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ULTRASOUND SYNTHESIS, SPECTRAL CHARACTERIZATION AND ANTIMICROBIAL ACTIVITIES OF FE (III) COMPLEXES OF B-DIKETONES

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β-diketones, Transition metal complexes, Ultrasound irradiation, Baker-Venkataraman transformation, Mass, ¹H-NMR, ¹³C-NMR, IR, UV, Antifungal, Antibacterial

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ABSTRACT: Consistently the preparation of transition metal complexes of β diketones has been (and it still is) an area of the highest interest for inorganic and organic chemists, as can be inferred considering the quantity and diversity of research developed in this field. 1,3- Diketones are wide range of valuable molecules including several types of carbocycles and heterocycles hence have high interest. In fact, excessive amount of therapeutical uses are associated with these 1,3- Diketone compounds. The present work is based mainly on the ultrasound irradiation synthesis of various β -diketone ligands and their Fe (III) complexes. At ambient temperature, the clinically active and functionalized various β-diketones has been synthesized from Baker–Venkataraman transformation and its Fe (III) complexes has been prepared and characterized by physical, spectral and analytical data. The different spectroscopic analysis like Mass, ¹H-NMR, ¹³C-NMR, IR, UV and elemental analysis were done. The functionalized beta-diketones showed a certain behaviour and behaved as bidentate ligand and co-ordinate with the transition metal atom through betadiketo system. The complexes have general formula [ML₂]. The biological activities like antibacterial and antifungal were performed for the synthesized compounds. The biological screening data indicated that the transition metal complexes are more potent antibacterial, antifungal and antioxidant agents than the parent functionalized beta diketones against different species of bacteria and fungi. This constitutes a new group of compounds. The thermal stability of the newly synthesized metal complexes has been studied.

INTRODUCTION: For almost a century the chemistry of 1,3-diketones has attracted the attention of scientists. Diketones are the key intermediates for the synthesis of heterocycles such as isoxazole ¹, flavones ², pyrimidine ³, triazole ⁴, pyrazole^[5] and benzodiazepines ⁶. β -diketone for eg. Anabena β -diketone hydrolase have some

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enzymatic activities ⁷, rare earth doped complexes of β -diketone were studied as high density optical recording materials for blue optoelectronics ⁸. β diketone lanthanide complexes are studied for their optical properties like fluorescence ^{9, 10} and electroluminescence ¹¹.

β-diketone are clinically important molecules hence they having some biological activities such as antiviral ¹², anti-tumor ¹³⁻¹⁴, anticancer ¹⁵, insecticidal ¹⁶, antioxidants ¹⁷ and antibacterial ¹⁸. Many naturally occurring 1,3-diketones such as dibenzoylmethane (DBM-1) has been the therapeutic option for cancer treatment, as well as for anti-inflammatory and dementia, has recently reviewed ¹⁹. On the other hand, some novel 3,4disubstituted pyrazole derivatives have shown and antifungal antibacterial properties Australifungin, isolated from sporomiella vities of Fe (III) ustralis shown a prominent antifungal activity²¹. Additionally the chemical structures, of β-diketone can be considered as excellent drug. candidate showing multi-target potency. For instance, asymmetrical 1,3-diketones with antiinflammatory and anti-cancer activities, synthesized by reaction between N-acyl benzotriazoles and ketones based on soft enolization ²². Thus β -diketones act as very important median to various heterocyclic compounds ²³ and also used as chelating agents ²⁴. It is used as a extractant for copper ions²⁵. The research being energizing by the versatility and these compounds as laser chelates ²⁶, chemical and photochemical catalysts ²⁷, shift reagents extraction agents ²⁹. Studies on the β -diketone and their metal complexes are being of more and more interest to the chemists and biochemists ³⁰. This paper reports the synthesis of various ligands by Baker-Venkatraman re-arrangement ³¹ and their Fe (III) complexes, spectral analysis and antimicrobial screening of the compounds.

EXPERIMENTAL METHODS:

Preparation of 2-acetylphenyl, 4-methoxy benzoate:

Compound 1: To the mixture of 2-hydroxy acetophenone (1.36g, 0.01mol) and 4-methoxy benzoic acid (1.52g,0.01mol), a dry pyridine (5-6ml) and phosphorus oxychloride (POCl₃) about 1ml were added drop wise at 0°C with the constant stirring. Then reaction mixture was kept in ultrasonicator for 4-5 hrs. After completion of the reaction (monitored by TLC), the reaction mixture was then cooled and poured on cold water containing HCl (1M) and solid product obtained was filtered and washed with cold methanol (10ml) and after that with distilled water. It was then re-crystallized from ethanol. A similar procedure was adopted for the preparation of other compounds namely:

- a) 2-acetylphenyl, 4-ethoxy benzoate
- **b**) 2-acetylphenyl, 4-bromo benzoate
- c) 5-chloro, 2-acetylphenyl, 4-methoxy benzoate

- **d**) 5-chloro, 2-acetylphenyl, 4-ethoxy benzoate
- e) 5-chloro, 2-acetylphenyl, 4-bromo benzoate

Preparation of 1-(2-hydroxyphenyl)-3-(4methoxyphenyl) propane-1,3-dione [L₁]:

Compound 2: For the preparation of 1-(2-hydroxyphenyl)-3-(4-methoxyphenyl)propane-1,3-dione compound 1, (2.70g,0.01mol) was dissolved in dry pyridine (about 10ml) and to this powdered KOH (1.12g,0.02mol) was added and the reaction mixture was irradiated in ultrasound for 1-2hrs. After completing the reaction (monitored by TLC), the mixture was poured on in ice-cold water and acidified with conc. HCl. The solid obtained was then filtered off and it was re-crystallized from absolute alcohol.

Yield: 85% M.P: 115°C. FTIR(KBr)cm⁻¹: FT-IR (KBr) cm-1: 2912.98 (-OH), 1708.01 (C=O), 1487.74 (Ar C=C).¹H-NMR (300 MHz, CDCl₃,d6): δ =7.9 (d, 3H, Ar-H), 6.8 (m, 5H, Ar-H),7.4 (q , 1H,=CH-), 3.9 (s,3H, OCH₃) ,12.2 (s, 1H, OH), 15.9 (s, 1H, Enolic-OH), ¹³C-NMR (300 MHz, CDCl₃), δ 190.0(s, C-1,C=O), 92.8 (s, C-2,-CH=), 185.1(S,C-3), 126.0(d, C-1',C-1''), 162.8 (s,C-2'), 118.4 (s,C-3'), 135.8 (s,C-4'), 119.3 (s,C-5'), 128.7 (s, C-6,'), 128.0 (d, C-2'',C-6''), 114.1(d, C-3'', C-5''), 162.0 (s, C-4''), 55.8 (s,C-7'',0CH₃). UV/Vis (DMSO) nm: 370,410; EC-MS: 270.28 (M+23).

A similar procedure was adopted to prepare following ligands:

- **a**) 1-(2-hydroxyphenyl)-3-(4-ethoxyphenyl) propane-1,3-dione,
- **b**) 1-(2-hydroxyphenyl)-3-(4-bromoyphenyl) propane-1,3-dione,
- c) 1-(5-chloro,2-hydroxyphenyl)-3-(4methoxyphenyl).propane-1,3-dione,
- **d**) 1-(5-chloro,2-hydroxyphenyl)-3-(4ethoxyphenyl) propane-1,3-dione,
- e) 1-(5-chloro,2-hydroxyphenyl)-3-(4bromoyphenyl)propane-1,3-dione.

Preparation of Fe (III) Complex: The mixture of compound 2 (5.40g, 0.02mol), anhydrous Fe (III) nitrate (4.04g, 0.01mol) and 20 ml anhydrous ethanol was added and irradiated for 1-2 hrs under

ultrasound. The obtained solid was washed with hot ethanol and recrystallised from ethyl acetate. The brownish crystals of Fe (III) β - diketonate obtained.

Yield: 78%; mp: 348°C. A similar procedure was adopted to prepare Fe (III) complexes of remaining ligands.



SCHEM 1: SYNTHESIS OF 1-(2-HYDROXYPHENYL)-3-(4-METHOXYPHENYL) PROPANE-1, 3-DIONE AND Fe(III) COMPLEX

RESULT AND DISCUSSION:

Antimicrobial Activity: Antimicrobial screening ³²⁻³³ is done by using the method called Kirby Baur's disc diffusion technique using dimethyl sulfoxide as a solvent. The streptomycin was used as a standard and the method were tested against bacteria such as *Staphylococcus aureus* and *Bacillus subtilis* (Gram +ve); *Escherichia coli* (Gram-ve) and against fungi like *Fusarium oxysporum* and *Aspergillus niger*. A uniform suspension of a test organism of 24 hours old

cultures was prepared in test tube holding a sterile saline solution. A 20 ml sterile Muller-Histon agar was then added in each of the petri plates. The plates were rotated to ensure the uniform mixing of micro-organism in agar medium which was then allowed to solidify. Then by keeping sterile Whatman filter paper disc were dipped in the solution of each compound and placed on labeled plates. Then these petri plates were kept in refrigerator for half an hour for diffusion then bacterial cultured plate incubated at 37°C for 24 hours and fungal cultured plate were incubated at 30°C for 24 hours. The antibacterial activity was examined by measuring the diameter of inhibition zone formed. The zones were measured in terms of mm. The results of antimicrobial activity of synthesized compounds have shown that the

transition metal complex reveals the greater antimicrobial activity than that of the ligand. The observed data of antimicrobial activity of synthesized compounds and the standard is given in **Table 1.**

 TABLE 1: DATA OF ANTIMICROBIAL ACTIVITY OF LIGANDS (L1-L6) WITH THEIR FE (III) COMPLEXES

 Compounds

 Zone of Inhibition in mm

Compounds									
	Α	ntibacterial activity		Antifunga	l activity				
	Bacillus subtilis	Staphylococcus	E. coli	Fusarium	Aspergillus				
		aureus		oxysporum	niger				
L ₁	6	6	7	7	7				
ML_1	8	7	8	7	9				
L_2	7	7	6	8	7				
ML_2	7	8	8	7	7				
L_3	7	7	7	7	7				
ML_3	7	6	8	8	6				
L_4	7	8	6	7	6				
ML_4	7	8	8	6					
L_5	7	6	7	7	7				
ML_5	8	7	8	6	7				
L ₆	7	7	7	7	7				
ML ₆	8	7	6	7	8				
stryptomycin	6	6	7	6	6				

TABLE 2: ANALYTICAL DATA OF Fe (III) COMPLEXES

Complex	Molecular formula	Mol. Wt	% Found (calculated)							
			С	Η	Br	Cl	0	Fe		
ML_1	C ₃₂ H ₃₆ FeO ₁₂	632	60.97	4.80			25.38	8.86		
			(60.11)	(4.97)			(25.11)	(8.17)		
ML_2	C33H38FeO12	660	62.02	5.20			24.30	8.48		
			(61.08)	(5.17)			(24.01)	(7.35)		
ML_3	$C_{32}H_{36}FeBrO_{12}$	730	49.48	3.32	21.95		17.58	7.67		
			(49.41)	(3.15)	(21.30)		(17.04)	(7.01)		
ML_4	$C_{32}H_{36}FeClO_{12}$	701	54.96	4.04		10.14	22.88	7.99		
			(54.41)	(4.12)		(10.02)	(22.30)	(7.12)		
ML_5	C33H38FeClO12	729	56.14	4.43		9.75	22.00	7.68		
			(55.23)	(4.47)		(9.13)	(21.45)	(7.07)		
ML_6	C ₃₂ H ₃₆ FeClBrO ₁₂	799	45.21	2.78	20.05	8.90	16.06	7.01		
			(45.15)	(2.01)	(19.71)	(8.19)	(15.77)	(6.91)		

TABLE 3: PHYSICAL CHARACTERISTICS OF LIGANDS WITH THEIR Fe (III) COMPLEXES

Sr. no.	Compounds	Compounds % Yield Colour		Melting point
1	ML_1	78	Blackish brown	348°C
2	ML_2	82	Blackish brown	324°C
3	ML_3	79	Blackish brown	330°C
4	ML_4	84	Blackish brown	319°C
5	ML_5	81	Blackish brown	338°C
6	ML_6	80	Blackish brown	345°C

Molar Conductance and Magnetic Susceptibility: The new synthesized ligands and their Fe(III) complexes are in the solid state and are very stable at room temperature. The synthesized ligands are soluble in common organic solvents, and their Fe(III) complexes are soluble in DMF and DMSO. Due to the continuous variation results, it is concluded that the stoichiometry of the complexes are conformable with the ratio 1:2 for metal to ligand. The molar conductivity of all complexes were measured in dimethyl formamide and values were observed between 61.3-67.2 ohm⁻¹ cm² mol⁻¹ indicating their non-electrolytic nature ³⁴.

TABLE 4: MOLAR CONDUCTANCE	AND MACNETIC SUSCEPTIBILITY	OF Fo(III) COMPPLEXES
IADLE 4: MIULAK CONDUCTANCE	AND MAGNETIC SUSCEF HIDILII I	OF FE(III) CONFFLEADS

Sr. no.	Complex	Molar conductance	Xdia × 10 ⁻⁶	$\mu_{\rm eff}$ (B.M.)
1	ML_1	61.3	-513.15	5.55
2	ML_2	62.4	-564.21	6.01
3	ML_3	67.2	-319.66	5.62
4	ML_4	63.1	-489.25	5.92
5	ML_5	65.6	-535.11	6.37
6	ML_6	64.8	-521.17	6.06

Spectral Characterizations:

TABLE 5: INFRARED SPECTRAL DATA, UV AND MASS OF SYNTHRESIZED COMPOUNDS											
Sr. no.	Compound			IR (cm ⁻¹)			UV	Mass			
								spectra			
		v(C=O)	v(C=C)	v(OH)	C-Br bond	M-O	λ_{\max} for		Donor		
							>C=O (nm)		atom		
1	L_1	1708	1599	2912			410,370	270.28	0-0		
	ML_1	1682	1602	3187,3290		620					
2	L_2	1702	1565	2921			410,360	284.3	0-0		
	ML_2	1692	1599	3243,3526		562					
3	L_3	1718	1558	3069	1223		412,360	319.15	0-0		
	ML_3	1685	1580	3271,3180		575					
4	L_4	1741	1591	2919			412,374	304.73	0-0		
	ML_4	1670	1597	3216,3417		562					
5	L_5	1720	1605	2977			410,370	318.75	0-0		
	ML_5	1650	1605	3244,3412		600					
6	L_6	1743	1587	2916	1239		410,360	353.6	0-0		
	ML ₆	1690	1634	3216,3072		606					

¹**H-NMR:** ¹H-NMR spectra of β-diketones showed two proton signals at a range δ 15.4-17.7 ppm and δ 11.9-12.2 ppm which corresponds to enolic proton and phenolic proton adjacent to carbonyl group. It confirms the formation of β-diketone. The compound in enolic form is more stable than that of ketonic one ³⁵. ¹³C-NMR: In the ¹³C-NMR spectra all synthesized β -diketone ligands gives characteristic peak at ketonic carbon C₁, C₂ and enolic carbon C₃ are in the ranges δ 189.5-193.8, δ 91.2-93.1 and δ 176.8-185.1 ppm confirms the formation of β -diketone ³⁶.

TABLE 6: INDUCED X-RAY DIFFRACTION OF COMPLEX Fe-L₁

Peak no.	20(obs)	2θ(cald)	d(obs)	d(cald)	Miller i	Miller indices of planes		
					h	k	Ι	(%)
1	25.796	25.963	3.4509	3.4291	1	0	1	40.0
2	27.298	27.015	3.2643	3.2979	-1	0	2	35.1
3	28.988	28.996	3.0777	3.0769	-1	1	2	23.1
4	30.697	30.708	2.9101	2.9091	-1	2	1	41.1
5	32.094	32.453	2.7866	2.7566	0	1	3	28.2
6	38.012	37.655	2.3652	2.3869	0	3	2	30.0
7	57.077	56.882	1.6123	1.6174	0	2	5	31.2
8	61.199	61.229	1.5132	1.2125	1	3	4	30.1
9	64.311	64.292	1.4473	1.4477	1	5	2	24.1
10	66.287	66.419	1.4088	1.4064	0	6	1	22.0

TABLE 7: INDUCED X-RAY DIFFRACTION OF COMPLEX Fe-L3

Peak no.	2θ(obs)	2θ(cald)	d(obs)	d(cald)	Miller indices of planes			Intensity (%)
					h	k	Ι	
1	21.700	21.960	4.0921	4.0443	-1	0	0	60.0
2	23.492	23.144	3.7839	3.8399	0	2	1	55.1
3	28.192	28.012	3.1628	3.1827	1	1	1	45.1
4	30.302	30.400	2.9472	2.9379	-1	2	0	42.2
5	34.115	34.313	2.6260	2.6113	-1	0	3	40.5

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6	35.597	35.930	2.5200	2.4974	-1	1	3	35.3
7	37.708	37.655	2.3835	2.3869	0	3	2	30.1
8	55.878	55.773	1.6440	1.6469	1	2	4	32.1
9	79.082	79.075	1.2099	1.200	-2	4	5	30.6
10	85.107	85.087	1.1390	1.1392	-3	2	5	28.1

Thermogram of Fe(III) Complexes: In nitrogen atmosphere using α -Al₂O₃ as reference, the simultaneous TG/DT analysis of a complex of Fe (III) was studied. At the temperature range 185-200°C the thermogram curve of Fe (III) complex shows weight loss 7.40% (calcd. 7.78%) and at 190°C it shows keen endotherm which distinctly designate removal of two coordinated water molecules ³⁷. The anhydrous complex revealed a single step decomposition with 70% mass loss and a broad endothermic peak in the DTA at the temperature range from 210°C to 820°C. The Fe₂O₃ is obtained as the end product.

CONCLUSION: In the present work ligands and its Fe(III) complexes were synthesized. On the basis of their spectral analysis the structures were elucidate. Due to presence of enolic proton and phenolic proton adjacent to carbonyl group, the prepared diketones having characteristics peaks of ¹H NMR and ¹³C NMR spectra. It has been suggested that the antibacterial and antifungal activity of ligands $(L_1)-(L_6)$ increased upon chelation/coordination with the transition of metal atoms. By coordinating metal ion with ligands, the chelation process reduces the polarity of metal ion which increase the lipophilic nature of the metals and enhanced its penetration through the lipoid layer of cell membrane of the microorganism. Also, it has been suggested that beta-diketones having combined two or more pharmacophore sites played an important role in antibacterial and antifungal activity. This functionalized system may be responsible for the enhancement of hydrophobic character and liposolubility of the molecules. The synthesized compounds were screened in vitro for antifungal, antibacterial and found to be promising candidates as new antibacterial, antifungal agents.

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CONFLICTS OF INTEREST: Nil

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