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Original Synthesis and Study of Substituted 1,3-Thiazine and its Nanoparticles on Phytotic Growth of Some Flowering Plants

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Abstract: *The synthesis, spectral analysis and biological activities of 4-phenyl-2-hydroxy-chlorosubstituted-2-imino-1,3 thiazines have been carried out. In this case 4-(2'-hydroxy-3',5'-dichlorophenyl)-6-(hexyl)-2-imino-3,6-dihydro-1,3- thiazine (G) has been screened. The compound G was synthesized from 2'-hydroxy-3',5'-dichlorophenyl-4-(hexyl)chalcone (a") by the action of thiourea.The compound (a") was synthesized from 2'-hydroxy-3',5'-dichloroacetophenone by the action of heptanaldehyde in ethanol and 40% NaOH. The nanoparticles of the compounds G has been prepared by using ultrasonic technique. The titled compound and its nanoparticles were screened for their growth promoting activity on some flowering plants viz. Crysanthemum coronarium, Dahalia pinnata, Verbena officinalis, Iberis amara.*

Keywords: Chalcone, thiazine, thiourea, growth promoting activities

I. INTRODUCTION

Thiazine is a six membered ring system, which contains two hetero atom [N and S] placed in the heterocyclic ring at 1, 3 positions. Many workers have synthesized different 1,3-thiazines1-3. Thiazines are very useful units in the field of medicinal and pharmaceutical chemistry and have been reported to exhibit a variety of biological activities such as antiserotinin4, antipsychotic5, anti-HIV6 ,antimicrobial7 , antiviral8 , blood platelet aggregation inhibitors9 and Ca++ antagonist10. Moreover thiazine nucleus is a pharmacophore of cephalosporin that occupy a very important
place in the antibiotics and antifungal activity of thiazine nucleus is due to the presence of thiourea linkage in the structure. Chalcones and their analogues having α,β-unsaturated carbonyl system are very versatile substrates for the evolution of various reactions and physiologically active compounds. The reaction of thiourea with α, β-unsaturated ketones results in 1,3-thiazine. It has been well focused that the presence of 4-phenyl-chlorosubstituted moieties as well as 2-substituted amino group present in thiazine ring is an important structural feature and the resulting molecule would exhibit promising biological activities11-13. In the present study, 4 -phenyl-2-substituted-imino-thiazine has been synthesized from chalcones by using thiourea. The synthesized compound was evaluated for its growth promoting activity on some flowering plants viz. Crysanthemum coronarium, Dahalia pinnata, Verbena officinalis, Iberis amara.

EXPERIMENTAL :-

All the glasswares used in the present work were of pyrex quality. Melting points were determined in hot paraffin bath and are uncorrected. The purity of compounds was monitored on silica gel coated TLC plate. IR spectra were recorded on Perkin-Elmer spectrophotometer in KBr pelletes, H1 NMR spectra on spectrophotometer in CDCl3 with TMS as internal standard. UV spectra were recorded in nujol medium. The analytical data of the titled compounds was highly satisfactory. All the chemicals used were of analytical grade. All the solvents used were purified by standard methods. Physical characterisation data of all the compounds is given in Table 1.

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2'-Hydroxy 3',5'-dichloroacetophenone :

2-Hydroxy-5-chloroacetophenone was dissolved in acetic acid (5 ml), Sodium acetate (3g) was added to the reaction mixture and then chlorine in acetic acid reagent (40 ml; 7.5 w/v) was added dropwise with stirring. The temperature of the reaction mixture was maintained below 200C. The mixture was allowed to stand for 30 minutes. It was poured into water with stirring. A pale yellow solid then obtained was filtered, dried and crystallized from ethanol to get the compound 2'-hydroxy 3',5'-dichloroacetophenone.

Preparation of 2'-hydroxy-3',5'-dichlorophenyl-4-hexylchalcone (a") :

2'-Hydroxy-3',5'-dichloroacetophenone (0.1 mol) was dissolved in ethanol (50 ml) and heptnaldehyde (0.1 mol) was added gradually to the above solution and the mixture was heated to boiling temperature. Aqueous sodium hydroxide solution [40%; 40 ml] was added dropwise with constant stirring. The mixture was stirred mechanically at room temperature for about half an hour and kept overnight. It was then acidified by hydrochloric acid solution (10%). The solid product separated, was filtered, and washed with sodium bicarbonate (10%) followed by water. The crude product was crystallized from ethanol acetic acid mixture (a").

Preparation of 4-(2'-hydroxy-3',5'-dichlorophenyl)-6-(hexyl)-2-imino-3,6-dihydro-1,3-thiazine (G) :

2'-Hydroxy-3',5'-dichlorophenyl-4-hexylchalcone (a")(0.01 mol) and thiourea (0.02 mol) were dissolved in ethanol (25 ml). To this aquous KOH solution (0.02 mol) was added. The reaction mixture was refluxed for three hours, cooled and diluted with water then acidified with 1:1 HCl. The product thus obtained was crystallized from ethanol to get the compound (G).

The newly synthesized compound was characterised on the basis of elemental analysis, molecular determination, UV, IR, NMR. spectral data.

The UV, IR, and NMR spectral data :-

Compound (G) :

UV : Spectrum No. 1

The UV-Vis spectrum of the compound (G) reported in dioxane showed □maxvalue397 nm corresponding to n□□□ transition.

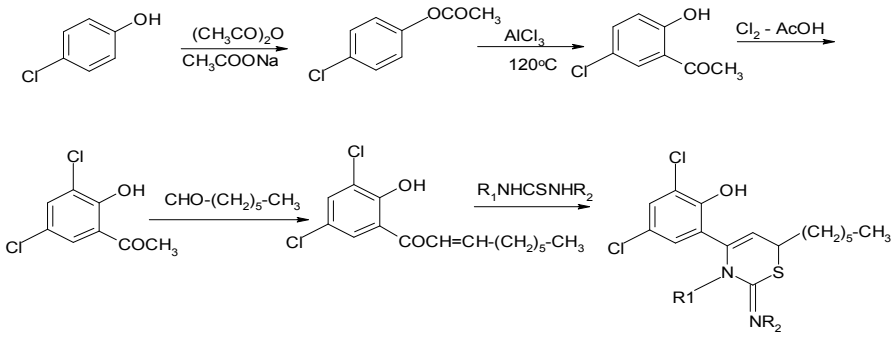
IR (KBr) :- Spectrum No. 2

3208.63 cm-1(-OH phenolic), 2928.36 cm-1 (aliphatic -C-H stretching), 2955.42cm-1(aromatic -C-H stretching), 3082.63 cm-1 (-N-H stretching), 1650.49 cm-1 (-C=N- stretching), 1023.73 cm-1 [(C-N) (C-NO2) stretching], 740.62 cm-1 (-C-Cl stretching in aliphatic), 1177.7 cm-1 (C-Cl) stretching in aromatic).

PMR :- Spectrum No. 3

∂ 1.25 (envelope of -CH2, 10H, -(CH2)5-CH3) ; ∂ 1.8 (d, 1H, -C=C-C-H) ; ∂ 3.5 (hump, 1H, =N-H) ; ∂ 6.7 (s,1H, =N-H),∂ 6.8 (d, 1H, =C-H) ; ∂ 7.0 to 7.8 (m, 2H, Ar-H) ; ∂ 12.36 (s, 1H, O-H)

Scheme:



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Where :
1) R1 = -H
2) R2 = -H

Growth Promoting Effect on some Flowering Plants :-

The experimental set up of the study was devided into two parts:
(i) Seed treatment (ii) Field experiment.

(i) Seed treatment :-

With a view to safeguard dormant seed's potential from harmful external agencies, the seeds of the test plants were treated by test compounds before sowing.

(ii) Field experiment :-

Pregerminated quality seeds of Crysanthemum coronarium, Dahalia pinnata, Verbena officinalis, Iberis amara. were procured from Department of Horticulture, Dr. PDKV, Akola.

The beds of cotton soil, 2.5 x 2.5 m size were prepared in an open field. The sowing of seeds of all four test flowering plants were done in separate beds and irrigated periodically.

The plants from each bed were devided into two groups i.e. A and B and designated as "Control" and "Treated" group plants respectively.

The plants from group B were sprayed with the solution of test compounds at weekly intervals. The field experiments were conducted to compare the treated plants of group B with untreated plants of controlled group A. In this context, the observations were recorded on 7, 14, 21, 28, 35, 42, 45, 56, 63, 70, 77, 84, 91 days after sowing corresponding to early vegetative, late vegetative, flowering, pod filing and pod maturation, with special reference to number of leaves and height of shoots.

The results of field's experiments are tabulated in the tables 2, 3 and 4.

II. RESULT AND DISCUSSION

The titled compound and its nanoparticles were screened for their growth promoting activity on test flowering plants viz, Crysanthemum coronarium, Dahalia pinnata, Verbena officinalis, Iberis amara.

When a comparison of morphological characters was made between those of treated and control group plants, it was interesting to note that all the treated plants exhibited significant shoot growth and considerable increase in the number of leaves as compared to those of untreated ones.

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Table 1 : Characterisation data of newly synthesized compounds :

Compounds	Molecular formula	M.P. in 0C	% of yield	% of element			
				C	H	N	S
	C8H6O2Cl2	54	80	47.90/48	2.95/3		
a”	C15H18O2Cl2	103	70	59.80/59.25	53.10/60.26		
G	C16H19ON2Cl2S	178	70	58.63/58.12	5.30/5.12	7.82/7.90	8.93/8.70

Activity of the test compound G:

Table No. 2

4-(2’-Hydroxy-3’,5’-dichlorophenyl)-6-(hexyl)-2-imino-3,6- dihydro-1,3-thiazine (G)

Periodicity of Observations [in days]	*Crysanthemum coronarium*				*Dahalia pinnata*				*Verbena officinalis*				*Iberis amara*			
	Shoot height		No. of leaves		Shoot height		No. of leaves		Shoot height		No. of leaves		Shoot height		No. of leaves	
	C	T	C	T	C	T	C	T	C	T	C	T	C	T	C	T
7	1.0	1.0	1	1	2.5	1.5	2	2	4.4	4	2	2	2	2	2	3
14	1.2	1.2	2	2	7.5	7	2	2	10	8	2	2	2.1	2.5	2	3
21	1.3	1.4	7	10	8	12	2	4	15	11	3	5	2.3	2.8	3	4
28	1.5	1.6	9	11	9	19	3	6	16	18	4	6	2.5	2.7	4	5
35	1.6	1.8	10	12	11	26	4	7	18	19	5	9	2.8	3.0	5	7
42	1.8	2.0	12	15	17	42	5	8	19	20	7	12	3.0	3.4	6	8
49	2.3	3.5	14	18	25	48	6	8	20	21	8	14	3.5	3.9	8	9
56	3.6	4.0	16	22	28	52	7	9	23	25	10	17	3.8	4.5	10	12
63	5.5	6.7	18	24	31	55	8	10	25	30	12	18	4.2	5.0	12	14
70	7	12	20	28	34	60	9	11	27	32	14	20	4.6	5.4	14	16
77	14	19	22	30	36	63	10	13	28	35	16	23	5.5	7.0	16	18
84	20	24	24	32	38	65	11	15	29	36	18	25	7,2	14	20	24
91	24	30	26	36	40	68	12	17	36	38	20	27	8.2	17	25	29

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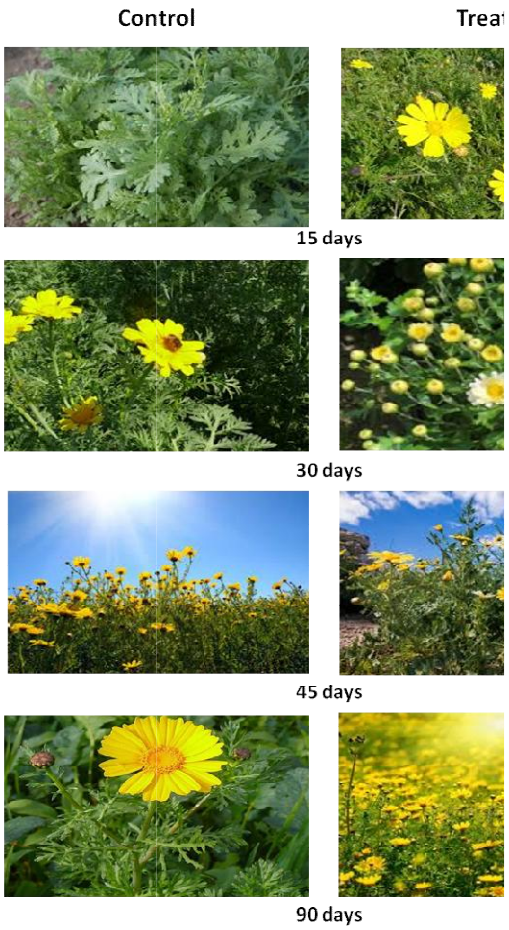
Impact of the 4-(2'-Hydroxy-3',5'-dichlorophen
imino-3,6- dihydro-1,3-thiazine (G) on phyto
Crysanthemum coronarium



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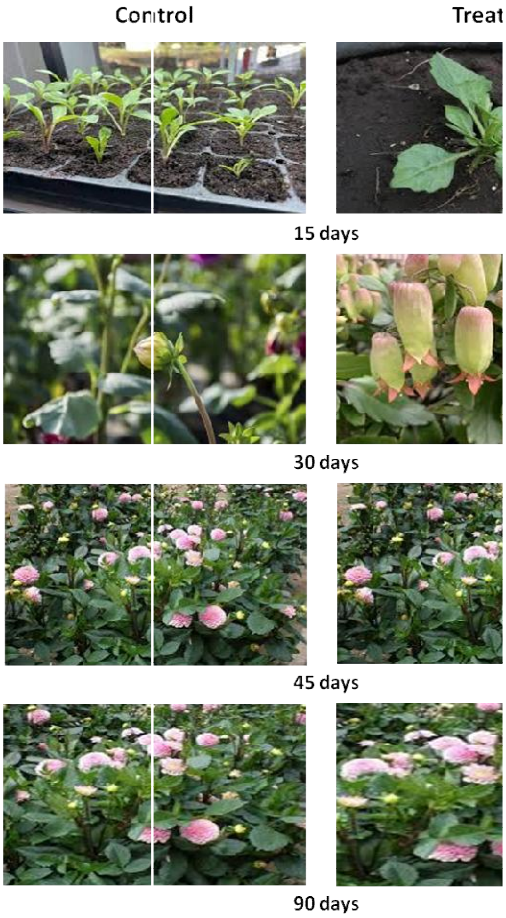
Impact of the compound 4-(2'-Hydroxy-3',5'-dic 6-(hexyl)-2-imino-3,6- dihydro-1,3-thiazine (G) growth of *Dahalia pinnata*



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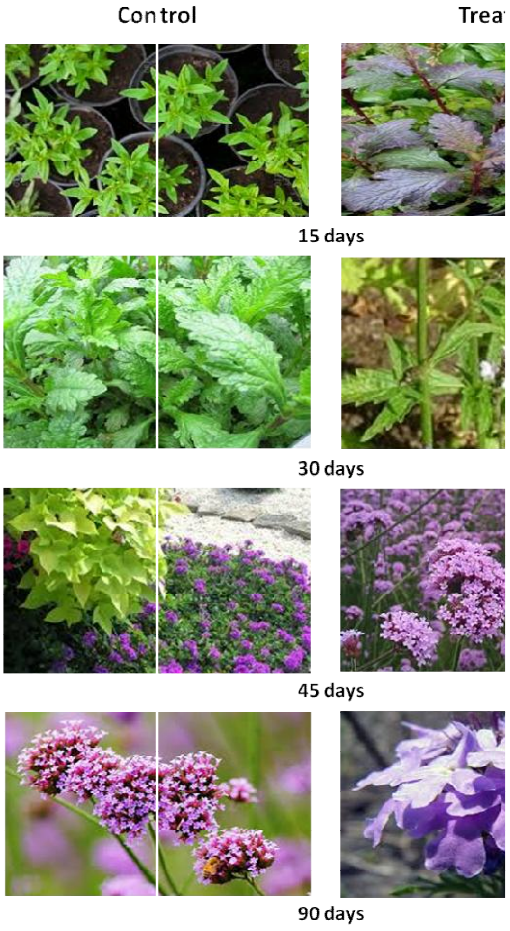
Impact of the compound 4-(2'-Hydroxy-3',5'-di
6-(hexyl)-2-imino-3,6- dihydro-1,3-thiazine (G
growth of *Verbena officinalis*



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Impact of the 4-(2'-Hydroxy-3',5'-dichlorophen
2-imino-3,6- dihydro-1,3-thiazine (G) on phytot
Iberis amara

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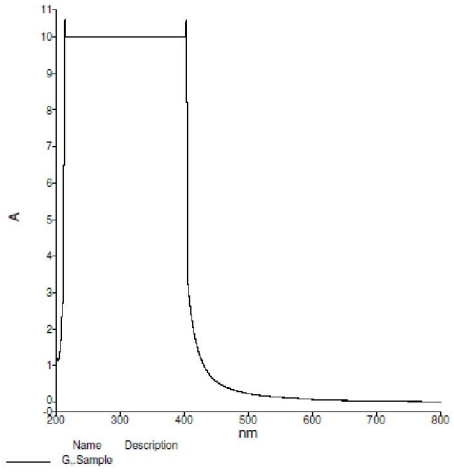
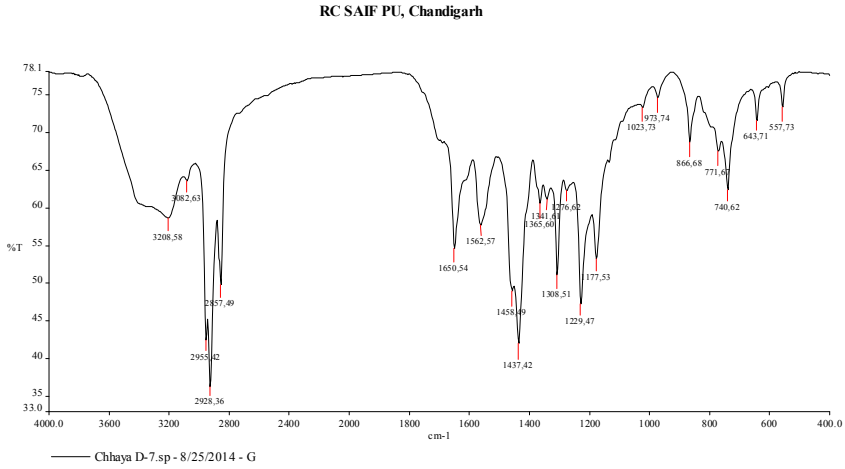


Spectrum No. 1



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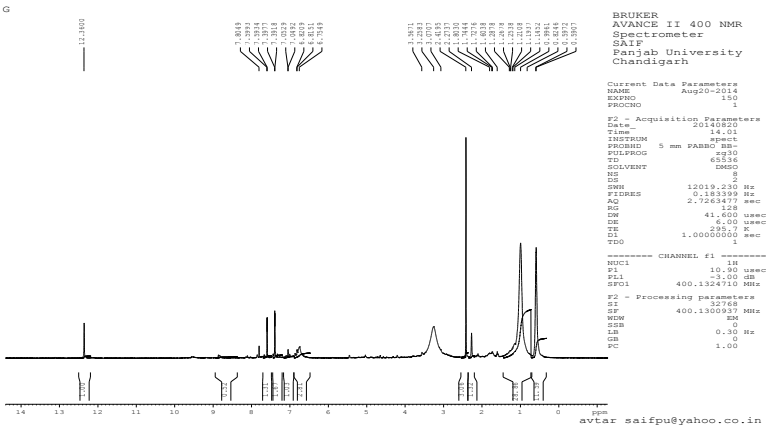

Spectrum No. 3

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