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Synthesis and Characterization of Some New Chalcones and Their Ti(III) and V(III) Complexes

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Some new chalcones have been prepared by the Claisen-Schmidt condensation using subtituted acetophenones and different aromatic aldehydes. Titanium(III) and vanadium(III) complexes of these synthesized chalcones have been prepared and characterized by elemental analysis, molar conductance, magnetic properties, electronic and IR spectral data. Octahedral geometry has been proposed for these metal chelates. Chalcones have been screened for their antimicrobial activities.

Key Words: Chalcone, Antimicrobial activity, Chelate, Octahedral.

INTRODUCTION

Due to the interesting activity of chalcone derivatives as biological agents, considerable attention has been focused on this class of compounds. The pharmaceutical importance of these compounds lies in the fact that they can be effectively utilized as antibacterial, antifungal, antiviral, antiparasitic, anticancer, antileishmanial and antitubercular agents[1-6]. Some of these compounds are also known to possess anti-inflammatory and analgesic properties[7-21].

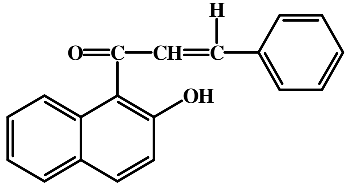
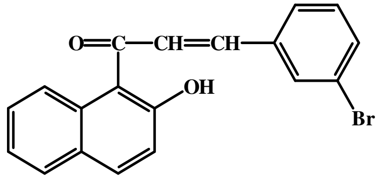
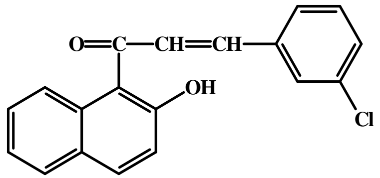
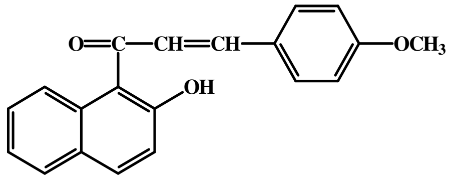
In the present work the chalcones have been prepared by condensing 2-hydroxy-1-acetonaphthone and different aldehydes. These chalcones were characterized and their complexing behaviour with titanium(III) and vanadium(III) has been studied.

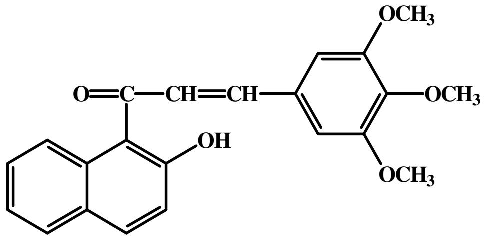
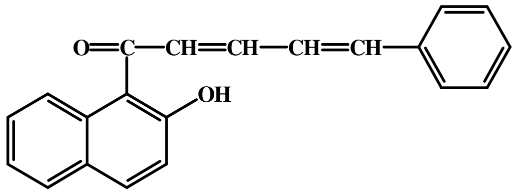
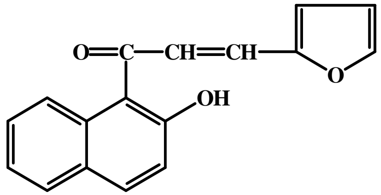
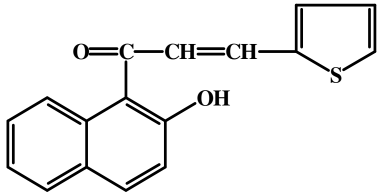
EXPERIMENTAL

Purity of the chalcones was cheked by TLC using benzene:ethanol mixture as mobile phase and characterized by determination of melting points, elemental analyses and NMR spectra. The Ti(III) and V(III) complexes of these chalcones have been prepared and characterized.

Synthesis of chalcones: The mixture of respective aldehydes and 2-hydroxy-1-acetonaphthone or 2-acetylbenzamidazole was stirred in ethanol for 1-2 h, 1.5 mL of 40 % KOH solution was added to it dropwise. The reaction mixture was kept over-night at room temperature. The mixture was then poured into ice cold water containing dil. HCl. The solid compound so obtained was filtered and recrystallized from ethanol (Table-1).

TABLE-1
ANALYTICAL DATA OF THE SYNTHESIZED CHALCONES

S. No.	m.f. (m.w.)	Molecular structure	m.p. (°C)	Elemental analysis (%): Found (calcd.)			IR spectra, (ν_{max}, cm^{-1})
				C	H	S	
1	$C_{19}H_{14}O_2$ (274)		170	83.21 (82.82)	5.10 (4.92)	–	3100 (-OH), 1720 (-C=O), 1640 (-CH=CH)
2	$C_{19}H_{13}O_2Br$ (353)		195	64.58 (63.91)	3.68 (3.12)	–	3110 (-OH), 1715 (-C=O), 1635 (-CH=CH-), 852 (-C-Br-)
3	$C_{19}H_{13}O_2Cl$ (308.5)		190	74.02 (73.77)	4.22 (3.94)	–	3100 (-OH), 1640 (-CH=CH-), 1721 (-C=O)
4	$C_{20}H_{16}O_3$ (304)		180	78.94 (78.67)	5.26 (5.10)	–	3070 (-OH), 1639 (-CH=CH-), 1720 (-C=O)

5	$C_{22}H_{20}O_5$ (364)		175	72.52 (72.13)	5.40 (5.36)	–	3106 (-OH), 1725 (-C=O), 1642 (-CH=CH-)
6	$C_{21}H_{16}O_2$ (300)		185	84.00 (83.68)	5.33 (5.12)	–	3100 (-OH), 1730 (-C=O), 1635 (-CH=CH-)
7	$C_{17}H_{12}O_3$ (264)		178	77.27 (76.94)	4.54 (4.12)	–	3105 (-OH), 1725 (-C=O), 1640 (-CH=CH-)
8	$C_{17}H_{12}O_2S$ (280)		188	72.85 (72.40)	4.28 (3.99)	11.42 (11.02)	3110 (-OH), 1735 (-C=O), 1635 (-CH=CH-)

Synthesis of metal complexes: The complexes were synthesized by the following general method. To a chloroform solution (30 mL) of the corresponding ligand (10 mmol), methanolic solution (20 mL) of the metal chloride (5 mmol) was added with constant stirring. The pH of the reaction mixture was maintained around 7.5 by adding 10 % methanolic solution of ammonia. The resulting metal complex was filtered and washed with chloroform, methanol, petroleum ether and dried over $CaCl_2$ in vacuum dessicator (Table-2).

TABLE-2
CHARCTERIZATION OF Ti(III) AND V(III) CHALCONE COMPLEXES

S. No.	m.f.	m.p. (ºC)	Elemental analysis (%): Found (calcd.)			μ_{eff} (BM)	Molar conductance	
			C	H	M		DMSO	DMF
1	$[Ti(C_{19}H_{13}O_2)_2(H_2O)_2]Cl$	210	68.51	3.90	7.21	1.69	55	80
			64.78	(3.67)	(7.11)			
2	$[Ti(C_{19}H_{12}O_2Br)_2(H_2O)_2]Cl$	215	55.37	2.91	5.83	1.71	55	85
			(54.99)	(2.45)	(4.85)			
3	$[Ti(C_{19}H_{12}O_2Cl)_2(H_2O)_2]Cl$	205	0.62	3.26	6.53	1.70	60	75
			(0.60)	(2.96)	(5.05)			
4	$[Ti(C_{20}H_{15}O_3)_2(H_2O)_2]Cl$	210	0.66	4.13	6.62	1.69	65	80
			(0.55)	(3.90)	(5.85)			
5	$[Ti(C_{22}H_{19}O_5)_2(H_2O)_2]Cl$	210	62.4	4.49	5.68	1.70	65	80
			61.2	(3.95)	(5.25)			
6	$[Ti(C_{21}H_{15}O_2)_2(H_2O)_2]Cl$	205	70.2	4.18	6.69	1.71	70	75
			(69.1)	(4.05)	(6.62)			
7	$[Ti(C_{17}H_{11}N_2O_3)_2(H_2O)_2]Cl$	215	0.63	3.41	7.44	1.70	60	85
			(0.55)	(3.30)	(7.25)			
8	$[Ti(C_{17}H_{11}O_2S)_2(H_2O)_2]Cl$	210	60.2	3.24	7.09	1.71	55	80
			(59.5)	(3.05)	(6.99)			
9	$[V(C_{19}H_{13}O_2)_2(H_2O)_2]Cl$	210	68.26	3.89	7.63	2.86	50	55
			(67.85)	(3.46)	(7.11)			
10	$[V(C_{19}H_{12}O_2Br)_2(H_2O)_2]Cl$	215	55.20	2.90	6.17	2.89	50	60
			(45.95)	(2.65)	(5.85)			
11	$[V(C_{19}H_{12}O_2Cl)_2(H_2O)_2]Cl$	220	61.87)	3.25	6.91	2.88	55	55
			61.35)	(3.02)	(6.45)			
12	$[V(C_{20}H_{15}O_3)_2(H_2O)_2]Cl$	220	65.93	4.12	7.00	2.92	60	75
			(65.45)	(3.89)	(6.85)			
13	$[V(C_{22}H_{19}O_5)_2(H_2O)_2]Cl$	210	62.26	4.48	6.01	2.88	50	70
			(62.02)	(4.25)	(5.65)			
14	$[V(C_{21}H_{15}N_2O_2)_2(H_2O)_2]Cl$	215	70.00	4.16	7.08	2.94	55	65
			(69.25)	(3.87)	(6.62)			
15	$[V(C_{17}H_{11}O_3)_2(H_2O)_2]Cl$	225	0.62	3.39	7.87	2.92	60	55
			(0.45)	(3.15)	(7.25)			
16	$[V(C_{17}H_{11}O_2S)_2(H_2O)_2]Cl$	210	60.00	3.23	7.50	2.94	50	60
			(59.00)	(2.95)	(6.99)			

RESULTS AND DISCUSSION

The melting points of complexes were determined and compared with the corresponding ligand in order to ensure the formation of complexes. The complexes were subjected to microanalyses for the C, H and N. The metals were estimated gravimetrically. These analytical data have suggested 1:2 (M:L) stoichiometry for all the synthesized complexes.

The molar conductance of the complexes were determined at 10^{-3} M dilution and 25 ºC in 2 solvents *viz.*, DMF and DMSO. The values in both solvents suggested 1:1 electrolytic nature for all the complexes.

The magnetic susceptibility of the complexes was determined by Gouy's balance using $CuSO_4·5H_2O$ as calibrant from the measured value of magnetic susceptibility the spin only values of magnetic moments were calculated. These values indicated paramagnetic character and octahedral geometry for all the titanium(III) and vanadium(III) complexes[22,23].

The IR spectra of all the ligands exhibited important bands in the range of 3120-3110 assignable to intramolecularly hydrogen bonded ν(OH) group and 1235-1230 cm^{-1} for ν(C-O)(phenolic) in addition to the band for ν(C=O). In the IR spectra of all the chelates the band due to ν(OH) has disappeared indicating deprotonation of phenolic group in coordination. This has been further supported by upward shift in ν(C-O)(phenolic) to the extent of 40-30 cm^{-1}. The ν(C=O) has shifted to lower frequency suggesting participation of ν(C=O) in coordination[24-26]. The IR spectra of the chelates show peaks around 3300 and 840-820 cm^{-1} due to the presence of coordinated water molecules[27]. The presence of non ligand bands in the IR spectra of the complexes in the region of 600-450 cm^{-1} may be assigned to ν(M-O) vibrations.

The electronic spectra of titanium(III) complexes was recorded in DMF solution. The spectra exhibited a band in the range of 19500-20500 cm^{-1} with a shoulder in the range of 18000-18500 cm^{-1}. The spectral band arises due to the transition $^2T_{2g}\rightarrow{}^2E_g$, which is characteristic of octahedral geometry[28].

The electronic spectra of all the complexes exhibited weak and broad bands in the range of 16700-16500 cm^{-1} with a shoulder in the range of 20500-20900 cm^{-1}. The bands at higher wave numbers were considerably more intense and better resolved of the two. The low energy bands may be assigned to $^3T_{1g}\rightarrow{}^3T_{2g}$ transition in octahedral geometry and high energy band to $^3T_{1g}\rightarrow{}^3T_{1g}$ transition[29].

On the basis of above mentioned facts these titanium(III) and vanadium(III) complexes have been tentatively assigned octahedral geometry.

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