
Email- anmol21086@gmail.com

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ABSTRACT

In present study three chalcones 2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one [HMPO], 2-hydroxy-4-methylphenyl-3-(p-tolyl) prop-2-en-1-one [HMTO] and 4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one [CHMPO] were synthesized by condensing 2-hydroxy-4-methylacetophenone with benzaldehyde, 4-methylbenzaldehyde and 4-chlorobenzaldehyde. The synthesized chalcones were characterized by IR, NMR and Mass spectral studies. With this chalcones Fe(III) metal complexes were prepared and also characterized by different spectroscopic techniques. IR spectra indicates that hydroxyl oxygen and nuetral carbonyl involved in the coordination with Fe(III) ion. All the complexes posses' octahedral geometry.

Keywords: Chalcone, metal complexes, IR, mass, TGA.

INTRODUCTION

Chalcone is an aromatic ketone and an enone that forms the central core for a variety of important biological compounds, which are known collectively as chalcones or chalconoids. The chemistry of chalcones has generated great scientific interest due to their biological and industrial applications. Chalcones are natural biocides and are well known intermediates in the synthesis of heterocyclic compounds exhibiting various biological activities.

Chalcones and their derivatives possess some interesting biological properties such as antibacterial, antifungal, insecticidal, anesthetic, anti-inflammatory, analgesic etc [1-4]. A number of chalcones having hydroxy group at different position having ability to form coordinate bond with different metal ion in order to form transition metal complexes.

METHODOLOGY

All chemicals used were of the analytical reagent (AR) grade and of highest purity available and purchased from SD-Fine Chem Limited. Melting points were determined with an Electro thermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu 4300 spectrometer. NMR spectra were recorded with a Brucker 80 instrument using TMS as internal standard. Mass analyses of the products were conducted with a Finnigan-Matt 8430 GC-Mass instrument.

Synthesis of chalcone

A mixture of 0.01 mol 2-hydroxy-4-methylacetophenone and 0.01 mol various aldehyde added into ethanol solvent. To this reaction mixture 20 % NaOH added and heated for several minutes' upto formation of solid residue. By keeping overnight residue nuetralized by ice cold HCl solution, filtered and dried in oven [5-7].

Synthesis of metal complexes

An equimolar mixture of respective chalcone and $FeCl_3.H_2O$ (0.01 mole) were added in RB flask containing ethanol and refluxed for 8-10 hrs to obtained solid residue. Residue filtered, dried and recrystallized with ethanol.

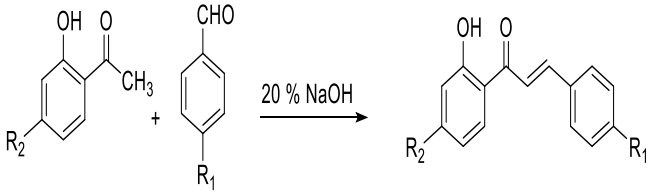

Fig 1: Synthesis of Chalcone

Synthesis of metal complexes

Newly synthesized chalcone (0.01 mol) and $FeCl_3.6H_2O$ (0.01 mol) were taken in R.B. flask already containing ethanol solvent and refluxed for 4-6 hrs to obtained solid residue. Residue filtered, dried and recrystallized by using ethanol.

RESULTS AND DISCUSSION

IR Spectra

IR spectral technique is helpful in order to determine binding mode of ligand towards metal ion. In chalcone hydroxyl stretching band is appeared at 3305-3315 cm^{-1} which is completely disappered in metal complexes confirm the participation of hydroxyl oxygen in coordination with metal ion. This confirms by the upward shift of C-O stretching frequency by 19-32 cm^{-1} is spectra of complexes. A band due to carbonyl group at 1687-1695 cm^{-1} again shifted to lower frequency at extent 20-34 cm^{-1} suggest coordination of carbonyl oxygen with metal ion. At the same time new spectral bands appeared in spectra of complexes in region 530-555 cm^{-1} due to M-O stretching vibration [8-10]

Sr No	Chalcone	R_1	R_2
1	2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one [HMPO]	H	CH_3
2	2-hydroxy-4-methylphenyl-3-(p-tolyl)prop-2-en-1-one [HMTO]	CH_3	CH_3
3	4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one [CHMPO]	Cl	CH_3

Spectroscopic data of 2-hydroxy-4-methylphenyl-3-phenylprop-2-en-1-one

[HMPO]: IR (KBr) ν_{max}: cm^{-1} : 3305(-OH), 3029(-Ar-CH), 1691(-C=O), 1590(-C=C)

^{1}H-NMR (DMSO-d_6) δ: 9.8(1H,s,OH), 6.98-7.60(7H,m,Ar-H), 2.5(3H,s,CH_3)

Spectroscopic data of 2-hydroxy-4-methylphenyl-3-(p-tolyl)prop-2-en-1-one

[HMTO]: IR (KBr) ν_{max}: cm^{-1} : 3312(-OH), 3015(-Ar-CH), 1687(-C=O), 1592(-C=C)

^{1}H-NMR (DMSO-d_6) δ: 9.5(1H,s,OH), 6.90-7.74(7H,m,Ar-H), 2.4(6H,s,CH_3)

Spectroscopic data of 4-chlorophenyl-2-hydroxy-4-methylphenyl-prop-2-en-1-one

[CHMPO]: IR (KBr) ν_{max}: cm^{-1} : 3315(-OH), 3035(-Ar-CH), 1695(-C=O), 1594(-C=C)

^{1}H-NMR (DMSO-d_6) δ: 9.2(1H,s,OH), 6.98-7.55(7H,m,Ar-H), 2.3(3H,s,CH_3)

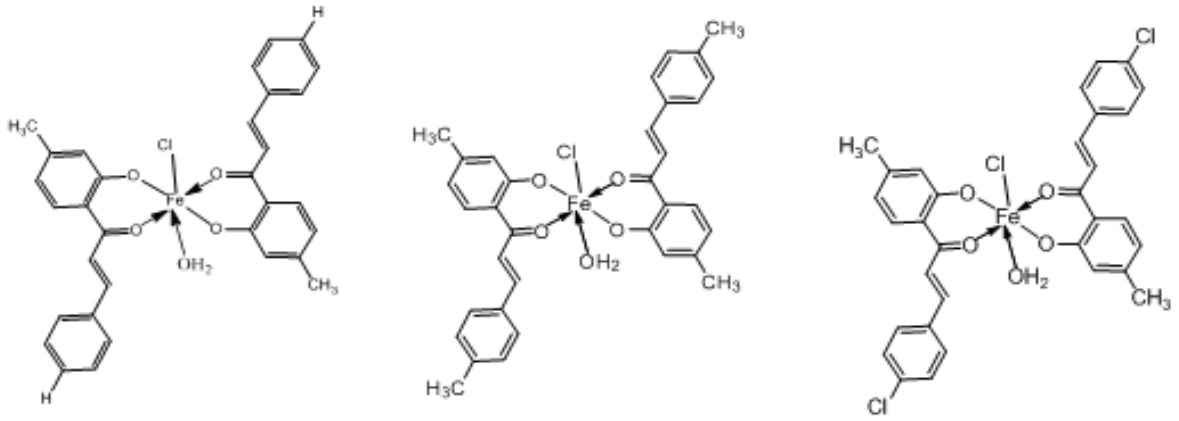

Fig 2: Probable structure for metal complexes

Mass Spectra

Mass spectrometry has been successfully used to determine the molecular ion peak for synthesized compounds. The various fragmentation peaks obtained for chalcone and all Fe(III) complexes are in good agreement with proposed structure. The mass spectral data shows that all Fe(III) complexes are dimeric. Molecular io peak and various fragment ion obtaibed are given below.

Fe(HMPO): m/z- 582, 564, 530, 292, 78.

Fe(HMTO): m/z- 611,592, 558, 502, 306.

Fe(CHMPO): m/z- 651, 633, 597, 326.

Table 1: Analytical data of chalcone and its Complexes

Sr. No.	Compounds	Molecular Formula	Molecular Weight	Colour	M. P. (^{0}C)
1.	HMPO	$C_{16}H_{14}O_2$	238.28	Yellow	120
2.	HMTO	$C_{17}H_{16}O_2$	252.31	Yellow	134
3.	CHMPO	$C_{16}H_{13}ClO_2$	272.73	Brown Yellow	125
4.	Fe(HMPO)	$C_{32}H_{28}ClFeO_5$	583.86	Brown	>300
5.	Fe(HMTO)	$C_{34}H_{32}ClFeO_5$	611.91	Green	>300
6.	Fe(CHMPO)	$C_{32}H_{26}Cl_3FeO_5$	652.75	Green	>300

Table 2: IR spectral bands of Fe(III) complexes of chalcones

Compounds	v(OH)	v(C=O)	v(C-O)	v(M-O)
Fe(HMPO)	--	1671	1309	530
Fe(HMTO)	--	1657	1317	555
Fe(CHMPO)	--	1661	1421	541

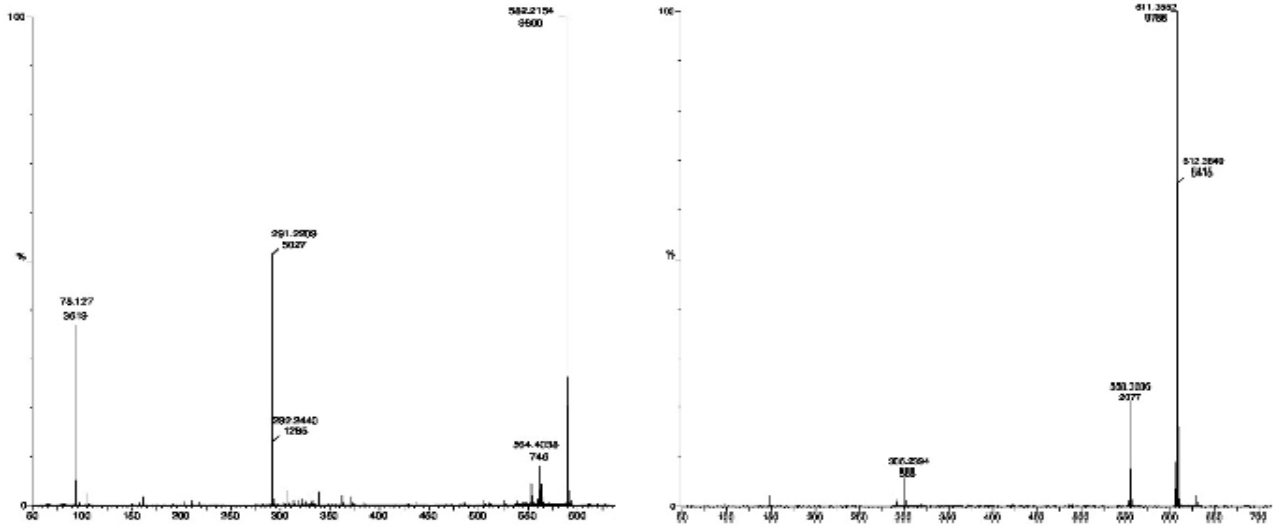

Fig 3: Mass Spectrum of Fe(HMPO) and Fe(HMTO) complex

Thermal Analysis:

Thermogravimetric analysis is the best techniques to find out the thermal stability of complexes. Thermogravimetric analysis was performed in nitrogen atmosphere with heating rate 10^0C per minute and thermograms are recorded in temperature range 40^0C to 800^0C. By using this technique several kinetic parameters like activation energy (Ea), order of reaction (n), entropy change (S) were calculate. The thermal data have been analyzed by using Freemann-Caroll and Sharp-Wentworth methods. All the complexes are stable upto 60^0C and further decomposed in several steps. There occurs no loss upto 120^0 C molecule in all metal complexes indicates the absence of any lattice water molecule. Above 220^0 C the complexes shows the loss of one coordinate water molecule. wt. loss obs/**calcd**: Fe(HMPO)- 3.32/**3.34**

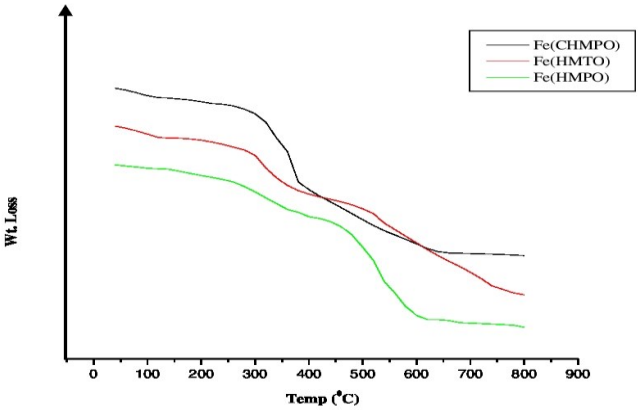

Fig 4: Thermal graph of Fe(III) complexes

Fe(HMTO)- 2.88/**2.94,** Fe(CHMPO)- 2.97**/2.88** Next steps includes the decomposition of coordinate part of ligands and finally all complexes are converted into their respective metal oxides [11].

Table 3: Kinetic Parameter for Fe(III) metal complexes.

Compounds	**Half Decomposition Temperature (^{0}C)**	**Activation Energy Ea (kJ/mole)**		**Order of Reaction (n)**	**Entropy Change -ΔS (J/mol/K)**	**Free Energy Change ΔF (kJ/mol)**
		FC	**SW**			
Fe(HMPO)	440	24.70	23.86	0.98	-145.91	70.65
Fe(HMTO)	490	17.13	17.69	0.99	-148.58	64.63
Fe(CHMPO)	510	21.33	20.89	0.98	-147.63	62.63

CONCLUSION

The present article includes the synthesis and characterization of three chalcones and its Fe(III) complexes. Characterization of compounds includes IR, Mass, ^{1}H NMR and thernogravimetric analysis. Chalcones coordinates to Fe (III) metal ion through hydroxyl oxygen and neutral carbnyl oxygen and act as a dibasic ligand (Metal ligand ratio 1:2) The thermal data shows that complexes were highly stable and its thermal decomposition as well as thermodynamic parameters was studied.

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Conflicts of interest: The authors stated that no conflicts of interest.

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