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Thermal Decomposition Kinetics of Fe(III) Complexes of Chalcone Schiff Bases

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ABSTRACT: In present study three chalcones 1-(2-hydroxy-4-methylphenyl)-3-(4hydroxyphenyl)prop-2-en-1-one $[L_1]$, - 1-(2-hydroxy-4-methylphenyl)-3-(p-tolyl)prop-2-en-1one $[L_2]$ and 3-(4-chlorophenyl)-1-(2-hydroxy-4-methylphenyl)prop-2-en-1-one $[L_3]$ were synthesized by condensing 1-(2-hydroxy-4-methylphenyl)ethan-1-one with 4hydroxybenzaldehyde, 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.

The thermal decomposition study of a series of Fe(III) complexes of chalcone Schiff bases was monitored by non-isothermal analysis in atmosphere. The thermal degradation takes place in three steps for each of the complexes. The decomposition steps were analyzed and the kinetic parameters: order of the decomposition reaction (*n*), activation energy(*Ea*)and frequency factor (*Z*) of the non-isothermal decomposition steps were computed using Freemann-Caroll methods. Thermodynamic parameters: entropy (S) and Gibbs free energy (F) of activation, were calculated using a standard relations and discussed. On the basis of half decomposition temperature, the thermal stability of the compound was found to be Fe-L₃> Fe-L₂ > Fe-L₁. The activation energies of the thermal degradation steps lie in the range 30.73 – 40.57 kJmol⁻¹.

INTRODUCTION

Schiff bases are continued to play an important role in the development of coordination chemistry. Schiff bases and their metal complexes have a variety of applications including biological1 and catalytic activities 2,4 and as compounds of interesting photoluminescent and electroluminescent properties5. In spite of the relatively large number of reports on Schiff base metal complexes, less work has been published on calculation of the kinetic and thermodynamic parameters of their decomposition. In this paper we report on the kinetics of the thermal decomposition for four different Schiff base complexes of Fe(III). The half

decomposition temperature and the basic parameters were calculated using Broido6 and Horowitz-Metzger methods. The complexes are thermally stable and their thermal decompositions are multistage processes. The complexes are subjected to aTG analysis from 50-800°C.

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.



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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 1-(2-hydroxy-4-methylphenyl)ethan-1-one 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.



L1 (R-OH) - 1-(2-hydroxy-4-methylphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one

L2 (R-CH3) - 1-(2-hydroxy-4-methylphenyl)-3-(p-tolyl)prop-2-en-1-one

L₃ (R-Cl) - 3-(4-chlorophenyl)-1-(2-hydroxy-4-methylphenyl)prop-2-en-1-one

Synthesis of metal complexes: An equimolar mixture of respective chalcone and FeCl₃.H₂O (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.

RESULT & 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.



Fig: ¹H NMR Spectrum of 1-(2-hydroxy-4-methylphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one

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Sr No	Compound	M. F.	M. W.	Color	M.P. (⁰ C)		
1	L ₁	$C_{16}H_{14}O_{3}$	254.29	Yellow	122		
2	L_2	$C_{17}H_{16}O_2$	252.12	Yellow	120		
3	L ₃	$C_{16}H_{13}ClO_2$	272.73	Brown	137		
4	$Fe(L_1)$	$C_{32}H_{26}FeO_6$	562.40	Green	>300		
5	Fe(L ₂)	C ₃₄ H ₃₀ FeO ₄	558.46	Green	>300		
6	Fe(L ₃)	C ₃₂ H ₂₄ Cl ₂ FeO ₄	598.29	Dark Green	>300		

Table 1:	Analytica	l data of	f chalcone	and its	complexes

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Thermogravimetric Analysis of the complexes:

All the complexes are stable upto 700C and are decomposed mainly in three stages. Elimination of lattice or coordinate water molecule takes place in the first step, a part of ligand decomposed in second step and the complete decomposition of ligand followed by oxid-red reaction leading to the formation of metal oxide in the last one15-18.

For Mn-L1 complex: Lattice Water upto 130 ^oC, Coordinate Water: 150-220 ^oC, Free Ligand: 230-450^oC, Coordinate Ligand: above 450^oC

Sr No	Compound	Half	Activation	Frequency	Entropy	Free
		Decomposition	Energy	Factor	Change	Energy
		Temp (⁰ C)	(kJmol ⁻¹)	(Sec ⁻¹)	(Jmol ⁻¹ K ⁻¹)	Change
						(kJmol ⁻¹)
1	Fe(L ₁)	472	30.73	26.49	224	45.71
2	Fe(L ₂)	480	40.57	60.04	218	54.73
3	Fe(L ₃)	510	36.25	58.84	222	61.11

Table 2: Thermal data of metal Complexes

For Mn-L₂ complex: Lattice Water upto 120^oC, Coordinate Water: 150-220^oC, Pyrolysis product Fe3O4: 250-600^oC.

For Mn-L₃ complex: Lattice Water upto 130^oC, Coordinate Water: 220^oC, Coordinate ligand:Above 350^oC, Pyrolysis product: 600-700^oC.





Fig: TGA graph of Fe(L2) Complex

CONCLUSION:

11.00

9.00

8.00

vvelght/mg

The activation energy calculated by the Freemann-Caroll methods are in good agreement with each other. Thermodynamic parameters have been calculated on the basis of thermal activation energy and values are given in Table 3. Due to abnormally low value of frequency factor [Z] it may be classified as a slow reaction and no other obvious reason can be given. The thermal stability of the compounds can be correlated with the substituent group attached to the ligands. It is found that more bulky ligand may have more thermal stability.

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