Synthesis, Characterization And Evaluation of Bacterial Activities Of Anew Vanadyl (II), Antimony (III) Complexes Of vitamine-C- Derivatives

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Abstract

Reaction of vanadyl (II) sulphate or antimony trichloride with 2,3,5,6-O,O,O,O-tetraacetic acid-L-ascorbic acid (L_1), 5,6,-O-isopropylidene-2,3-O,O-acetic acid-L-ascorbic acid (L_2) and methylidene-carboxyl-5,6-O-isopropylidene-L-ascorbic acid (L_3) in ethanol as solvent. The formed of complexes were characterized by metal content analysis, elemental analysis and electronic vibrational spectral studies. Molar ratio method showed that the two ligands (L_1) and (L_2) binds through the C-3-CH_2COOH. The ligand (L_3) was bind to metal through carboxylate group. The vanadyl and antimony complexes posses square pyramide arrangement around metal. The complexes were screened for their antibacterial activity against *Staphylococcus aureu* and *Escherichia coli* showed various activities.

Key words: Synthesis, Evaluation of Bacterial, vanadyl, antimony, and vitamine-C-derivatives.

Introduction

There is a long-standing chemical interest in the interaction of metal ions with L- ascorbic acid[(1)]. Authentic metal ascorbates have been isolated and characterized[(2-8)]. Our famillarity[(1,9)] with preparation of new derivatives of L-ascorbic acid and their complexes prompted us to extend this area by prepararing six complexes formed by the reaction of VO(V) and Sb (III) with three ligands derived from L-ascorbic acid.

Experimental

Materials

All chemicals were purchased from BDH, and used without further purifications.

Instrumentation

1. Infra-red spectra between (400-4000 cm^{-1}) were performed with (FT-IR) 8300 Shimadzu spectrophotometer.
2. The electronic spectra were recorded on the UV-Visible spectrophotometer type (spectra 190-900) nm CECIL, England, using water as a solvent.
3. The melting point was recorded on "Gallen kamp melting point Apparatus".
4. The conductance measurements were recoded on W.T.W. conductivity meter.
5. Elemental analysis for carbon, hydrogen was using a Euro Vector EA 3000 A Elemental Analysis (Italy). This analysis was done in at AL-al-Bayt University, Al- Mafrag, Jordan.
6. Metal analysis. The metal contents of the complexes were determined by atomic absorption technique. Using a Shimadzu PR-5. ORAPHIC PRINTER atomic obsorption spectrophotometer.

Synthesis

A. preparations of ligands and their complexes

The ligands L_1, L_2 and L_3 were prepared according to the published methods[(1,9)], and checked by ^1H, ^{13}C-NMR and IR spectra.

B. preparation of 2,3,5,6 tetra acetic acid L-ascorbic acid vanadyl sulphate.

All complexes were prepared as follows: To a solution of 0.408 g. (1 m mole) (L_1) in (20 ml) ethanol the solution of 0.163 g. (1 m mole) of vanadyl sulphate in (20 ml) of ethanol was added. The solution was warmed and stirred for one hour, then filtered. The filterate was evaporated dryness to give the product. Recrystallization from (10 ml) methanol gave green crystalline residue, decomposed at 30 D°, yield 86%. A similar method was followed for the preparation of other complexes; L_2VO, L_2 (0.332 g 1 m mole), $VOSO_4$ (0.163 g, 1 m mole), (40 ml) ethanol (10 ml) methanol, decomposed at 140 D°, yield 95%. L_3VO, L_3 (0.272 g, lm mole), $VOSO_4$ (0.163 g, 1 m mole), (40 ml) ethanol (10 ml) methanol, decomposed at 170 D°, yield 80%. The physical properties of all complexes are listed in Table (1).

Table (1): The physical properties for synthesized ligands and its complexes

Compound	Colour	M.P.C° or D°	Yield %	(CalC), Found.		
				C	H	Metal
L_1	brown	138-139 C°	85	–	–	–
$[L_1VO.2H_2O]SO_4.6H_2O$	green	130 D°	86	(23.49) 23.20	(4.47) 4.10	(7.13) 6.90
$[L_1SbCl_3]7H_2O$	brown	160 D°	90	–	–	(15.90) 16.80
L_2	brown	109-110 C°	92	–	–	–
$[L_2VO.2H_2O]SO_4.H_2O$	brown	140 D°	95	(28.51) 27.85	(3.65) 3.94	(9.32) 9.10
$[L_2SbCl_2H_2O]Cl$	brown	170 D°	88	–	–	(21.64) 21.80
L_3	brown	Semisolid	92.6	–	–	–
$[L_3VO.2H_2O]SO_4$	green-brown	170 D°	80	(29.13) 28.90	(3.09) 3.55	(11.25) 11.60
$[L_3SbCl_2H_2O]Cl.4H_2O$	brown	190 D°	90	–	–	(20.54) 19.70

C. Synthesis of 2,3,5,6 tetra acetic acid L-Ascorbic acid antimonytrichloride.

All complexes were prepared as follows:

To a solution of (0.408 g, 1 m mole) (L_1) in (20 ml) ethanol, the solution of (0.227 g., 1 m mole) of antimony trichoride in (20 ml) of ethanol was added. The solution was warmed and stirred for one hour, the solution formed was filtered. The filterate was evaporated dryness to give the product. Recrystallization from (10 ml) methanol gave brown crystalline, decomposed at 130 D°, yield 90%. A similar method was followed to the preparation of the other complexes; L_2Sb, L_2 (0.332 g, 1 m mole), $SbCl_3$ (0.227 g, 1 m mole), (40 ml) ethanol (10 ml) methanol decomposed at 170 D°, yield 88%. L_3Sb, L_3 (0.272 g, lm mole), $SbCl_3$ (0.227.5 g, 1 m mole), (40 ml) ethanol, (10 ml) methanol decomposed at 190 D°, yield 90%. The physical properties of all complexes are listed in Table (1).

Results and discussion

Reaction of the ligands (L_1–L_3) with $VOSO_4$ or $SbCl_3$ were carried out in ethanol. All complexes are stable, soluble in water, DMF and DMSO, Table (1). The essential infrared data are summarized in Table (2). The observed broad bands at (3458-3414 cm^{-1}) and at (1755-1700 cm^{-1}) in the IR spectra of all complexes were assigned to υ(O–H) carboxylic group or water of hydrogen and υ(C=O) Lacton ring respectively[10-11]. Distinguishing between coordinate and uncoordinate carboxylic group in L_n, (where n = 1, 2, 3) complexes (VO^{2+}, Sb^{3+}) can be made readily from their infrared spectra and compared with the other infrared spectra of the free ligands, illustrates the infrared spectra of vanadyl and antimony complex, the uncoordinated COOH group absorbs at 1704 cm^{-1}, unlike the other peaks absorb at 1560 cm^{-1} (υ_{asymm}. COO^-) and 1473 cm^{-1} (υ_{symm}. COO^-)[12-13]. Accordingly, the antisymmetric and symmetric stretching vibration modes υ_{asymm} (COO^-) and υ_{symm}. (COO^-) groups may help in elucidating the structure of our complexes. The separation value (Δ) between υ_{asymm}. (COO^-) and υ_{symm}. (COO^-) in metal complexes was in the range 75-30 cm^{-1} and this suggests the coordination of carboxylate group in all complexes in a bidentate fashion[14]. The strongly coupled C=O and C=C stretching of all three ligands were observed at 1681-1635 cm^{-1} shifted toward lower frequencies may be due to complex formation Table (2). The vanadyl complexes showed strong bands at (952, 1100) cm^{-1} indicate a monomeric V–O unit and SO_4^{-2}[15]. Conclusive evidence of the bonding is also shown by the observation new band in the spectra of all metal complexes in the low frequency region at 505-447 cm^{-1} stretching vibration (M-O)[16-18].

Table (2): Infrared spectral data (wave number u^-) cm^{-1} for the ligand L and its complexes

Compound	υ(O–H) $\upsilon(OH_2)$	υ(C–H) Aliph	υ(C–I=O) lactone	Δcm^{-1}	$\upsilon_{a\,symm.}$ $\upsilon_{symm.}$ COO^-	Δ cm^{-1}	υ(M–O)	υ(V=O)	υSO_4^{-2}	Additional peaks
L_1	3421(br)OH-water 2700-2500(OH–COOH)	2954(w)	1755(s)							1608 C=O, C=C 1149-941 C–O,C–C
$[L_1VO.2H_2O]SO_4.6H_2O$	1704 OH-COOH 780 (OH_2)eq.	3020	1742	13	1560(w) 1485(s)	75	455	972	1100	1635 C=O, C=C 1066-806 C–O, C–C
$[L_1SbCl_3]7H_2O$	3360(br) OH-water 1704 (OH–COOH)	2938	1708	47	(1512)w (1485)s	27	447			1647 C=O, C=C 1060-808 C–O, C–C
L_2	3402(w) OH-water	2920	1755							1662 C=O, C=C 1100-900 C–O, C–C
$[L_2VO.2H_2O]SO_4.H_2O$	3417 (OH–COOH) 780 (OH_2)eq.	3016	1734	21	(1560)w (1485)w	75	505	952	1100	1631 C=O, C=C 1066-806 C–O, C–C
$[L_2SbCl_2H_2O]Cl$	3441(br) OH- water	2990	1716	39	(1510)m (1473)w	37	482			1627 C=O, C=C 1053-821 C–O, C–C
L_3	3402 OH-water 2596 (OH–COOH)	2993	1755							1662 C=O, C=C 1627,1327 carboxyl group asymm. & symm. (900-500) C–O, C–C
$[L_3VO.2H_2O]SO_4$	3414 OH-water 780 (OH_2)eq.	2835	1750	5	1515(w) 1485(s)	30	505	952	1100	1620 C=O, C=C 1068-875 C–O, C–C
$[L_3SbCl_2H_2O]Cl.4H_2O$	3456 OH-water	2992	1700	15	1515 1480	35	486			1631 C=O, C=C 1053-864 C–O, C–C

Recorder as KBr disk br = broad, s=strong, w=weak, m=medium, d=bending, aliph. Aliphatic

Electronic spectra

Table (3) gives the electronic spectral data of the prepared complexes. The vanadyl complexes showed one broad band in the visible region which attributed to square pyramidal symmetry. This band is due to transition; $^2E \leftarrow B_2$ and good analogous with those reported for oxovanadium (IV) complexes[19-20]. The UV-Vis absorption spectra of the Sb (III) complexes showed a new shoulder at (271-280) nm compared with three ligands indicating to charge transfer bands[16-18].

Table (3): Electronic spectra data of ligands and its metal complexes

compounds	λ nm	u⁻ wave number cm^{-1}	(e_{max} $molar^{-1}$ cm^{-1})	Assignments bands	proposed structure
L_1	246	40650	1175	$\pi \rightarrow \pi^*$	
$[L_1VO.2H_2O]SO_4.6H_2O$	296.5	33726	2452	$^2E \leftarrow B_2$	Square pyramidal
L_2	248.5	40241	952	$\pi \rightarrow \pi^*$	
$[L_2VO.2H_2O]SO_4.H_2O$	291	34364	2309	$E^2 \leftarrow B_2$	Square pyramidal
L_3	261	38314	1307	$\pi \rightarrow \pi^*$	
$[L_3VO.2H_2O]SO_4$	293	34129	2275	$^2E \leftarrow B_2$	Square pyramidal
$[L_1SbCl_3]7H_2O$	271	36900	1883	L.F.C.T.	Square pyramidal
$[L_2SbCl_2H_2O]Cl$	270	37037	1830	L.F.C.T.	Square pyramidal
$[L_3SbCl_2H_2O]Cl.4H_2O$	280	35714	1200	L.F.C.T.	Square pyramidal

L.F.C.T.= Ligand Field Charge Transfer

Solution chemistry

Molar conductivity of the complexes.

The molar conductance of the complexes in (water), Table (4) lie in the range of (110-161.5) S. cm^2 $molar^{-1}$, which is indicating to their electrolytic nature with (1:1) ratio, except for the complex $L_1SbCl_3.7H_2O$ which lies in the 25 S. cm^2 $molar^{-1}$ range, indicating a non-electrolyte nature[21].

Table (4): The molar conductance of the complexes

Compound fragment ions	L **M.S cm^2 $molar^{-1}$**	ratio
$[L_1VO.2H_2O]SO_4.6H_2O$	147	1:1
$[L_1SbCl_3]7H_2O$	25	Non electrolytic
$[L_2VO.2H_2O]SO_4.H_2O$	160.4	1:1
$[L_2SbCl_2H_2O]Cl$	110	1:1
$[L_3VO.2H_2O]SO_4$	161.5	1:1
$[L_3SbCl_2H_2O]Cl.4H_2O$	113.4	1:1

Mole- ratio method

In this method the ratio (L:M) was calculated by measurement the absorption of a series of solutions contain an increased, the based molar concentrations of one component (Ligand) with constant concentration of the other component (Metal) at wave length which occurs in is higher absorption from the product complex and does not occur the absorption to the chelate ligand alone or to the metal ion alone.

The relation ship between the absorption which was presented as (Y) axis and the concentration of the two reactants (Metal:ligand), which was presented as (X) axis was plotted, also the rectum contiguity was drown until they intersect and from the intersection point equivalent metal concentration was limitated as it was shown in the Tables, and Figures below[22].

Determination of the mole-ratio for the ligands complexes $[L_1VO.2H_2O]SO_4.6H_2O$, $[L_1SbCl_3]7H_2O$, $[L_2VO.2H_2O]SO_4.H_2O$,$[L_2SbCl_2.H_2O]Cl$, $[L_3VO.2H_2O]SO_4$ and $[L_3SbCl_2H_2O]Cl.4H_2O$.

The absorbance value against mole ratio values are sunmmerizd in Table (5). The mole ratio curves of these complexes in water are illustrated in Fig. (1).

Table (5): VM, VL and Absorption of ligand L_1, VM = volume of metal in ml, VL= volume of ligand in ml

$[L_1VO.2H_2O]SO_4.6H_2O$ λ(250)nm			$[L_1SbCl_3]7H_2O$ λ(250)nm		
VM	**VL**	**Abs**	**VM**	**VL**	**Abs**
1 ml	0.25	1.220	1 ml	0.25	1.513
1	0.50	1.610	1	0.50	1.800
1	0.75	1.870	1	0.75	2.200
1	1	2.000	1	1	2.440
1	1.25	2.100	1	1.25	2.440
1	1.50	2.170	1	1.50	2.380
1	1.75	2.280	1	1.75	2.440
1	2.0	2.290	1	2	2.450
1	2.25	2.300	1	2.25	2.440
1	2.50	2.300	1	2.50	2.500
1	2.75	2.350	1	2.75	2.440
1	3.0	2.400	1	3	2.520

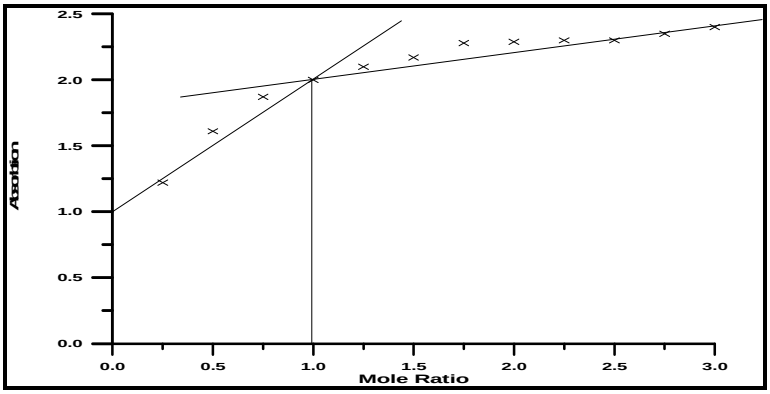


Fig. (1): The mole ratio curve of complex $[L_1VO.2H_2O]SO_4.6H_2O$ in solution (1×10^{-3} mole. l^{-1}) at (l=250 nm)

Determination of the stability constant and ΔG for the complexes.

The stability constants were determined according to[23]:

M + L ↔ ML ……(1)

M = metal ion

L = The ligand

K = ML / [M] [L] …….(2)

K=stability constant (or formation constant).

[ ] = concentration

α = (Am – As)/ Am ……. (3)

α = decomposition degree

As = The absorption at the equivalent point of mole ratio.

Am=The maximum absorption to mole ratio

The equation (2) is written to mole ratio (1:1) as following.

$K = (1 - \alpha)\ \alpha^2 C$ …….(4)

C = The complex concentration. (mole. L^{-1}).

The ΔG is Gibbes free energy.

It is a function determine the direction (way) of the chemical reaction thermodynamically, since the chemical reaction is directed toward the side which ΔG is less than zero: (ΔG < 0).

The ΔG were determined according to the following equation:

ΔG = -2.303 RT Log K., Table (6).

Table (6): Stability constant and ΔG for the ligand (L_1) complexes [$L_1VO.2H_2O]SO_4.6H_2O$ and $[L_1SbCl_3]7H_2O$.

Compounds	As	Am	α	K	Log K	1/K	ΔG
$[L_1VO.2H_2O]SO_4.6H_2O$	2.000	2.290	0.126	$5.5x10^4$	4.7	0.21	-27
$[L_1SbCl_3]7H_2O$	2.440	2.450	0.004	$6.2x10^6$	6.8	0.15	-39

$[L_1SbCl_3]7H_2O > [L_1VO.2H_2O]SO_4.6H_2O$

Evaluation of bacterial activity for the ligands and some of its complexes

The biological activity of the ligands L_1, L_2, L_3 and some of its complexes were studied by using inhibition method[24] for two types of pathogenic bacteria. One type of bacteria was gram positive which is *staphylococcus aureu*. The second one was gram negative which is *escherichia coli*.

The inhibition effect of the three ligands and its complexes were evaluated for the two types of bacteria as shown in Table (7) and Figs. (2–3).

Table (7): showed the inhibition zone in millimeter for the bacteria after 24hr. in cubation at 37°C

Compounds	***Staphylococcus aureu* (gram positive)**	***Escherichia coli* (gram nagative)**
L_1	17 mm	18 mm
$[L_1VO.2H_2O]SO_4.6H_2O$	20 mm	17 mm
$[L_1SbCl_3]7H_2O$	32 mm	35 mm
L_2	27 mm	25 mm
$[L_2VO.2H_2O]SO_4.H_2O$	13 mm	17 mm
$[L_2SbCl_2H_2O]Cl$	35 mm	30 mm
L_3	15 mm	17 mm
$[L_3VO.2H_2O]SO_4$	33 mm	30 mm
$[L_3SbCl_2H_2O]Cl.4H_2O$	37 mm	35 mm

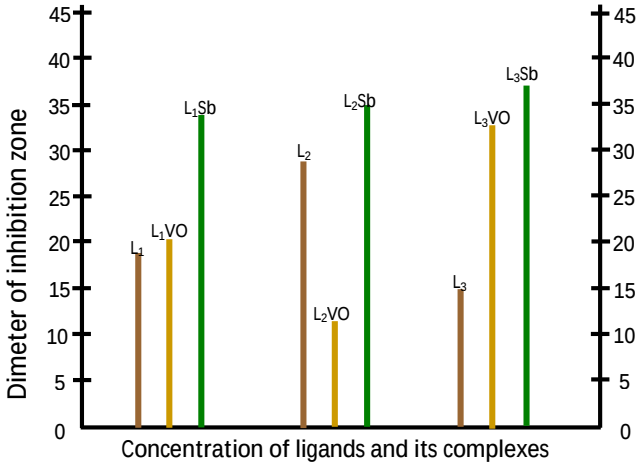


Fig. (2): Effect of *staphylococcus aureu* gram positive

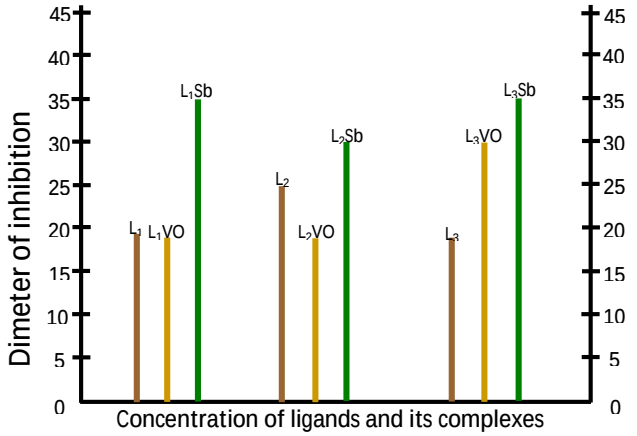


Fig. (3): Effect of *Escherichia coli* gram negative

The rate of inhibition diameter was varied according to the variation of type of ligands and their some of complexes.The Fig. (4) showed the values of inhibition diameter for the L_1, L_2, L_3 and complexes $[L_1VO.2H_2O]SO_4.6H_2O$,$[L_1SbCl_3]7H_2O$, $[L_2VO.2H_2O]SO_4.H_2O$,$[L_2SbCl_2.H_2O]Cl$, $[L_3VO.2H_2O]SO_4$ and $[L_3SbCl_2H_2O]Cl.4H_2O$ for the two types of bacteria respectively.

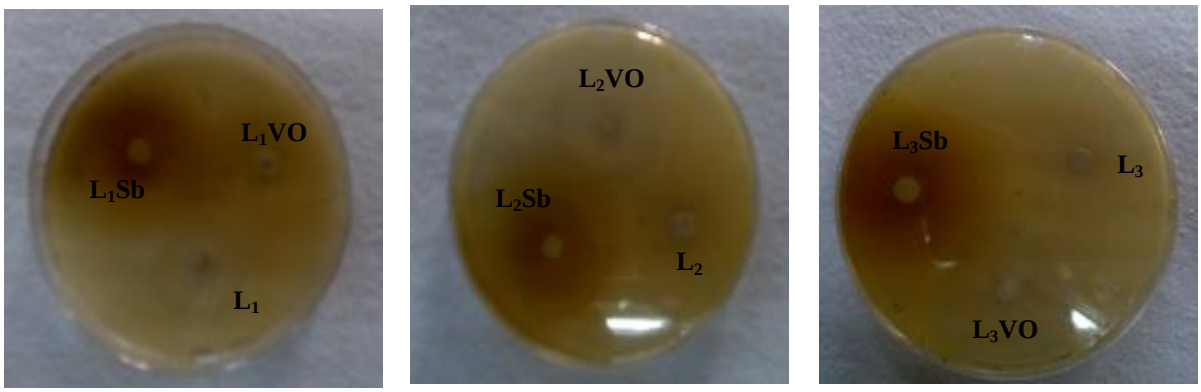
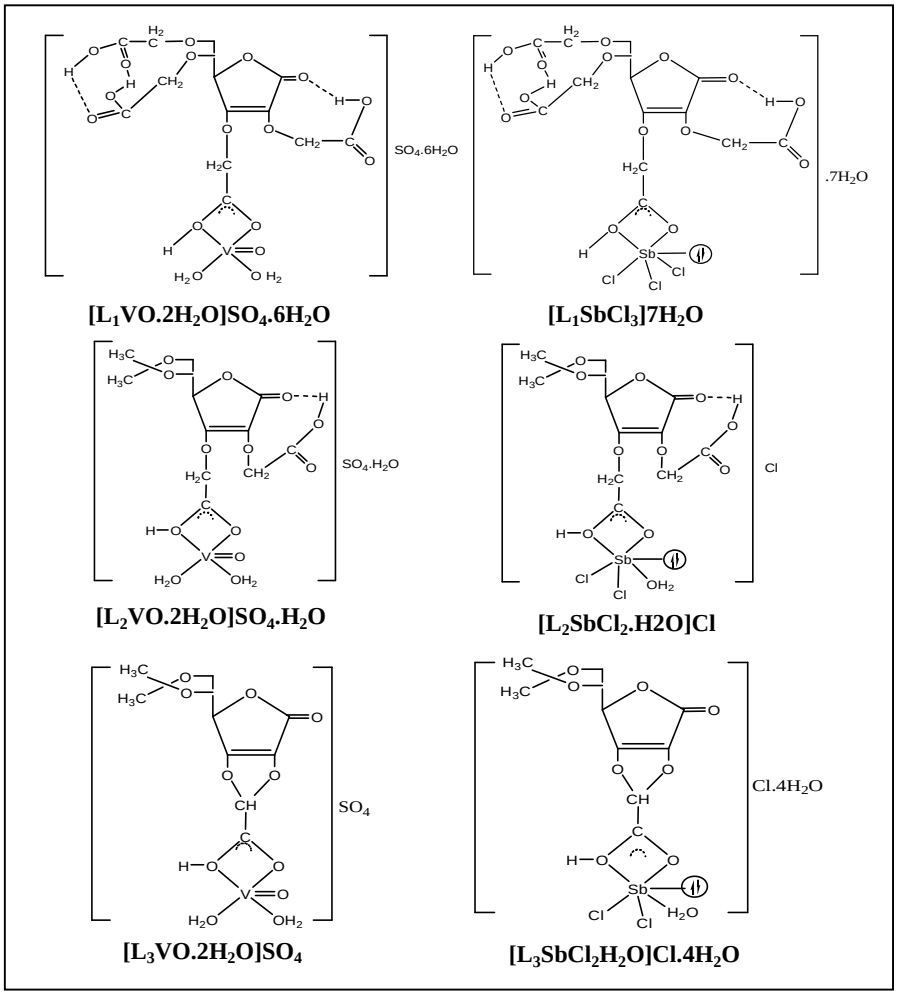


Fig. (4): Inhibition diameter for ligands and complexes E = *Escherichia coli*

Conclusion

A six complexes of VO(II) and Sb(III) with three ligands derived from L-ascorbic acid have been prepared and characterized.

The ligand L_1- L_3 bind to metal ion through C-3-CH_2COOH, Vanadyl and antimony complexes posses square pyramide as bellow:



$[L_1VO.2H_2O]SO_4.6H_2O$ **$[L_1SbCl_3]7H_2O$**

$[L_2VO.2H_2O]SO_4.H_2O$ **$[L_2SbCl_2.H2O]Cl$**

$[L_3VO.2H_2O]SO_4$ **$[L_3SbCl_2H_2O]Cl.4H_2O$**

The complexes were screened for their antibacterial activity against *staphylococcus aureu* and *Escherichia coli* showed various activities.

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تحضير، تشخيص وتقييم الفعالية البكتيرية لمعقدات جديدة للفناديل الثنائي والانتيمون الثلاثي لمشتقات فيتامين–C–

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الخلاصة:

تفاعل كبريتات الفناديل الثنائي، ثلاثي كلوريد الانتيمون مع 2، 3، 5، 6–*O*،*O*،*O*،*O*– رباعي حامض الخليك–L– حامض اسكوربك (L_1)، 5، 6–*O*–ايزوبروبليدين–2، 3–*O*،*O*– حامض الخليك–L–حامض اسكوربك (L_2) ومثيليدين– كاربوكسيل– 5، 6–*O*– ايزوبروبليدين–L– حامض اسكوربك (L_3) بالتعاقب في الكحول الاثيلي كمذيب. المعقدات المتكونة شخصت بواسطة تقنية تحليل العناصر، الدراسات الطيفية والنسبة المولية. الليكاندات (L_1)، (L_2) ثنائية السن والارتباط عن طريق $C–3–CH_2COOH$، الليكاند الثالث (L_3) الارتباط عن طريق مجموعة الكربوكيسلات. معقدات الفناديل والانتيمون معطية شكل هرم مربع مستوي. أعطت المعقدات فعالية مختلفة تجاه نوعي البكتريا *staphylococcus aureu* و *Escherichia coli*.

الكلمات المفتاحية: تحضير، تقييم الفعالية البكتيرية، فناديل، الانتيمون ومشتقات فيتامين–C–.