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Preparation, Spectra and Biological Properties of Transition Metals ((III) and (II) Mixed - Ligand Complexes with 5-Chlorosalicylic Acid and L-Valine

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Abstract :

Mixed ligand metal complexes of Cr^{III}, Fe^{III,II}, Ni^{II} and Cu^{II} have been synthesized using 5-chlorosalicylic acid (5-CSA) as a primary ligand and L-Valine (L-Val) as secondary ligand. The metal complexes have been characterized by elemental analysis, electrical conductance, magnetic susceptibility measurements and spectral studies. The electrical conductance studies of the complexes indicate their electrolytic nature. Magnetic susceptibility measurements revealed paramagnetic nature of the all complexes. Bonding

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of the metal ion through $-OH^-$ and $-COO^-$ groups of bidentate to the 5-chlorosalicylic acid and through $-NH_2$ and $-COO^-$ groups of bidentate to the L-valine by FT-IR studies. The agar diffusion method has been used to study the antibacterial activity of the ligands and complexes against the pathogenic bacteria *E.Coli*, *Pseudomonas*, *Bacillus* and *Staphylococcus*.

Key words : 5-Cl-Salicylic acid, L-Valine , Mixed ligand complexes, Elemental analysis, Spectral studies and Antibacterial activities .

1. Introduction:

Salicylic acid (5-CSA) (likewise recognized such as 2-hydroxy benzoic acid) is a simple carboxylic aromatic acid; it includes both a hydroxy group as well a carboxy group. It is vastly applied at food manufacture such as a plant growth organizer, such as ingredient at antimicrobial wash to prevent damage from fresh vegetables as well like a preservative on products of food (antimicrobial agent also antifungal) [1-3]. It has lately be catchy to the experiments because of biological importance, specially on medication also chemistry [4-6]. A number of experiments approach were made at try to expound the biological effect for salicylates. One interpretation of these impacts has demonstrated the ability to bind metal salicylates [7]. Chlorosalicylic acid has been studied so extensively as unsubstituted salicylic acid. Cu(II) complexes for (5-CSA) [8] as well some lanthanide complexes for (5-CSA) as well found which anion co-ordinated like bidentate ligand [9]. Among these, compounds the metal for carboxylates were studied intensively because the carboxyl group may be coordinated with metal ion at different methods. [10]. In particular, copper compounds of salicylate are gaining increasing of both structural also biological point for view. Salicylic acid has been known for its own therapeutic for centuries [11]. Salicylic acid also its derivatives are known to be nonsteroidal anti-inflammatory, anti-pyretic also analgesic. Copper compounds for antiinflammatory drugs were offered to be more effective as well eligible than drugs compared into same ligands [12]. In addition, some of its pharmacological activities have been reported at existence from ancillary nitrogen- donor ligands [13]. Cu^{II}, was offered to work at a synergistic method for salicylic acid derivatives [14].

L-Valine (L-Val) is an α -amino acid, It is one branched protein-organic amino acids vastly distributed but seldom exists at an amount greater than 10%. It is a chain of amino acid branching as well may be derived of alanine through introduction two methyl group existing at α -carbon atom [15-16]. Mixed ligand complexes play an significant role in the biological process [17-18]. Amino acids including active $-NH_2$ and -COOH groups are well known with tendency to the form compounds for metals as well have large importance in the biological also pharmaceutical fields also are directly involved in the all enzymatic metabolic activities from organism [19-21]. In present paper, the synthesis, physical spectroscopic properties of the Cr^{III}, Fe^{III,II}, Ni^{II} and Cu^{II} complexes of mixed

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ligands(5- CSA) and (L-Val) and the present work deals with the study of the antibacterial of these reported compounds .

Material and Methods:

Experimental: All chemical were of reagent grade quality and were purchased of L-Val. and 5- CSA from commercial sources (BDH and Merck), metals salts and solvents from (B.D.H., Riedel and Merck). They were used without further purification.

Physical characterization: The measured molar conductance values (Λ_m) in DMF solution from compounds have been measured on 25°C with 10⁻³M solution from samples utilizing PW9527 Digital Conductivity Meter (Philips) . The infrared (FT-IR) spectra were recorded in 4000-400 cm⁻¹ rang of ligands and 4000-200 cm⁻¹ of metal complexes on a Shimadzu IR-470 Spectrophotometer using KBr pellts . The electric spectra of the complexes in DMF solution (10⁻³) were monitored at 25 °C in the 200-1100 nm rang with a Shimadzu-U.V-160.Altra Violet-Visible- Spectrophotometer for 1.000±0.001 cm correspond quartiz cell . C, H and N content of the complexes were determined by the micro analytical unit of Eurverctor EA 3000A Elemental Analyzer . When metal includes from complexes have been specified through Atomic Absorption (A.A) Technique / Flame Emission Spectrophotometer employing AA – 680 Shimadzu . Magnetic susceptibility measurements have been measured utilizing Bruker magnet BM6 instrument on 298 °K following the Faraday's manner . Melting points have been determined through employing Stuart Scientific melting point apparatus .

Preparation the Complexes [M(5-CAS)₂(L-Val)] M=Cr^{III},Fe^{III,II}, Ni^{II} and Cu^{II}: The synthesis of the ligands solutions , 5-CAS and L-Val were prepared of reported method in literature [22]. Solution of CrCl₃.6H₂O[0.267gm, 1mmol], FeCl₃[0.1622gm, 1mmol], (NH₄)₂Fe(SO₄)₂ [0.2841gm, 1mmol], NiCl₂.6H₂O [0.237gm, 1mmol], and CuCl₂.H₂O [0.134gm, 1mmol] in the stoichiometric ratio metal: ligand[M:2(5-CAS) (L-Val)],(1:2:1). The mixtures were heated at a water bath to reflux for a period of one hour with all complex at 10 °C. The precipitated complexes were thereafter filtrated off and washed for ether followed through desiccation on room temperature and analyzed employing standard manners .

In Vitro Antibacterial Activity: All the metal complexes, ligands have been checked versus *Bacillus* and *Staphylococcus* (gram positive) and *E. Coli* also *Pseudomonas* (gram negative) bacteria through utilizing wall agar diffusion method employing solvent (DMF); condensation for ligands and complexes on reveal was (10^{-3} M) through employing disc sensitivity check. The manner includes reveal for zone inhibition toward

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diffusion for micro-organism at an agar plate. Plates have been brood with 24 hours from bacteria on 37°C.

Results and Discussions :

The analytical date and some of the physical properties are collected of the aqueous solution of the metal ions and ethanolic solution of (5-CSA) and the ethanolic solution of (L-Val) in Table 1. The complexes soluble in H₂O, CH₃Cl, CH₃OH , C₂H₅OH, (CH₃)₂CO, DMF and DMSO but non soluble in CCl₄ , C₆H₆ and (CH₃CH)₂O . Elemental analysis data Table- 1. From metal complexes are fixed for their general formula at (M:2(5-CSA):(L-Val)] as 1:2:1 mixed ligand complexes. Molar conductance values from complexes at DMF on 10⁻³M condensation and type of electrolyte for each compound could be determined [23] .

Table	1.	A	4.001]	Distant	Char			C 1	I income		Com	10
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Compounds	M.wt.	Color	M.P [°] C	Analysiz Calc. (Found		nd)	
				M%	С%	Н%	N%

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(L-Val)	117.15	White	295-300	-	-		
Compounds	172.57 v(N	H_2 + v_a	171-173 s(COO) 1	v _s (COO)	บ(M-	N) v	(M-O)
	(v	DH)					
$Na_2[Cr(5-C(A)_2(V-Val)]$	555.2312	4 br _{Deep} 1	504 <u>sh</u> 50	136 losh.	41.10 -	2.90	- 2.52
		green	Dec.	(8.59)	(40.77)	(2.48)	(1.59)
Na ₂ [Fe(5-CSA) ₂ (L-Val)]	559.08	Pale	>250	10.03	40.82	2.88	2.51
		brown	Dec.	(9.86)	(40.07)	(2.19)	(2.12)
Na ₃ [Fe(5-CSA) ₂ (L-Val)]	582.07	Deep	>250	9.59	39.21	2.77	2.41
		brown	Dec.	(8.93)	(38.71)	(2.20)	(1.70)
Na ₃ [Ni(5-CSA) ₂ (L-Val)]	584.91	Pal green	>250	10.03	39.02	2.76	2.39
			Dec.	(9.31)	(37.34)	(2.37)	(2.37)
$Na_3[Cu(5-CSA)_2(L-Val)]$	589.77	Deep	>250	10.78	38.69	2.76	2.38
		green	Dec.	(8.08)	(37.62)	(1.44)	(2.93)

Dec. = **Decomposition**

IR Spectral Study:

The characteristics frequencies of FT-IR spectral for free ligands and mixed ligand complexes from metals are given in Table -2. And the main parts to the IR spectral are presented to the (5-CSA) and (L-Val) ligands and Fe^{III, II} complexes in Figure -1. IR spectrum from (L-Val) displayed broad band on 3124 cm⁻¹ due into stretching mode from $v(NH_2)$ and v(OH), in complexation a shift for change at shape have been watched of this band, when rising at intensity was observed [24]. Bands on 1504 cm⁻¹ also 1361 cm⁻¹ in amino acid spectrum were assigned to $v(COO^{-})$ asymmetric and symmetric respectively, suffered a great change in position and at density at complexation for metal ion [25]. IR spectrum of (5-CSA) appeared wide band on 3232 cm⁻¹, that has been specified into stretching vibration from v(OH) group. The band was absent at spectral from prepared complexes, that specified deprotonation as well participation from enol oxygen on chelation [26]. Bands on 1608 cm⁻¹ also 1361 cm⁻¹ were attributed into $v(COO^{-})$ asymmetric and symmetric respectively [14], these bands shifted in position to higher and lower frequency in the spectra of all prepared complexes which indicated the coordination for metal ion. The manifestation for new bands at region around 428-563 cm^{-1} are temporarily appointed into v(M-N) also v(M-O) (Metal-Ligand) stretching bands[27-28].

Table - 2: Important IR Spectral Bands (cm⁻¹) for Ligands also Complexes

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(5-CSA)	3232 br.	1608 sh.	1361 sho.	-	-
Na ₂ [Cr(5-CSA) ₂ (L-Val]	br.3425	1573 s.	1313 s.	543 w.	484 w.
	3321 br.				
	-				
Na ₂ [Fe(5-CSA) ₂ (L-Val]	br.3444	1581 s.	1311 s.	486 w.	459 w.
	3383 br.				
	-				
Na ₃ [Fe(5-CSA) ₂ (L-Val]	br.3352	1597 s.	1307 s.	493 w.	428 w.
	3140br.				
	-				
Na ₃ [Ni(5-CSA) ₂ (L-Val]	br.3452	1581 s.	1311 s.	563 w.	443 w.
	br.3363				
	-				
Na ₃ [Cu(5-CSA) ₂ (L-Val]	br.3298	1550 sh.	1319 sh.	532 w.	466 w.
	3251 br.				
	-				
br.= broad, sh.=sharp, s.=stro	ng, sho.=shoulder	r, w.= weak	•	•	
	-				

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Figure-1: FT – IR Spectra of Ligands and Fe^{III,II}- Complexes

Electronic Data:

Electronic spectral for ligands as well their complexes have been registered on DMF as indicate in Table-3. The UV spectral for free ligand of L-valine show peak on 349 nm (28653 cm^{-1}) attributed into $(\pi - \pi^*)$ electronic transition [29], while electronic spectral from (5-CSA) appeared peak at 301 nm (2.332cm⁻¹) attributed into $(\pi - \pi^*)$ electronic transition[30].Electronic spectral for Cr^{III} complex gave absorption peaks on 272 nm also 305 nm related into ligand felid, thereafter other three peaks on 762 nm, 834 nm also 942 nm have been specified into electronic transition type ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(P)$, ${}^{3}A_{2g}(F) \rightarrow$ ${}^{3}T_{1g}(F)$ and ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)$ respectively and μ_{eff} value is 3.81 B.M. pointing three unpaired electrons close into spin only value, which agreement of the structure of Cr^{III} to be an octahedral geometry [31]. The spectral for Fe^{III} complex appeared peak on 299nm indicated to ligand felid, thereafter other peaks at 444nm and 824nm were caused by electronic transition ${}^{6}A_{1g} \rightarrow {}^{4}T_{2g}(G)$ and ${}^{6}A_{1g} \rightarrow {}^{4}T_{1g}(G)$ respectively. The value of the measured magnetic moment for [B₄] complex was (5. 76 B.M.) which could be related to high-spin d⁵ ferric ion in octahedral geometry. [32]. The spectral for Fe^{II} complex offered peaks on 298 nm also 463 nm due into ligand field and charge transfer respectively. Other peak at 747 nm assigned to ${}^{5}\text{Eg} \rightarrow {}^{5}\text{T}_{2}\text{g}$ and the μ_{eff} value is 5.64 B.M. suggest octahedral geometry[33]. The spectral for Ni^{II} complex offered peak on 300 nm due into ligand felid. Other peak on 811 nm has been found to be caused through (d-d) electronic transition type³A₂g(F) \rightarrow ³T₁g(F) and the µeff value is 2.78 B.M. pointing out octahedral geometry about Ni^{II} ion [34]. Spectral with Cu^{II} complex gave absorption peak on 300 nm due into ligand felid. The peak on 771 nm has been caused through electronic transition²Eg \rightarrow ²T₂g and the μ_{eff} of this complex was found at 1.68 B.M that was very close into octahedral environment [35].

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Compounds	$\lambda_{max}(nm)$	ABS	Wave	€ _{max}	$\Lambda_{\rm m}$	$\mu_{\rm eff}$
			number	(L.mol-1.cm-1)	$(S.cm^2.mol^{-1})$ in	(B.M)
			(cm ⁻¹)		DMF(10 ⁻³ M)	
(L-Val)	349	0.018	28653	18	2.82	-
(5-CSA)	301	2.332	33222	2332	7.90	-
Na ₂ [Cr(5-CSA) ₂ (L-Val]	272	0.226	36764	226	92.40	3.81
	305	0.243	32786	243		
	762	0.024	13123	24		
	834	0.021	11990	21		
	942	0.021	10615	21		
$Na_2[Fe(5-CSA)_2(L-Val)]$	299	2.422	33448	2422	88.60	5.76
	444	0.245	22522	245		
	824	0.012	12135	12		
Na ₃ [Fe(5-CSA) ₂ (L-Val)]	298	2.417	33557		90.80	5.64
	463	0.643	21598	643		
	747	0.016	13386	16		
Na ₃ [Ni(5-CSA) ₂ (L-Val)]	300	2.313	33333	2313	85.80	2.78
	811	0.009	12330	9		
Na ₃ [Cu(5-CSA) ₂ (L-Val)]	300	2.342	33333	2342	82.60	1.68
	771	0.026	0.026	26		

Table -3.	Reflectance	Datum &	& Magnetic	Moment for	Metal (Complexes
I UNIC O	munumu	Datum	a magnetic	10110110110101	THE COLL V	JumpicAco

Based on the resulting data from spectral analysiz of the ultraviolet and infrared according of the following structures (figure-2 and 3) were proposed.



Figure-2: Structure for M^{III} ComplexesFigure-3: Structure for M^{II} Complexes $M^{III} = Cr^{III}$ and Fe^{III} $M^{II} = Fe^{II}$, Ni^{II} and Cu^{II}

The above structures were suggested according to the following conditions of the reactions:

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$$M^{III} + 2(5-CSA) + (L-Val) \xrightarrow{NaOH} Na_2[M(5-CSA)_2(L-Val)]$$

$M^{II} + 2(5-CSA) + (L-Val)$ Na₃[M(5-CSA)₂(L-Val)]

Antibacterial Activity:

The ligands and complexes were screened in vitro with antibacterial efficacy versus pathogenic bacterial species utilizing disc diffusion method. The outcomes for bacterial checking from synthesized compounds are registered at Table- 4. and Chart -1. As expected, growth inhibition was observed for DMF. The ligands have moderate activity with *Bacillus*, *E. Coli* and *Pseudomonas* and are no active in comparison for *Staphylococcus* for Fe^{II}, Ni^{II} and Cu^{II} complexes. They have high activity Gram negative approximately of the Cr^{III} complex with against Gram positive with *Bacillus*.

Table -4. Outcomes for Antibacterial Efficacy Checking from Ligands also Complexes

Compounds	E. Coli	Pseudomonas	Bacillus	Staphylococcus
	(G-ev)	(G-ev)	(G+ev)	(G+ev)
Control (DMSO)	11	11	-	10
(L-Val)	15	15	10	11
(5-CSA)	11	11	15	-
Na ₂ [Cr(CSA) ₂ (L-Val]	14	20	18	13
Na ₂ [Fe(CSA) ₂ (L-Val)]	11	11	13	15
Na ₃ [Fe(CSA) ₂ (L-Val)]	12	-	13	-
Na ₃ [Ni(CSA) ₂ (L-Val)]	15	16	15	-
Na ₃ [Cu(CSA) ₂ (L-Val)]	12	18	10	-

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Chart - 1: Graphical representation of Ligands and Complexes against tested Becteria *Bacillus*, *Staphylococcus*, *E. Coli* and *Pseudomonasat*.

Conclusions

On this paper, we reported the synthesis, spectroscopic also structural study for complexes from Cr^{III}, Fe^{III,II}, Ni^{II} and Cu^{II} ions employing (5-CSA) like a primary ligand as well (L-Val) like a secondary ligand. It may be thus deduced that (5-CSA) is bidentate ligand into all the status tested coordinating during its –OH and –COO⁻ groups and (L-Val) acts as a bidentate ligand via coordinating by its –NH₂ and –COO⁻ groups at FT.IR. spectrum study, visible spectra and magnetic moments all complexes were high spin octahedral stereochemistry. The srtucture in Figure-4 is proposed for the Cu^{II} complex . The current work deals for the antibacterial study of these reported ligands and complexes. The results of biological screening references compared with ligands using four types of bacteria . The outcomes to the biological screening point out that at some complexes, as in case antibacterial efficiency in all its kinds for the Cr^{III} complex is more active than free ligands and rest for complexes, as well as with the complexes increased efficiency to the complexes may be expounded on basis for chelation theory.

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Fig.(5) :Structure of Cu^{II} complex as 3D model

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