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Synthesis and Characterisation of the Mixed Ligand Complexes, $[M(ox)(caf)_2]$, H_2O, $M=Cu^{2+}$, Zn^{2+}, Mn^{+2}, Fe^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+}; ox=oxalato; caf=caffeine

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ABSTRACT

In this work, new mixed ligand complexes $[M(ox)(caf)_2]$, H_2O , *M=* Cu^{2+}, Zn^{2+}, Mn^{+2}, Fe^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+} *; ox=oxalato; caf=caffeine have been prepared. They have been characterized by molar conductance, IR, UV-Visible, EPR spectroscopy. From analytical and spectral data, the stoichiometry of the complexes has been found to be 1: 1: 2 (metal: ox: caf). The infrared and UV-Visible spectra suggest that the oxalato ligand is bidentate chelate and the caffeine behave as a monodentate ligand with N9 donor towards metal ions. The physico-chemical data suggest octahedral divalent metal complexes of dicaffeine oxalato.*

Keywords: Oxalato complexes, mixed ligand complexes, caffeine, infrared, UV-Visible, EPR spectroscopy, conductivity.

INTRODUCTION

The ligand oxalate can be considered as the dicarboxylate ion. The structure involves two carboxylates groups connected directly through the carbon-carbon bond. Not only this molecule lacks any spacer function between the carboxylate groups but also it has got connectivity through two sp^2 hybridized carbon atoms, forming a perfect planar configuration [1]. The predominant coordination mode is bidentate chelate [2]. This makes the oxalates moitey a very strong chelate ligand through σ-π coordination at six electrons resulting in polymeric metal complexes $[M(C_2O_4)(H_2O)_2]$, M= Co^{2+}, Zn^{2+}, Ni^{2+}, Ru^{2+}… with rigid stuctures and electronic properties [3]. The introduction of the ligands containing N-donors, like 2,2'- bipyridine or 1,10- phenanthroline into the metal-oxalato systems further stabilizes their solid-state structures [4,5].

In this work, it was interesting to examine the influence of the nitrogen atoms in the nitrogen aromatic ligands on the properties of mixed caffeine oxalato complexes. A survey of literature shows that the pyridine, 1,10-phenanthroline, bipyridine oxalato complexes $[M(py)_2(ox)]$ and [M(L)(ox)]; L= bipy ; 1,10-phen ; M= Zn^{2+}, Cd^{2+}, Ni^{2+}, Cu^{2+}, Co^{2+}, Mn^{2+}, Fe^{2+} are well known but the caffeine oxalato complexes are unknown [4,5].
The stable complexes were obtained and isolated in all case bases on the metal analysis data, infrared, UV-Visible spectroscopies, molar conductance and EPR spectroscopy studied, the general formula of the

complexes is $[M(ox)(caf)_2], H_2O$, M= Cu^{2+}, Zn^{2+}, Mn^{+2}, Fe^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+} ; ox= oxalato ; caf= caffeine

MATERIALS AND METHODS

All chemicals were obtained from commercial sources and were used without purifications: ($NiCl_2$, $6H_2O$ BDH; $ZnCl_2$, $2H_2O$ BDH; $CdCl_2$, $1/2H_2O$ Panreac; $CuCl_2$, $6H_2O$ BDH; $MnCl_2$, $2H_2O$ BDH; $CoCl_2$, $6H_2O$ BDH ; $FeCl_2$, $4H_2O$ BDH), oxalate Sigma Aldrich, salicylaldehyde SAFC, Ethanol and DMSO Sigma Aldrich, double distilled water was used.

Infrared spectra were recorded as KBr pellets on a JASCO FT-IR 660 plus Spectrophotometer in the range of 4000-400 cm^{-1} at 298 K while the electronic spectra (UV Visible) were obtained on a Shimadzu UV-1800 Spectrophotometer. The EPR spectrum was recorded on a conventional X band Bruker ER 200D spectrometer operating at 9.5 GHz. Conductivity measurements were performed at 25°C in DMSO using Hach HQ430d flexi.

Synthesis of the aquo oxalato complexes $[M(ox)(H_2O)_2]$; (Where M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}) : The aquo oxalato complexes $[M(ox)(H_2O)_2]$; M= Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} were prepared by the reaction of MCl_2, xH_2O with $K_2C_2O_4$ [6-8]. The synthesis of these complexes is relatively simple by direct reaction of aqueous solution of a potassium oxalate and metal chloride in ethanol or methanol, in nitrogen atmosphere. For $Zn(AC)_2$, $2H_2O$ and $K_2C_2O_4$, the reaction can be performed at room temperature [9], whereas of metal chloride ($NiCl_2$, $6H_2O$.) it is necessary to boil the mixture by refluxing for 3-15 h. After completion of the reaction, the precipitated crystalline or powder complexes were separated by filtration, washed with small portions of hot water and finally air dired.

Synthesis of the caffeine oxalato $[M(ox)(caf)_2]$, H_2O; M= Cu^{2+}, Zn^{2+}, Mn^{+2}, Fe^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+} : The caffeine oxalato complexes $[M(ox)(caf)_2]$, H_2O M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, caf = caffeine were obtained by direct reaction of ethanolic solution of the caffeine and that of the aquo oxalato complexes $[M(ox)(H_2O)_2]$ M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} at 50-60 °C under continuous stiring for 3-6 h. Then, the resulting products were filtered and washed with small portions of hot water and finally air dired.

RESULTS AND DISCUSSION

Characterization of the complexes $[M (ox)(H_2O)_2]$ and $[M(ox)(caf)_2]$ H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} : The physico-chemical characteristics of the synthesized complexes are given in the table 1. The complexes are soluble in DMF and DMSO. The observed molar conductance of the complexes in DMF for 10^{-4} M solutions at room temperature are consistent with the non-electrolytic nature of the complexes.

Table 1 : Physico-chemical data of the complexes

Complexes	Colour	M.P. (°C)	Yield (%)	Λ (Ohm^{-1} cm^2 mol^{-1})
$[Zn(ox)(H_2O)_2]$	White	>260	52	12.65
$[Cd(ox)(H_2O)_2]$	White	>260	43	12.14
$[Cu(ox)(H_2O)_2]$	Blue	>260	48	11.95
$[Ni(ox)(H_2O)_2]$	Green	>260	56	12.23
$[Mn(ox)(H_2O)_2]$	White	>260	50	11.88
$[Co(ox)(H_2O)_2]$	Pink	>260	40	11.90
$[Fe(ox)(H_2O)_2]$	Yellow	>260	41	11.53
$[Zn(ox)(caf)_2],H_2O$	White	>260	54	10.12
$[Cu(ox)(caf)_2]$,H_2O	Blue	>260	50	10.28
$[Mn(ox)(caf)_2]$,H_2O	White	>260	56	10.35

$[Fe(ox)(caf)_2]$,H_2O	Yellow	>260	43	10.18
$[Cd(ox)(caf)_2]$,H_2O	White	>260	46	09.98
$[Co(ox)(caf)_2]$,H_2O	Pink	>260	42	11.02
$[Ni(ox)(caf)_2]$,H_2O	Green	>260	50	10.66

Infrared spectroscopy of the complexes $[M(ox)(caf)_2]$, H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} : The transition metal diaquo oxalato complexes have been studied for infrared, Raman, X-Ray structure and thermal decomposition.

The assignments of the seven studied caffeine oxalato complexes $[M(ox)(caf)_2]$, H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} are reported in table 2. The infrared spectrum of $[Co(ox)(caf)_2]$, H_2O is given in figure 2.

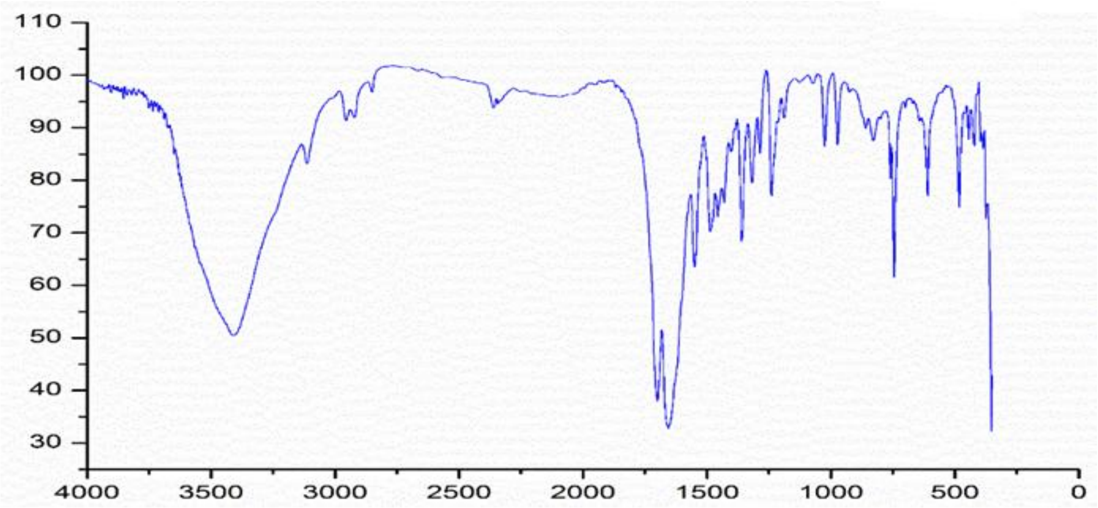

Figure 2: Infrared spectrum of the oxalato caffeine complex $[Zn(ox)(caf)_2]$ in KBr

Table 2: Characteristic IR frequencies in (cm^{-1}) of the caffeine oxalato complexes $[M(ox)(caf)_2]$, H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} in KBr

C_2O_4	**Caf**	$[Fe(ox)(caf)_2]$,H_2O	$[Cd(ox)(caf)_2]$,H_2O	$[Co(ox)(caf)_2]$,H_2O	$[Ni(ox)(caf)_2]$,H_2O	$[Zn(ox)(caf)_2]$, H_2O	$[Mn(ox)(caf)_2]$,H_2O	$[Cu(ox)(caf)_2]$,H_2O	**Attribution**
3400		3342	3414	3422	3400	3412	3423	3445	υ(H_2O)
	3114	3100	3102	3102	3102	3100	3103	3100	υ(CH)
	2954	2950	2947	2950	2947	2949	2948	2946	υ(CH_3)
	1700	1696	1697	1700	1693	1697	1696	1700	υa(CO)
	1660	1650	1652	1648	1651	1653	1646	1653	υs(CO)
1632		1625	1614	1626	1628	1624	1628	1625	υa(CO)
	1600	1580	1580	1580	1578	1576	1578	1580	υ(C=C)+ υ(HCN)
	1550	1540	1540	1540	1540	1546	1540	1540	δ(HCN) + υ(imid)+ υ(pyr)
	1470	1477	1480	1483	1477	1478	1476	1479	δ(HCN)+ δ(CH_3)

	1456	1445	1446	1443	1447	1450	1443	1443	υ (CH_3)+ δ(CH_3)
1433	-	1361	1376	1371	1360	1378	1379	1356	υs(CO) + υ(C-C)
	1380	1393	1398	1377	1378	1376	1376	1395	δ(CH_3)+ υ(imid)
	1360	1352	1354	1350	1353	1349	1353	1356	δ(HCN)+ υ(imid)+ δ(CH_3)
	1331	1316	1318	1318	1317	1314	1317	1317	υ(imid)+ υ(pyr)
1322	-		1313	1315	1316	1316	1312	1318	υs(C-C) + δ(O-C=O)
	1285	1280	1280	1284	1282	1280	1280	1280	υ(pyr)
	1237	1238	1240	1239	1238	1237	1238	1240	υ(CN) +ρr(CH_3)
	1210	1193	1217	1197	1193	1188	1192	1194	δ(CH) + ρr(CH_3)
	1025	1023	1024	1023	1027	1023	-	1025	ρ(CH_3) + ρ(CH)
	973	978	977	978	976	976	975	978	υ(NCH_3)+ ρr(CH_3)+ δ(imid)
	923	922	921	923	926	924	924	922	γ(CH)
	862	860	840	823	827	860	860	858	ρ(CH3)+ υ(NCH3)+ δ(C=O)
	800	800	800	800	793	796	796	798	ρ(pyr) + δ(C=O)
785	-		618	617	615, 594	617	615	618	υ(M-O) + δ(O-C=O)
	745	742	742	742	742	742	742	743	ɤ(pyr)+ ɤ(imid)+ Ʈ(caffeine)
	642	640	640	643	641	641	643	640	δ(pyr)+ ρ(imi)
	611	614	615	615	614	613	613	615	ɤ(imidazol)
519			514, 498	486	480	502, 496	496, 460	500, 460	υ (C-C)
	481	479	479	478	478	477	479	480	Ʈ(caffeine)
	450	443	440	448	453	446	450	440	δring(pyr)
		530	510	510	510	490	510	510	υ(M-N)
		416	415	418	417	418	417	416	υ (C=O)

The characteristic frequencies of the free oxalato, caffeine ligand and their complexes [M(ox)(caf_2)], H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} were readily assigned based on comparaison with the littérature [10,11]. Upon substitution of water coordination by caffeine leads to the appearance of the band of coordinated caffeine and disappearance of the stretching and roking vibrations of the coordinated water ligand.

The carbonyl group in the caffeine oxalato complexes exhibit a strong absorption band in the (1693-1700) cm^{-1} range due to υ(CO) asymmetric. Another strong band in the (1646-1653) cm^{-1} range belong to υ(CO) symmetric and υ(C=N) is shifted to lower frequencies by (7-14) cm^{-1}, compared with the free caffeine, indicating coordination of the caffeine through the azomethine nitrogen atom (N9) [12]. In addition, the band at 1550 cm^{-1} is assigned to (δHCN+υring imid + υring pyrimi) which is shifted to lower frequencies by (6-10) cm^{-1} compared with the free caffeine.
Another set of a strong band was also observed in the (1628-1614) cm^{-1} range due to the asymmetrical νas(COO^-) stretching vibration and νs(COO^-) in the (1379-1356) cm^{-1} range [13]. Well compared to the

difference ν= νas (COO$^-$)- νs (COO$^-$) of the metal diaquo oxalato complexes gave little modified [14]. The Δν values and there of the Literature of the complexes [M (ox)(L)$_2$] ; L= pyridine, bipyridine, 1,10-phenanthroline suggest the bidentate chelate coordination of oxalato ligand which is conserved [15-17]. The band of oxalate ligand at 1322 cm^{-1} which involves the νs(C-C)+δ(OCO) stretching as the major coordinate shifts to (1312-1318) cm^{-1} region for all caffeine oxalato complexes [18].

New bands are attributed to υ (M-O) and υ(M-N) vibrations, respectively. The appearance of these new bands vibrations in (490-530) cm^{-1} and (415-418) cm^{-1} range indicates that the caffeine and the oxalate coordinated to metal [19,20].

Electronic spectra of the complexes [M(ox)(caf)$_2$], H$_2$O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}: The electronic spectra of the caffeine oxalato complexes were recorded in their solution in DMSO in the (200-1000) nm range. The spectra of the free ligands (the oxalate and the caffeine) showed two and three bands (Table 4), suggesting the presence of π –π* and n–π* transitions.

The electronic spectra of the Zn(II) and the Cd(II) caffeine oxalato complexes display three bands in the (220-330) nm region. The first two bands are due to π –π* and n–π* transitions but the last band is attributed to the charge transfer [21-23].

The electronic spectrum of the Ni(II) caffeine oxalato complex shows two d-d absorption bands at 612 nm and 878 nm, which are due to to $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ and $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ [21,24] transitions, indicating octahedral geometry.

The Co(II) caffeine oxalato complex shows two d-d absorption bands in the visible region at 424 nm and 895 nm, attributed to $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ and $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ transitions [21,25], suggest the octahedral geometry of Co^{2+}.

The Cu(II) caffeine oxalato complex shows a broad band at 634 nm which can be assigned to $^2B_{1g} \rightarrow {}^2A_{1g}$ transition [19,26]. This band refers to the distorsion of octahedral geometry.

The Fe(II) oxalato caffeine complex exhibits an absorption band at 487 nm which belong to electronic transition $^6A_{1g} \rightarrow {}^4T_{1g}$ [26,27], agreement with octahedral geometry around Fe^{2+}.

The electronic spectrum of the Mn(II) caffeine oxalato complex shows two absorption bands at 396 nm and 437 nm assignable to the $^6A_{1g} \rightarrow {}^4T_{2g}$ and $^6A_{1g} \rightarrow {}^4T_{1g}$ transitions [28,29] consistent with an octahedral geometry for Mn^{2+}.

Table 4: UV-Visible data of the caffeine, the oxalate and their complexes [M(ox)(caf)$_2$], H$_2$O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} in DMSO

	λmax (nm)	**Assignment**
caf	275 316 365	π→π* n→π* n→π*
xo	250 283	π→π* n→π*
[Ni(ox)(caf)$_2$] ,H$_2$O	250 314 612 878	π→π* n→π* $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$

$[Co(ox)(caf)_2]$,H_2O	241 278 401 645	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ $^4T_{1g} \rightarrow {}^4A_{2g}$
$[Cu(ox)(caf)_2]$,H_2O	234 283 423 634	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ CT $^2B_{1g} \rightarrow {}^2A_{1g}$
$[Fe(ox)(caf)_2]$,H_2O	232 276 329 487	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ CT $^6A_{1g} \rightarrow {}^4T_{1g}$
$[Mn(ox)(caf)_2]$,H_2O	227 273 316 396 437	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ CT $^6A_{1g} \rightarrow {}^4T_{2g}$ $^6A_{1g} \rightarrow {}^4T_{1g}$
$[Zn(ox)(caf)_2]$,H_2O	227 273 326	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ M →L
$[Cd(ox)(caf)_2]$,H_2O	237 283 308	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ M →L

EPR spectra of the $[Fe(ox)(caf)_2]$, H_2O: The solid state EPR spectrum of the $[Fe(ox)(caf)_2]$, H_2O complex is displayed at room temperature on X band at frequency 9.7 GHz under the magnetic field strength 3300 G. ESR spectrum of the complex shows only an intense and broad signal without hyperfine splitting (giso = 2.128). The shape of the spectrum is consistent with octahedral environment around Fe(II) ion. The higher g value for the complex, when compared to that of free electron (g = 2.0023) revealing an appreciable covalency of metal ligand bonding characteristic of octahedral stereochemistry [30].

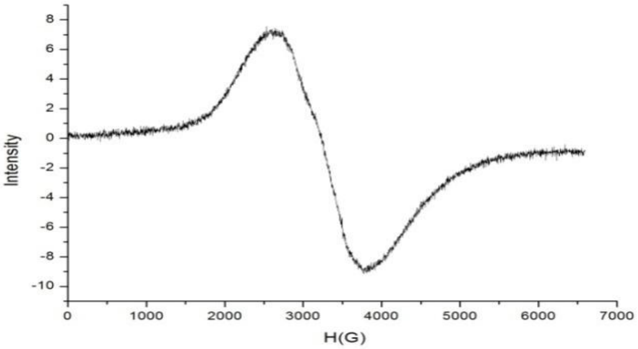

Figure 9: The solid state EPR spectrum of $[Fe(ox)(caf)_2]$, H_2O at room temperature on X band

CONCLUSIONS

In this article, the oxalato complexes of Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} with caffeine ligand were synthesized and characterized. On the basis of conductivity measurements, infrared, UV-Visible, EPR spectral data, the formulae of the complexes are $[M(ox)(caf)_2]$, H_2O; M= Zn^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}. The results suggest the octahedral symmetry for the all complexes which the oxalato may act as bidentate ligand and the caffeine is coordinated as a monodentate ligand.

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