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Synthesis, characterization and biological assessment of Ni (II) complexes of β -Diketones

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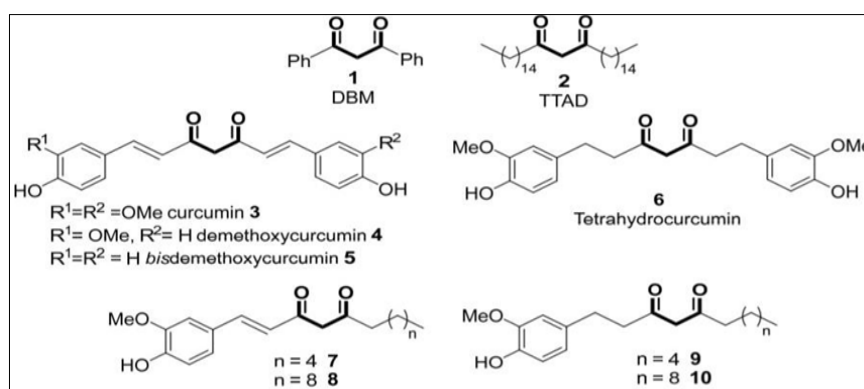
Abstract

The present work on β -diketone is mainly on the synthesis of various ligands and their Ni(II) complex by the method called ultrasound irradiation method. By using the Baker-Venkataraman rearrangement various ligand were prepared at ambient temperature, their Ni (II) complexes were synthesized and characterized by the different methods of spectroscopic analysis like UV, ^1H NMR, Mass, ^{13}C NMR, IR, XRD, DSC elemental analysis. Also the biological assessment like antimicrobial activity was checked for the synthesized compounds.

Keywords: β -diketone, transition metal complex, ultrasound irradiation, Baker-Venkataraman rearrangement, UV, ^1H NMR, Mass, ^{13}C NMR, IR, XRD, DSC, antimicrobial assessment

Introduction

In many of the natural products the 1, 3 -diketones (β -diketones) are found, exhibiting a wide range of biological activities. Thus, many of the naturally occurring 1, 3-diketones such as n-tritriaconfane-16, 18-dione (TTAD) are typical examples [Scheme1] of these type of compounds, naturally obtained from eucalyptus leaves of plant ^[1, 2], vanilla beans ^[3], licorice roots ^[4] or sunflower pollen ^[5] the natural antioxidants possess prominent anti-cancer properties as well as dementia and diabetes have been reviewed recently ^[6]. Curcumin, an active ingredient found in turmeric having the antibacterial activity ^[7]. Curcumin is the most investigated for evaluating its applicability for the management and the prevention of many illnesses including liver disease and cardiovascular obesity, diabetes, inflammation, fibrosis and arthritis, skin diseases ^[8, 9].



Scheme 1: Some linear 1, 3-diketones possessing biological activity

The metal complexes of 1, 3-diketone have broad spectrum such as sensors optical materials, luminescent and electroluminescent technology ^[10-12]. They have also used in healthcare, both as cosmetic additives and as active pharmaceutical ingredients [or as a substrate for medicine manufacturing] which lowers their detrimental effects of UV radiations on the skin ^[13]. The β -diketone ligands and their transition metal complexes shows different types of activities like antimicrobial ^[14], antitumor ^[15, 16], insecticidal activity ^[17], antimalarial ^[18], antioxidant ^[19] and bioinorganic applications ^[20-22] and recently found to be used against HIV ^[23, 24] these compounds also have the engrossment in nucleic acid chemistry as well as their use in biomedicines ^[25-28].

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Co-ordination compounds involving β -diketones are widely covered in the various aspects of the structure, bonding, in the literature and other properties have been treated in several articles [29-39]. This paper reports the synthesis of ligands with their one in all metal complex, spectral analysis and antimicrobial screening of the compounds.

Experimental Methods

Preparation of 2-acetylphenyl, 4-Bromo benzoate: Compound 1

To the mixture of ortho-hydroxy acetophenone (1.36g, 0.01mol) and 4-bromo benzoic acid (2.01g, 0.01mol), a dry pyridine (5-6ml) and phosphorus oxychloride (POCl_3) about 1ml were added dropwise at 0°C with the constant stirring. The reaction mixture was then kept for 4-5hrs in ultra sonicator then reaction mixture was poured into cold water containing HCl (1M) and solid obtained was filtered and washed with 10ml ice-cold methanol and then with distilled water. It was then recrystallized from ethanol.

A similar procedure was adopted for the preparation of other compounds namely,

- 2-acetylphenyl, 4-methoxy benzoate
- 2-acetylphenyl, 4-ethoxy benzoate
- 5-chloro, 2-acetylphenyl, 4-methoxy benzoate
- 5-chloro, 2-acetylphenyl, 4-ethoxy benzoate
- 5-chloro, 2-acetylphenyl, 4-bromo benzoate

Preparation of 1-(2-hydroxyphenyl)-3-(4-bromophenyl) propane-1, 3-dione [L]: Compound 2

For the preparation of 1-(2-hydroxyphenyl)-3-(4-bromophenyl) propane-1, 3-dione, compound 1, (3.19g, 0.01mol) was dissolved in dry pyridine (about 10ml) 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), and the mixture

was poured in ice-cold water and acidified with conc. HCl. The solid obtained was then filtered off and it was recrystallized from absolute alcohol.

Yield: 87% M.P: 130°C FTIR(KBr) cm^{-1} : 3069(OH), 1558(C=C), 1718(C=O), 1429(-CH₂); $^1\text{H-NMR}$ (300MHz, $\text{CDCl}_3\text{-d}_6$); δ =7.3(s, 1H,=CH), 12.1(s,1H, phenolic-OH), 15.5(s,1H,enolic-oH) $^{13}\text{C-NMR}$ (300MHz, CDCl_3); δ 190.0(s, C-1, C=O), 93.0(s, C-2,-CH=), 184.2(s, C-3), 126.5(s, C-1'), 162.0(d, C-2'), 119.0(s,C-3'), 136.0(s,C-4'), 119.5(t, C-5'), 131.3(s, C-6'), 131.6(s, C-3'', C-5''), 128.6(s, C-2'', C-6''). UV/Vis (DMSO) nm:360, 412; EC-MS : 319.15 (M+23).

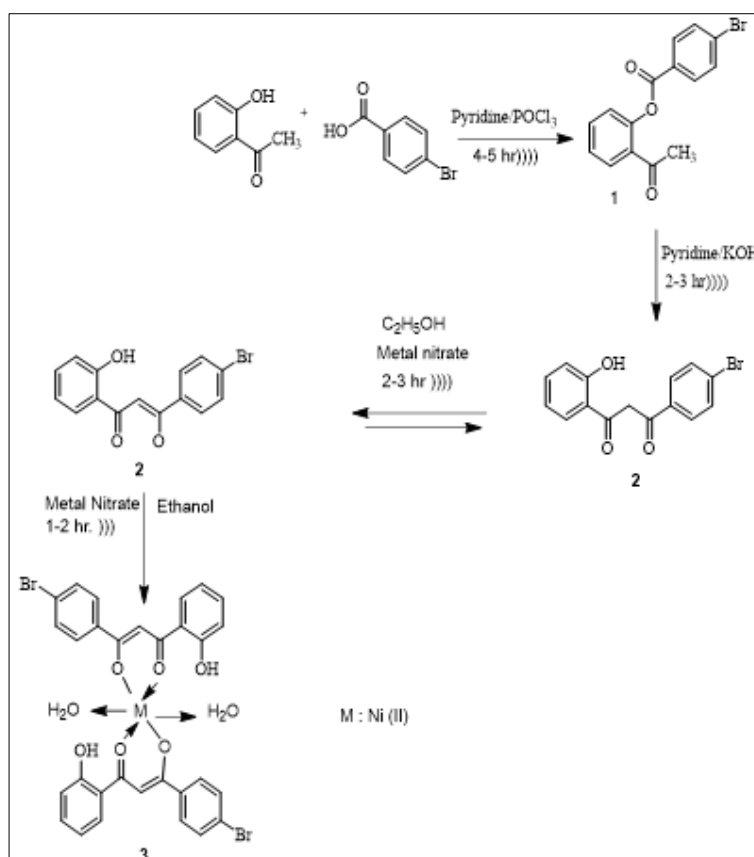
A similar procedure was adopted to prepare other type of ligands that are

- 1-(2-hydroxyphenyl)-3-(4-methoxyphenyl) propane-1, 3-dione,
- 1-(2-hydroxyphenyl)-3-(4-ethoxyphenyl) propane-1,3-dione,
- 1-(5-chloro, 2-hydroxyphenyl)-3-(4-methoxyphenyl).propane-1, 3-dione,
- 1-(5-chloro, 2-hydroxyphenyl)-3-(4-ethoxyphenyl) propane-1, 3-dione,
- 1-(5-chloro, 2-hydroxyphenyl)-3-(4-romoxyphenyl) propane-1, 3-dione.

Preparation of Ni (II) complex

The mixture of the 1-(2-hydroxyphenyl)-3-(4-bromophenyl) propane-1,3-dione (6.38g, 0.02mol), anhydrous Ni (II) (2.90g,0.01mol) and 20ml anhydrous ethanol was added and the mixture was irradiated in ultrasound for 1-2hrs. The precipitated solid was washed with ethanol and recrystallized from ethyl acetate to give crystals of Ni (II) β -diketonato. Yield: 78% M.P: 222°C

A similar procedure was adopted to prepare Ni(II) complexes of remaining ligands.



Scheme 2: Synthesis of 1-(hydroxyphenyl)-3-(4-Bromo) Propane 1, 3-dione and its Ni (II) complex

Result and Discussion

Table 1: Analytical data for Ni (II) complex

complex	Molecular formula	Mol. Wt	% Found (calculated)					
			C	H	Br	Cl	O	Ni
ML ₁	C ₃₂ H ₃₆ O ₁₂ Ni	635	60.69 (59.97)	4.77 4.67	----	----	25.26 25.16	9.27 8.85
ML ₂	C ₃₃ H ₃₈ O ₁₂ Ni	663	61.75 (61.66)	5.18 5.21	----	----	24.18 24.01	8.88 8.12
ML ₃	C ₃₂ H ₃₆ BrO ₁₂ Ni	733	49.29 (49.13)	3.31 3.12	21.86 20.91	----	17.51 17.18	8.03 7.85
ML ₄	C ₃₂ H ₃₆ ClO ₁₂ Ni	704	54.74 54.07	4.02 4.11	----	10.10 10.0+6	22.79 22.19	8.36 8.07
ML ₅	C ₃₃ H ₃₈ ClO ₁₂ Ni	732	55.92 55.13	4.42 4.17	----	9.71 9.23	21.91 21.09	8.04 7.69
ML ₆	C ₃₂ H ₃₆ ClBrO ₁₂ Ni	801	45.05 45.16	2.77 2.92	19.98 (18.88)	8.86 8.13	16.00 15.66	7.34 7.11

Antimicrobial activity

Antimicrobial substances put a stop on the growth of micro-organism and metal complexes shows a very good antimicrobial activity.

Antimicrobial screening [40] 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 eg. *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-Hinton 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 2.

Table 2: Antimicrobial activity of compound

Compounds	Zone of Inhibition in mm				
	Antibacterial activity			Antifungal activity	
	<i>Bacillus subtilis</i>	<i>Staphylococcus aureus</i>	<i>E. coli</i>	<i>Fusarium oxysporum</i>	<i>Aspergillus niger</i>
L ₁	6	6	7	7	7
ML ₁	15	13	14	15	13
L ₂	7	7	6	8	7
ML ₂	15	15	13	14	14
L ₃	7	7	7	7	7
ML ₃	13	14	13	13	14
L ₄	7	8	6	7	6
ML ₄	13	14	13	13	13
L ₅	7	6	7	7	7
ML ₅	14	15	13	12	13
L ₆	7	7	7	7	7
ML ₆	14	14	13	15	14
Streptomycin	6	6	7	6	6

Table 3: Physical characteristics of ligands with their metal complex

Sr No.	Compounds	% Yield	Time	Colour	Melting point
1	L ₁	85	410	Light green	115°C
	ML ₁	78	120		222°C
2	L ₂	85	430	Light green	146°C
	ML ₂	82	110		213°C
3	L ₃	87	510	Light green	130°C
	ML ₃	79	130		215°C
4	L ₄	82	420	Light green	164°C
	ML ₄	84	120		224°C
5	L ₅	84	415	Light green	132°C
	ML ₅	81	130		219°C
6	L ₆	88	430	Light green	138°C
	ML ₆	80	110		217°C

Solubility and molar conductivity

The new synthesized ligands and their Ni(II) complexes are in the solid state and are very stable at room temperature. The synthesized complexes are soluble in DMF and DMSO and ligands are soluble in common organic solvents, The molar

conductivity of all complexes were measured in dimethyl formamide and values were observed between 38.8-53.6 ohm⁻¹cm²mol⁻¹ indicating their non-electrolytic nature. The magnetic moments of complexes given in Table 4 suggest Ni(II) complexes have paramagnetic characters ranging from 2.42–3.02 BM [41].

Table 4: Molar conductance and magnetic susceptibility of metal complex

Sr no.	Complex	Molar conductance Ω ⁻¹ cm ² mol ⁻¹	Xdia × 10 ⁻⁶	μ _{eff} (B.M.)
1	Ni-L ₁	41.2	-314.21	2.42
2	Ni-L ₂	43.6	-348.82	2.58
3	Ni-L ₃	38.8	-355.15	3.08
4	Ni-L ₄	47.6	-313.57	3.02
5	Ni-L ₅	51.2	-377.12	2.80
6	Ni-L ₆	53.6	-389.22	2.78

Table 5: Infrared spectral data, UV and Mass of synthesized compounds

Sr. no.	Compound	IR (cm ⁻¹)					UV		Mass spectra	
		$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{C})$	$\nu(\text{OH})$	C-Br bond	M-O	λ_{max} for $>\text{C}=\text{O}$ (nm)		Donor atom	
1	L ₁	1708	1599	2912	----	----	410,370	270.28	O-O	
	ML ₁	1663	1608	3157 3572	----	570				
2	L ₂	1702	1565	2921	----	----	410,360	284.3	O-O	
	ML ₂	1685	1612	3136 3417	----	643				
3	L ₃	1718	1558	3069	1223	----	412,360	319.15	O-O	
	ML ₃	1665	1582	3363 3517	----	597				
4	L ₄	1741	1591	2919	----	----	412,374	304.73	O-O	
	ML ₄	1678	1593	2916 3085	----	560				
5	L ₅	1720	1605	2977	----	----	410,370	318.75	O-O	
	ML ₅	1680	1612	3185 3501	----	636				
6	L ₆	1743	1587	2916	1239	----	410,360	353.6	O-O	
	ML ₆	1680	1607	3378 3417	----	561				

¹H-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 [42].

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 Page 58 ranges δ 189.5-193.8, δ 91.2-93.1 and δ 176.8-185.1 ppm confirms the formation of β -diketone [43].

IR Spectra

In the IR spectra of all Ni (II) complexes, a broad band is exhibited in 1663-1680 cm⁻¹ suggesting (C=O) stretching mode. This shows down shift of frequency by 40-50 cm⁻¹ than the free ligands. This is because of the active participation of oxygen atoms of carbonyl group in complex formation. In all present complexes, two additional broad bands were shown in the region 3417- 3572 cm⁻¹ due to the water molecules present in the form of lattice water or coordinated water. In the far infrared region of all complexes, a band was observed in the region 560-643 cm⁻¹ which can be assigned to M-O bonding [44].

Table 6: Induced X-ray diffraction of complex Ni-L

Peak no.	2 θ (obs)	2 θ (calcd)	d (obs)	D (calcd)	Miller indices of planes h k l			Intensity (%)
1	19.489	19.489	4.5511	4.5510	1	1	3	78.2
2	22.750	22.833	3.9056	3.8915	1	2	3	65.5
3	24.251	24.126	3.6671	3.6859	1	1	4	62.2
4	28.895	28.835	3.0874	3.0937	1	4	0	55.1
5	32.802	32.742	2.7280	2.7329	2	4	0	45.2
6	38.610	38.603	2.3300	2.3304	4	2	1	38.6
7	40.325	40.400	2.2348	2.2308	0	2	7	35.7
8	42.237	42.206	2.1379	2.1394	3	0	6	32.0
9	43.993	43.966	2.0565	2.0577	3	5	0	30.1
10	45.495	45.519	1.9921	1.9911	2	6	0	25.6

Thermogram of Ni (II) complex

The thermogram of the Ni (II) complex showed that from 155-190 °C a mass loss of 6.88% (calcd. 6.42%) and with 170 °C, endothermic DTA peak which suggested the loss of two coordinated water molecule. From 360 °C to 410 °C with a 24.50% (calcd. 22.70%) mass loss, the anhydrous complex first showed decomposition and with 350 °C in the DTA, a broad exothermic peak which may be attributed to the removal of the non-coordinated part of the ligand. From 460-890 °C with a mass loss of 56.30% (calcd. 55.65%), the second step of the decomposition corresponds to the decomposition of the coordinated part of the ligand. [45] A broad endothermic peak in the DTA was observed for this step. The mass of the final residue NiO, 18.02% (calcd. 17.80%) is in agreement with earlier workers. Mapari et. al. [46] reported thermogram of mixed ligand complexes of N- (2-

hydroxy-1-naphthylidene)-2, 6-diisopropylaniline and N-(2-hydroxy benzylidene)-2, 3-dimethylaniline with Ni (II) ions. The Ni (II) complexes lost their weight in the temperature range 125-260 °C corresponding to two coordinated water molecules with an endothermic peak in DTA curve. After the total loss of water, the organic moiety decomposed on further increment of temperature. At 450-650°C, the decomposition of ligands occurred complete and the detected residue corresponds to the respective oxide.

Conclusion

In the present work ligands and its Ni (II) complexes were synthesized. On the basis of their spectral analysis the structures were elucidate. The ¹H NMR and ¹³C NMR spectra acknowledged that the synthesized diketones having characteristics peaks caused by the presence of enolic proton

and phenolic proton adjacent to carbonyl group. The biological assessment data showed that the Ni (II) complexes are more potent antibacterial and antifungal agents than the parent functionalized beta-diketone against different bacterial and fungal species. This established a new group of compounds that can be used as potential metal derived drugs. Here we can promote about the use of these Ni (II) complexes for the drugs.

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