



TRAILBLAZING SYNTHESIS AND STUDY OF IMPACT OF SUBSTITUTED 1,3-THIAZOLE AND ITS NANOPARTICLES ON PHYTOTIC GROWTH OF SOME FLOWERING PLANTS

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ABSTRACT

The synthesis, spectral analysis and biological activities of 5-phenyl-2-hydroxy-chlorosubstituted-2-amino-1,3-thiazoles have been carried out. In this case 5-(2'-hydroxy-3',5'-dichlorophenyl)-4-(4'-nitrobenzoyl)-2-phenyl-amino-1,3-thiazole (E) has been screened. The compound E was synthesized from 1-(2'-hydroxy-3',5'-dichlorophenyl)-2-bromo-1,3-nonanedione(a₄) by the action of phenylthiourea. The nanoparticles of the compound E have been prepared by using ultrasonic technique. The titled compound and its nanoparticles were screened for growth promoting activity on some flowering plants viz. *Crysanthemum coronarium*, *Dahlia pinnata*, *Verbena officinalis*, *Iberis amara*.

KEYWORDS: Chalcone, thiazole, phenyl thiourea, growth promoting activities.

INTRODUCTION

Heterocyclic nucleus plays an important role in medicinal chemistry and it is a key template for the growth of various therapeutic agents. Thiazole is a heterocyclic compound featuring both a nitrogen atom and sulfur atom as part of the aromatic five-membered ring. Thiazoles and related compounds are called 1,3-azoles (nitrogen and one other hetero atom in a five-membered ring.) They are isomeric with the 1,2-azoles, the nitrogen and sulphur containing compound being called isothiazoles. Thiazoles are found naturally in the essential vitamins. Molecules that possess sulfur atoms are important in living organisms. The researchers^[1-6] have reported the synthesis of several thiazoles and also their potent biological activities such as antimicrobial^[7], antibacterial^[8-11], antifungal^[12-13], fungicidal^[14] and insecticidal agent.^[15] Chalcones and their analogues having α , β -unsaturated carbonyl system are very versatile substrates for the evolution of various reactions and physiologically active compounds.

In the present study, various 5-phenyl-2-hydroxy-chlorosubstituted-2-amino-1,3 thiazoles have been synthesized from 1,3 propanediones by using thiourea, phenyl thiourea and diphenyl thiourea. The synthesized compounds along with their nanoparticles were evaluated for their growth promoting activity on some

vegetable crop plants viz. *Crysanthemum coronarium*, *Dahlia 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 pellets, ¹H 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.

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 20°C. The mixture was allowed to stand for 30 minutes. It was poured into cold 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-(4''-nitrophenyl) chalcone (a)

To the boiling solution of the 2-hydroxy-3,5-dichloroacetophenone (0.01 mol) and p-nitrobenzaldehyde (0.01 mol) in ethanol (20 ml) a 40% solution of NaOH was added gradually. The reaction mixture was stirred mechanically at room temperature for 1 hour and kept steady for 6 to 8 hours, followed by decomposition with ice cold HCl (1:1). The yellow granules thus obtained were filtered, washed with 10% NaHCO₃ solution and then crystallized from ethanol-acetic acid mixture to obtain the compound (a).

Preparation of 1-(2'-hydroxy-3',5'-dichlorophenyl)-2,3-dibromo-3-(4''-nitrophenyl)-propan-1-one (a₁)

2'-Hydroxy-3',5'-dichlorophenyl-4-(4''-nitrophenyl)chalcone (a) (0.001 M) was suspended in bromine-glacial acetic acid reagent (25% w/v) (6.4 ml). The reagent was added dropwise with constant stirring and the reaction mixture was kept at room temperature for about 30 minutes. The solid product, thus separated, was filtered and washed with a little petroleum ether to get the compound (a₁).

Preparation of 2-(4''-nitrophenyl)-6,8-dichloroflavone (a₂)

1-(2'-Hydroxy-3',5'-dichlorophenyl)-2,3-dibromo-3-(4''-nitrophenyl)-propan-1-one (a₁) (0.01 mol) was dissolved in ethanol (25ml). To this, aqueous KOH solution (25 ml) was added. The reaction mixture was refluxed for 1 hour, cooled and diluted with water. The product thus separated was filtered and crystallized from ethanol to get the compound (a₂).

Preparation of 1-(2'-hydroxy-3',5'-dichlorophenyl)-3-(4''-nitrophenyl)-1,3-propandione (a₃)

2-(4''-Nitrophenyl)-6,8-dichloroflavone (a₂) (0.01 mol) was dissolved in ethanol (25ml). To this, aqueous solution of HCl (25 ml) was added. The reaction mixture was then refluxed for 1 hour, cooled, and diluted with water. The product, thus separated, was filtered, and crystallized from ethanol to get the compound (a₃).

Preparation of 1-(2'-hydroxy-3',5'-dichlorophenyl)-2-bromo-3-(4''-nitrophenyl)-1,3 propanedione (a₄)

1-(2'-Hydroxy-3',5'-dichlorophenyl)-3-(4''-nitrophenyl)-1,3-propandione (a₃) (0.01 mol) was dissolved in a mixture of ethanol and dioxane. To this, calculated amount of liquid bromine was added. The product was not separated even after standing for one hour. It was then diluted with water, washed with water several times and extracted with ether. The solvent was removed under reduced pressure to get the white solid of the compound (a₄).

Preparation of 5-(2'-hydroxy-3',5'-dichlorophenyl)-4-(4''-nitrobenzoyl)-2-phenyl- amino-1,3-thiazole (E)

1-(2'-Hydroxy-3',5'-dichlorophenyl)-2-bromo-3-(4''-nitrophenyl)-1,3-propandione (a₄) (0.01 mol) and phenyl thiourea (0.01 mol) were dissolved in ethanol. To this, aqueous KOH solution (0.02 mol) was added. The reaction mixture was refluxed for 3 hours, cooled, diluted with water and acidified with conc. HCl. The product, thus separated, was filtered and crystallized from ethanol to get the compound (E).

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

**The UV, IR, and NMR spectral data
Compound (E)**

UV: Spectrum No. 1

The UV-Vis spectrum of the compound (E) reported in dioxane showed $\lambda_{\max}$ value 490 nm corresponding to $n \rightarrow \pi^*$ transition.

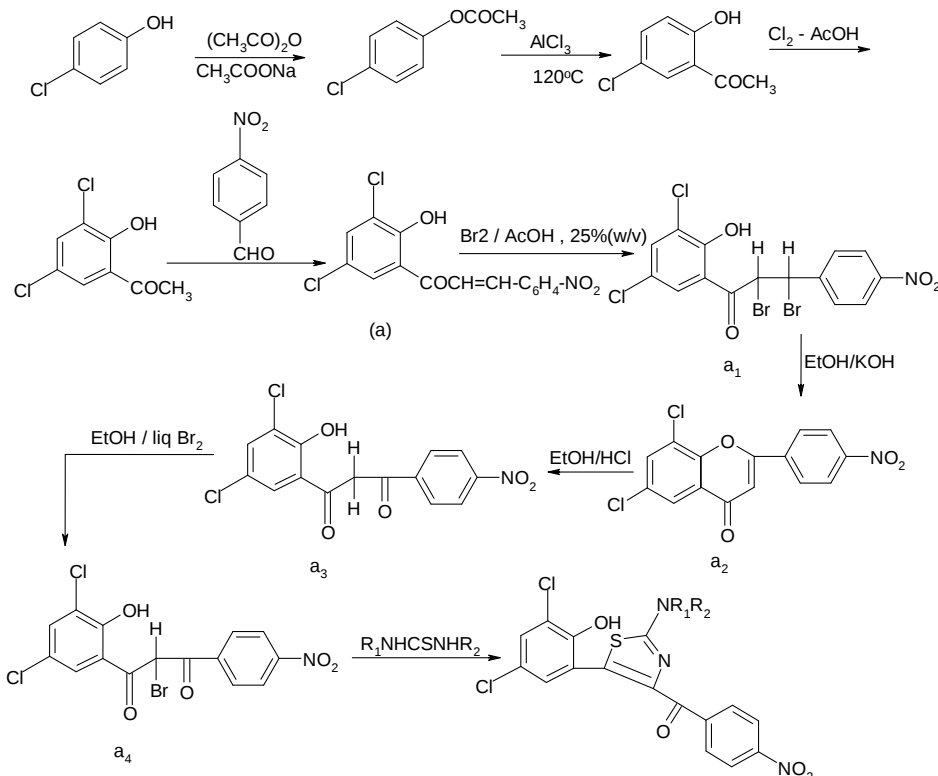
IR KBr: Spectrum No. 2

3337.18 cm⁻¹ (O-H phenolic), 2923.20 cm⁻¹ (aliphatic C-H stretching), 3073.17 cm⁻¹ (aromatic C-H stretching), 3925.40 cm⁻¹ (-NH stretching), 1218.9 cm⁻¹ (-C=N stretching), 769.23 cm⁻¹ [C-Cl stretching in aliphatic], 1053.18 cm⁻¹ [C-Cl stretching in aromatic].

PMR :- Spectrum No. 3

δ 3.58 (hump, 1H, =NH); δ 6.64(d 1H, -CH=C-H-); δ 6.69 (d, 1H, -CH=C-H-); δ 7.1 to 8.3 (m, 11H, Ar-H); δ 12.5 (s, 1H, O-H).

Scheme



Where :

- 1) $\text{R}_1 = -\text{H}, -\text{C}_6\text{H}_5$
- 2) $\text{R}_2 = -\text{H}, -\text{C}_6\text{H}_5$

Preparation of nanoparticles of the titled compounds

Ultrasonic Processor Sonapros PR-250MP was used to produce nanoparticles of the test compounds. The test compounds were dissolved in dioxane to prepare 0.1 M solutions. These solutions were taken in beakers and the probe of the sonopros 250 MP was dipped in solution. These solutions were exposed to sonopros MP 250 for 10 minutes separately. The test compounds were converted to nanoparticles. The solvent dioxane was evaporated by conventional heating method. The size of nanoparticles of the test compounds was confirmed by X-ray diffraction studies using Benchtop x-ray diffraction (XRD) instrument (Miniflex).

The thin film of the nanoparticles of the test compounds was prepared on glass slide. This slide was introduced to the X-ray diffraction instrument to get graphical information which was used for the calculation of the crystal size of test compounds.

Characterisation of size of nanoparticles of the test compounds

The crystal size of nanoparticles of the test compounds calculated by using Debye-Scherrer equation.

$$D = \frac{0.94 \lambda}{\beta \cdot \cos \theta}$$

Where,

D = The average crystalline size.

0.94 = The particle shape factor which depends on the shape and size of the particle.

λ = is the wavelength.

β = is the full width at half maximum [FWHM] of the selected diffraction 0020 peaks ($\beta = 0.545$)

θ = is the Bragg's angle obtained from 2θ values which was corresponding to the maximum intensity peak in XRD pattern ($\theta = 0.7501$ rad).

Growth Promoting Effect on some Flowering Plants

The experimental set up of the study was divided 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 solution of test compounds (0.01 dilution) prepared in dioxane before sowing.

(ii) Field experiment

Pregerminated quality seeds of *Crysanthemum coronarium*, *Dahlia 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 vegetable crop plants were done in separate beds and irrigated periodically.

The plants from each bed were divided 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, fruitification and fruit 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.

RESULT AND DISCUSSION

The titled compounds and their nanoparticles were screened for their growth promoting activity on test flowering plants viz, *Crysanthemum coronarium*, *Dahlia 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.

Table 1: Characterisation data of newly synthesized compounds.

Compounds	Molecular formula	M.P. in °C	% of yield	% of element					
				C	H	N	S	Cl	Br
	C ₈ H ₆ O ₂ Cl ₂	54	80	47.90/48	2.95/3			34.15/34.58	
a	C ₁₅ H ₉ O ₄ NCl ₂	250	70	53.10/53.25	2.40/2.66	3.98/4.18		21/21.77	
a ₁	C ₁₅ H ₉ O ₄ NCl ₂ Br ₂	72	70	36.01/36.14	1.78/1.80	2.78/2.81		14.20/14.25	32.08/32.12
a ₂	C ₁₅ H ₇ O ₄ Cl ₂ N	132	60	53.14/53.57	2.07/2.08	4.13/4.16		21.03/21.13	
a ₃	C ₁₅ H ₉ O ₅ Cl ₂ N	117	50	50.74/50.84	2.45/2.54	3.90/3.95		20.03/20.05	
a ₄	C ₁₅ H ₈ O ₅ Cl ₂ BrN	78	60	41.12/41.57	1.78/1.84	3.20/3.23		16.08/16.39	18.34/18.47
E	C ₂₂ H ₁₅ O ₄ N ₃ Cl ₂ S	168	70	54/54.09	3.0/3.07	8.56/8.60	6.50/6.55	14.50/14.54	

Activity of the test compounds E.

Table No. (02): 5-(2'-Hydroxy-3',5'-dichlorophenyl)-4-(4"-nitrobenzoyl)-2(phenylamino)-1,3-thiazole (E)

Periodicity of Observations [in days]	<i>Crysanthemum coronarium</i>				<i>Dahalia pinnata</i>				<i>Verbena officinalis</i>				<i>Iberis amara</i>			
	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

Impact of the compound 5-(2'-Hydroxy-3',5'-dichlorophenyl)-4-(4''-nitrobenzoyl)-2(phenylamino)-1,3-thiazole (E) on phytotoxic growth of *Crysanthemum coronarium*

Control



Treated



15 days



30 days



45 days



90 days

Impact of the compound 5-(2'-Hydroxy-3',5'-dichlorophenyl)-4-(4''-nitrobenzoyl)-2(phenylamino)-1,3-thiazole (E) on phytotoxic growth of *Dahalia pinnata*

Control



Treated



15 days



30 days



45 days



90 days

Impact of the compound 5-(2'-Hydroxy-3',5'-dichlorophenyl)-4-(4''-nitrobenzoyl)-2(phenylamino)-1,3-thiazole (E) on phytotoxic growth of *Verbena officinalis*

Control



Treated



15 days



30 days

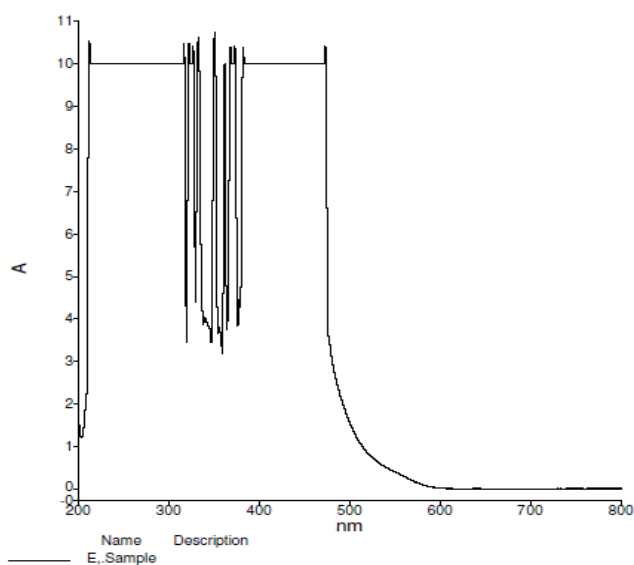


45 days



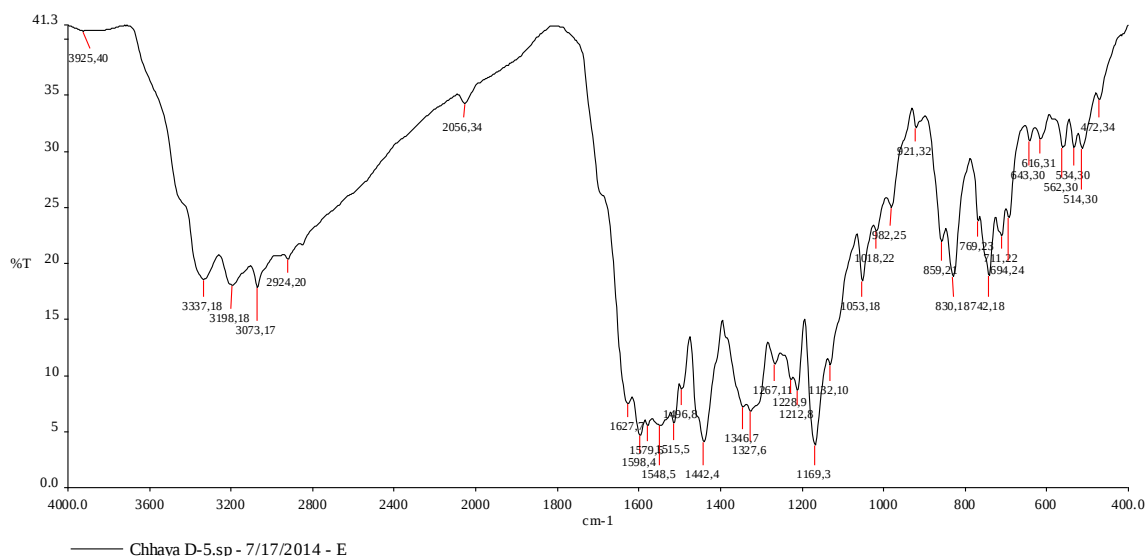
90 days

Impact of the 5-(2'-Hydroxy-3',5'-dichlorophenyl)-4-(4''-nitrobenzoyl)-2(phenylamino)-1,3-thiazole (E) on phytotic growth of *Iberis amara*

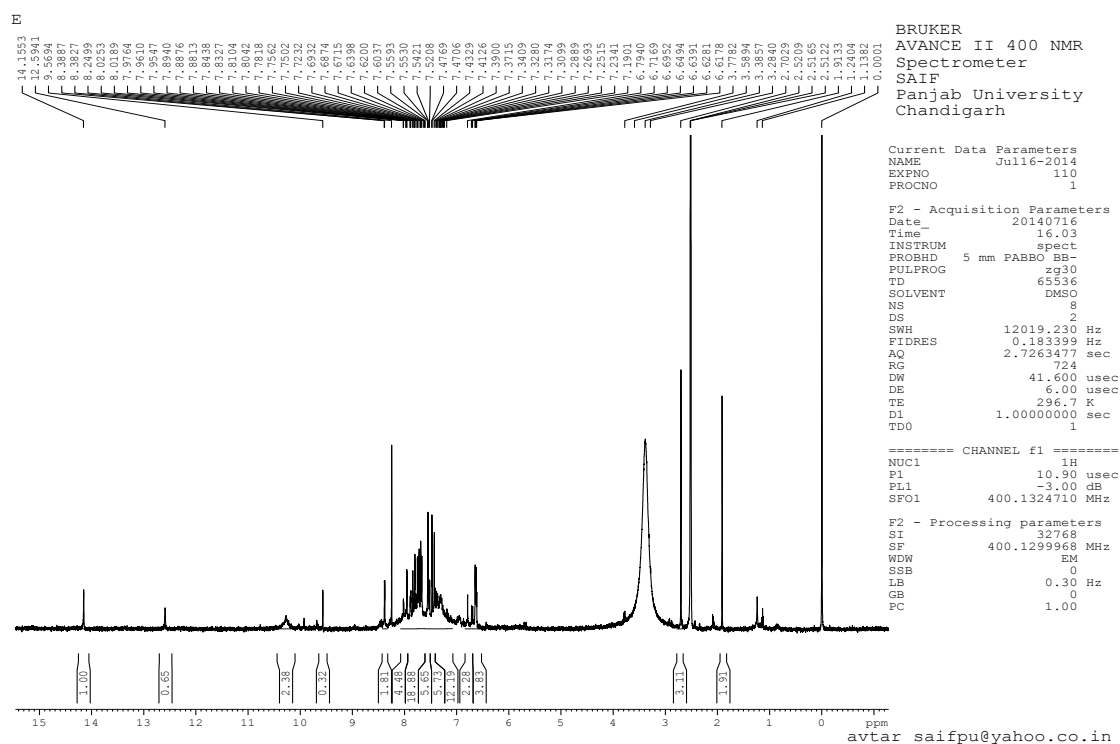


Spectrum No. 1.

RC SAIF PU, Chandigarh



Spectrum No. 2.



Spectrum No. 3.

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