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Research Article

SYNTHESIS, CHARACTERISATION AND BIOLOGICAL ACTIVITY OF VANADIUM (V) WITH 2,4-DIHYDROXY CINNAMALDEHYDE THIOSEMICARBAZONE (2,4-DCTSC)

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ABSTRACT

The 2,4-Dihydroxy cinnamaldehyde thiosemicarbazide has been synthesized. Complex of 2,4-dihydroxy cinnamaldehyde thiosemicarbazide with transition metals, Vanadium (v) has been synthesized in methanol medium. The formation of the complex are endothermic processes. The ligand was characterized by melting point, elemental analysis, absorption spectra and antimicrobial activity while the complex was characterized by melting point, absorption spectra, A simple and sensitive spectrophotometric method was developed for transition metal complex of 2,4-dihydroxy cinnamaldehyde thiosemicarbazide. The optimum condition for complete colour development have been established. The stability constant, dissociation constant & change in free energy of vanadium (v) has been determined by Job's variation & mole ratio method indicate that the M:L is 2:1. Tolerance limit of diverse ions in the determination of vanadium (v) with is investigated. Antimicrbial activity and antifungal activity have been studied.

Key words: 2,4-Dihydroxy cinnamaldehyde, thiosemicarbazide, vanadium (v), spectrophotometry, antimicrobial activity.

INTRODUCTION

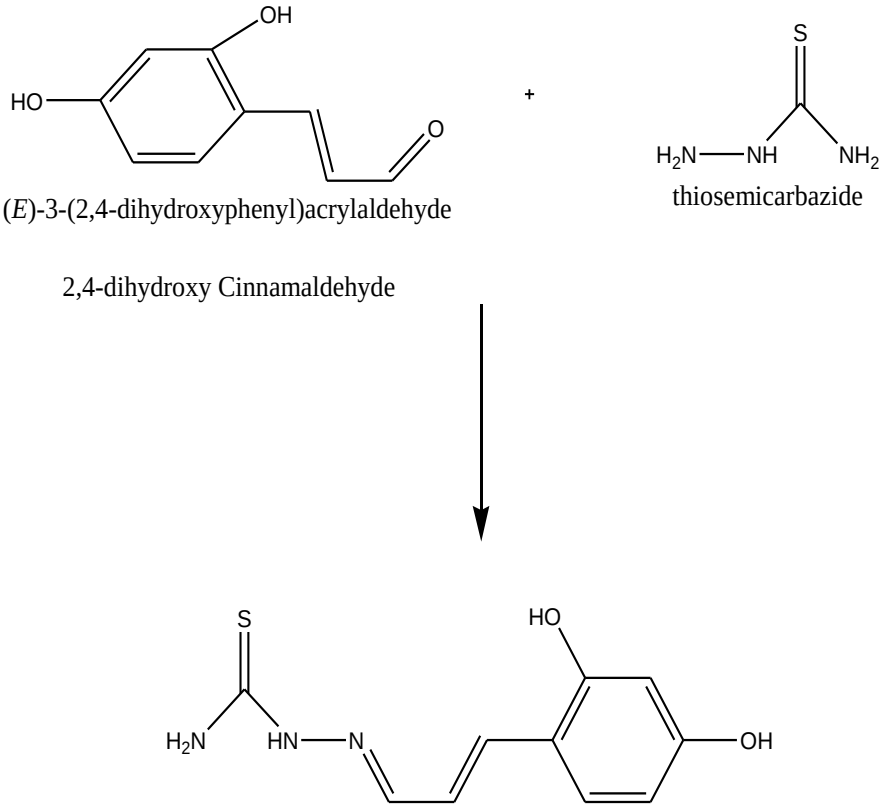
Thiosemicarbazones are the compounds which forms complex with metal ions. They act as a complexing agent. The complex of thiosemicarbazones shows a number of medicinal properties. They also have antitubercular activity[1]. Its complex is active against viruses, influenza, protozoa, small pox[2]. They are also used in pesticides and fungicides[3]. A number of thiosemicarbazides and derivatives have good antibacterial[4], antifungal[5], herbicidal[6] and antiacetylcholinesterase activities[7]. The thiosemicarbazide and thiosemicarbazones have special attraction due to their activity against certain kinds of tumours[8]. It is known that some drugs have increased the activity due to the metal complexes[9]. The number of metal chelates inhibits the growth of tumours[10]. In cancer treatments, it has been shown that the active species is not the thiosemicarbazone itself but a metal chelates of the thiosemicarbzones[11]. Semicarbazone and thiosemicarbazone complexes of transition and non-transition metals are known for their diverse biological activities[12]. Dimethoxy benzaldehyde thiosemicarbazones and their related compounds also has biological activities[13]. Bis thiosemicarbazones and their related compounds also has been studied for their biological activities[14]. Thiosemicarbazone complex with transition metal containing sulphur and nitrogen as donar atoms have attracted special attention[15]. Thiosemicarbazide and thiosemicarbazone used as a potential ligands[16]. Condensation of aldehydes or ketones with thiosemicarbazons forms heterocyclic thiosemicarbazones. They are shows the antibacterial, antiviral and also antitumor activities[17]. The antitumour activity of the heterocyclic thiosemicarbazones is due to the compounds inhibiting DNA synthesis[18,19].

Vanadium is a hard-grey metal. The abundance of the vanadium is 150 parts per million in the igneous rocks of the earth's crust. Vanadium is an important catalyst in oxidation reaction such as naphthalene phthalic acid and toluene benzaldehyde. V_2O_5 is important catalyst in the manufacture of SO_3 by the catalyst process. Vanadium is important trace element. It is beneficial and essential for humans[20]. It is essential for some living organisms[21-30]. The chemistry of the coordination compound of vanadium is typical due to their involvement in the processes of biological importance. Vanadium ions can play a role in biology as counter ions for protein, DNA, RNA, and various biological structures. Vanadium ions have many structural roles reflected by its structural and electronic analogy to phosphorus[31-39]. Vanadium is also an enzyme co-factor[25,27,38-48]. Vanadium can act and function in the biosphere include investigation in to the fundamental co-ordination and redox chemistry of the element[49].

MATERIALS AND METHODS

All chemical and solvents used were of analytical grade. An Elico pH meter LI-610 is used for the pH measurements. An Elico UV-visible spectrophotometer model UV-SL-164 equipped with 1 cm quartz cell used for spectrophotometeric measurements taken on the instrument. Elemental analysis and antimicrobial activity was done in Laboratory approved by Central Government for AGMARK.

Synthesis and characterization of 2,4-dihydroxy cinnamaldehyde thiosemicarbazide
Synthesis of 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazide



(*E*)-3-(2,4-dihydroxyphenyl)acrylaldehyde

thiosemicarbazide

2,4-dihydroxy Cinnamaldehyde

(1*E*)-1-((*E*)-3-(2,4-dihydroxyphenyl)allylidene)thiosemicarbazide

2,4-dihydroxy cinnamaldehyde thiosemicarbazide

The crude product is crystallized in methanol. The recrystallized product has melting point is 186 ^{0}C and molecular weight by formula is 237.00

Characterisation of isonicotinoyl hydrazone (PIH)
Absorption Spectra of 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC)

Absorption Spectra of 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC) was recorded against a blank solution containing buffer (pH 5), Absorption spectra was recorded in the wave length range 260 nm to 560 nm. It shows an absorption maximum at 395 nm wavelength the molar absorptivity of is 0.9843521 x 10^3 L.mol^{-1}.cm^{-1}. Figure 1.

Elemental Analysis of 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC)

The elemental analysis of PIH was done in Laboratory approved by Central Government for AGMARK. It shows the result of elemental analysis in **Table 1**.

Effect of Reagent concentration

Effect of Reagent concentration was studied by taking varying amount of reagent and fixed amount of transition metal Figure 2.

Effect of Reagent PH

Effect of PH was studied by taking varying amount of reagent and fixed amount of transition metal Figure 3.

Validity of Beer's Law and Composition of Complex

For the study of Beer's law the solutions were prepared which containing different amounts of Vanadium (v), same amount of ligand pH 3. The composition of the V (v)-metal complex is found to be 1:2. It was determined by studying Job's method. The ratio of metal ion to ligand molecule in the coloured complex was found to be 1:2 composition of complex. Figure 4 & 5

Antimicrobial Activity of 2,4-Dihydroxy Cinnamaldehyde Thiosemicarbazone (2,4-DCTSC)
Antimicrobial Activity of 2,4-Dihydroxy Cinnamaldehyde Thiosemicarbazone (2,4-DCTSC) was done in Laboratory approved by Central Government for AGMARK. It shows the result of elemental analysis in Table 2.

Physico-chemical Characteristic 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC)
Physico-chemical and Analytical characteristic of transition metal complex of ligand was studied and given in Table 3 and Tolerance limit of diverse ions in the determination of ligand shown in Table 4.

RESULTS AND DISCUSSION

Table 1: Elemental analysis of 2,4-DCTSC

Sr.No.	Chemical Analysis	Percentage Found	Percentage Expected
1)	Carbon	50.63%	52.55 %
2)	Hydrogen	04.64 %	05.12 %
3)	Oxygen	13.50 %	14.02 %
4)	Nitrogen	17.72 %	16.39 %
5)	Sulphur	13.50 %	12.80 %

Table 2: Antimicrobial activity of 2,4-DCTSC

Sr.No.	Antimicrobial	Activity
1)	*Klebsiella pneumoniae*	Nil
2)	*Vibriae cholerease*	Nil
3)	*Bacillus megaterium*	Nil
4)	*Salmonalla typhi*	Nil
5)	*Shigella flexneri*	Nil

Table 3: Experimental results and physical data of 2,4-DCTSC and V(v) complex

Code No	Compound M.P. (^{0}C)	Colour	Molecular weight by formula gm/mole	Yield
2,4-DCTSC	186^{0}C	Greenish blue	237.00	87 %
V(V)-2,4-DCTSC	175^{0}C	Yellow blue	332.94	81%

Table 4: Physico-chemical and analytical characterisation of V(v) complex of 2,4-DCTSC

Sr.No.	Characteristics	Result
		V (V)-ligand
1)	Absorption Spectra	395 nm
2)	Molar absorptivity	0.9843521 x 10^3 Lit. mol^{-1}. cm^{-1}
3)	pH range (optimum)	5.0
4)	Reagent required for maximum complexation	0.557 ml
5)	pKa	5.134 x 10^8
6)	Beer's law validity range (ppm)	1.0 ppm
7)	Composition of complex (M : L)	1:2
8)	Stability Constant	4.4128031 x 10^7
9)	Dissociation Constant	3.992216 x 10^{-8}
10)	Change in free energy	-29.42 KJ/mole
11)	Sandell's Sensitivity µg /cm^2)	0.001994 µg /cm^2

Table 5: Tolerance limit of diverse ions of V(v) complex of 2,4-DHCTSC

Sr. No.	Metal ion	Salt	Interference
1)	Mg (II)	$MgSO_4$	89
2)	Ca (II)	$CaCl_2.2H_2O$	31
3)	Cd (II)	$CdCl_2$	53
4)	Mn (II)	$MnCl_2$	Interferes
5)	Co (II)	$CoSO_4$	62
6)	Ce (IV)	Ce $(SO_4)_2$	62
7)	Ba (II)	$BaCl_2$	55
8)	Cr (III)	$K_2Cr_2O_7$	32
9)	Hg (II)	$HgCl_2$	74
10)	Ti (V)	K-titanyl oxalate	92
11)	Ni (II)	$NiCl_2$	34
12)	Sn (II)	$SnCl_2$	27
13)	Na (I)	NaCl	67
14)	Pb (II)	$PbSO_4$	Interferes
15)	Zn (II)	$ZnSO_4$	25
16)	Al (III)	$AlCl_3$	16
17)	Pd (II)	$PdCl_2$	Interferes
18)	K (II)	KCl	84

2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC) has greenish blue colour with yield 87 % and vanadium (V)- 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC) yellowish blue in colour with 81 % yield. Absorption spectra of vanadium (V)- 2,4-Dihydroxy cinnamaldehyde thiosemicarbazone at 395 nm with molar absorptivity 0.9843521 x 10^3 Lit. mol^{-1}. cm^{-1} and optimum pH range is 5.0. Reagent required for maximum complexation is 0.557 ml. Stability constant, dissociation constant and change in free energy is 4.4128031 x $10^{7,}$ 3.992216 x 10^{-8} and -29.42 KJ/mole.

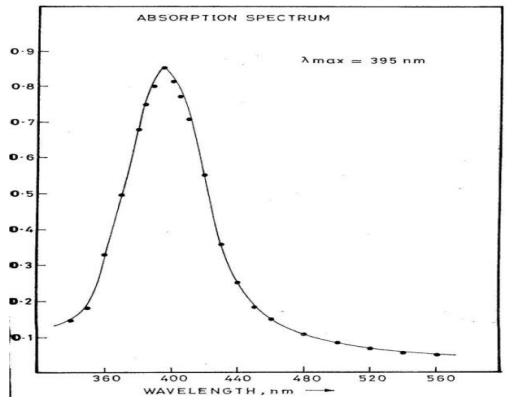


Figure 1: Absorption Spectra of 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC)
Absorbance Vs Wavelength

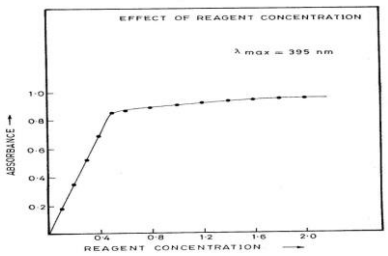


Figure 2: Effect of reagent concentration of V(v)-2,4-Dihydroxy cinnamaldehyde thiosemicarbazide (2,4-DCTSC)

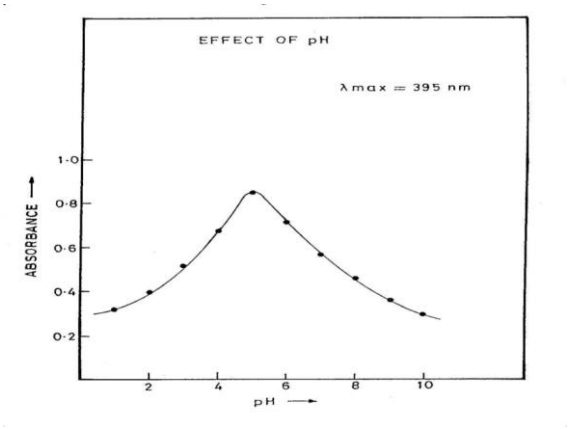


Figure 3: Effect of PH of V(v)-2,4-Dihydroxy cinnamaldehyde thiosemicarbazide (2,4-DCTSC)

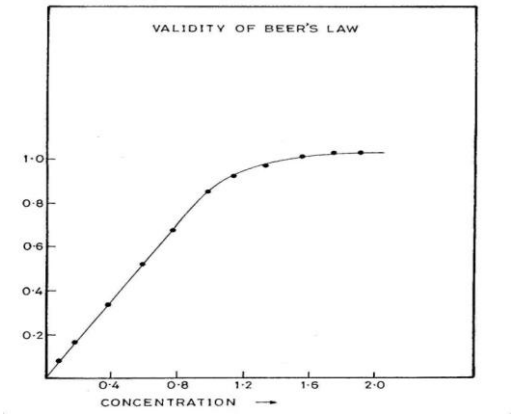


Figure 4: Validity of beer's law of V(v)-2,4-Dihydroxy cinnamaldehyde thiosemicarbazide (2,4-DCTSC)

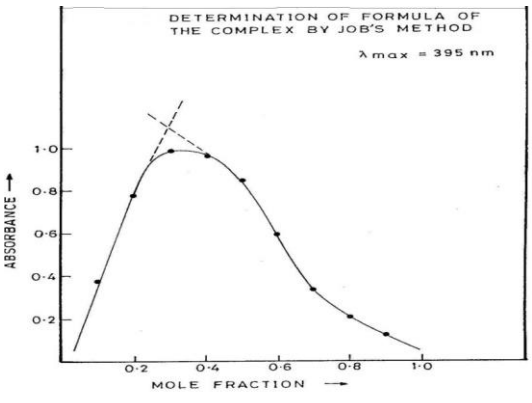
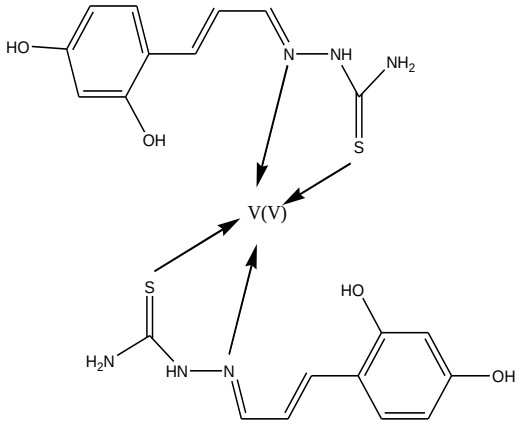


Figure 5: Determination of formula of V(v) complex of 2,4-dihydroxy cinnamaldehyde thiosemicarbazide (2,4-DCTSC)



Structure of Vanadium (V)- 2,4-dihydroxy cinnamaldehyde thiosemicarbazone (2,4-DCTSC)

CONCLUSION

Vanadium (V)- 2,4-Dihydroxy cinnamaldehyde Thiosemicarbazone (2,4-DCTSC) complex is 1:2. Sandell's Sensitivity is 0.001994 µg /cm ^{2}

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