



Research Article

SYNTHESIS AND CHARACTERIZATION OF 5-SUBSTITUTED SERIES OF 3-(4-CHLOROPHENYL)-2,4-DITHIO-1,3,5-TRIAZINES CONTAINING CHALCONE MOITIES

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ABSTRACT

Present research comprises, isomerisation of series of (2E)-1-{4-[2-(4-chlorophenyl)imino-4-substitutedimino]amino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one (**Ia-e**) were successfully carried using sodium bicarbonate in aqueous ethanol in to (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-substituted-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ia-e**). The structure of all the synthesized compounds was justified on the basis of chemical tests, elemental analysis and spectral characterizations.

Keywords:(2E)-1-{4-[2-(4-chlorophenyl)imino-4-substitutedimino]amino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4 dimethoxyphenyl)prop-2-en-1-one (**Ia-e**), sodium bicarbonate, ethanol and spectral Characterization etc.

INTRODUCTION

Chemistry of the heterocycles involves structural activities of the drugs are directly related to the heterocycles present their in and substituent attached. Heterocycles having size five member, six member, eight member, spiro-heterocycles and fused heterocycles are the potent biological drugs. Also the heterocycles containing sulphur and nitrogen as a hetero atom in the ring resulted excellent drugs. 1,3,5-Triazine is nitrogen ring heterocycle containing alternate nitrogen with carbon in the six member ring. Such heterocycles having appropriate substituent's also enhances existing activities of the basic nucleus of 1,3,5-triazine.

A big research carried on the synthesis and biological activities of the 1,3,5-triazines. Tayade¹, Khrobragade², Panpalia³, Pund⁴, Waghmare⁵ and Shelke⁶ reported different derivatives of 1,3,5-triazine for variety of biological applications.

Taking all the references in the consideration, it was thought to synthesize the novel series of 1,3,5-triazine and validate the synthesize compounds using elemental analysis, IR spectra and PMR spectra. The method employed for the synthesis is cheaper, convenient and less time consumable. It includes, isomerisation the series of (2E)-1-{4-[2-(4-chloro phenyl)imino-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (**Ia-e**) on refluxing with 10% aqueous sodium bicarbonate solution in ethanol to isolate series (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-substituted-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ia-e**).

MATERIALS & METHOD

Materials

The entire chemicals used in the present research were MERCK'S Chemicals (India Made). Starting compounds (**Ia-e**) were synthesized by literature method¹⁻⁵.

Method

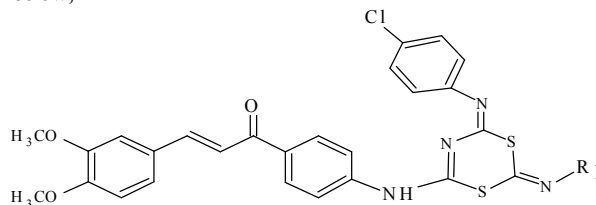
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on SCIMADZU FTIR spectrometer in the range 4000-400 cm⁻¹ in KBr pellets. PMR spectra were recorded on BRUKER AVANCE II 400 NMR spectrometer with TMS as an internal standard using CDCl₃ and DMSO-d₆ as a solvent.

General Procedure

(2E)-1-{4-[2-(4-chlorophenyl)imino-4-substitutedimino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ia-e**) were isomerised by 10% aqueous sodium bicarbonate solution in ethanol medium. During heating reactant dissolved into the solvent. After distillation of excess solvent yellow crystals were separate out. The changing of colour shows that the product was obtained. It was recrystallized from glacial acetic acid to obtained (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-substituted-1,3,5-triazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ia-e**).

The tentative reaction is given below,

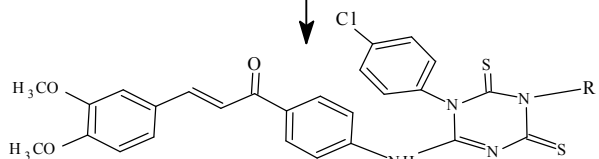
Reaction-



(2E)-1-{4-[2-(4-chlorophenyl)imino-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(Ia-e)

NaHCO₃/EtOH Isomerisation



(2E)-1-{4-[2,4-Dithio-3-(4-chlorophenyl)-5-substituted-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(IIa-e)

Where R₁ = -allyl, ethyl, -t-butyl, -phenyl, -p-cl-phenyl

Reaction Scheme

Similarly, (2E)-1-{4-[2-(4-chlorophenyl)imino-4-(prop-2-en-1-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ia**), (2E)-1-{4-[2-(4-chlorophenyl)imino-4-ethylimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ib**), (2E)-1-{4-[2-(4-chlorophenyl)imino-4-(2-methyl prop-2-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ic**), (2E)-1-{4-[2-(4-chlorophenyl)imino-4-phenylimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Id**) and (2E)-1-{4-[2-(4-chlorophenyl)imino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ie**) were interacted with 10% Sodium bicarbonate in ethanol by the given method to isolate (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(prop-2-en-1-yl)-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIa**), (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-ethyl-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIb**), (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(2-methylprop-2-yl)-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIc**), (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-phenyl-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IId**) and (2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(4-chlorophenyl)-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIe**) respectively.

RESULT & DISCUSSION

Spectral analysis of the all the synthesized compound using elemental analysis, IR Spectra and PMR spectra is given below,

(2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(prop-2-en-1-yl)-1,3,5-triazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIa)

Yellow solid, C₂₉H₂₅N₄O₃S₂Cl, Yield-80%, M.P.-178^oC, Composition-found(calculated) C-60.16 (60.35), H-4.54 (4.37), N-9.71 (9.71), S-11.17 (11.11) and Cl-5.42 (6.14); **FTIR (KBr) v cm⁻¹**-3074.33 (ArC-H stretching), 3368.53 (N-H stretching), 1686.03 (C=O stretching), 1141.89 (C=S stretching), 1032.06 (C-O-C stretching) and 1215.12 (C-N stretching); **¹H NMR (400 MHz CDCl₃ δ)** singlet of 6H of -OCH₃ at δ 3.41ppm, singlet of 2H of -CH=CH- at δ 3.55-3.72ppm, multiplet of 11H of Ph at δ 6.65-7.88ppm, singlet of 1H of -NH at δ 8.92ppm and pentate of 1H, 2H and of 2H of allyl at δ 2.23, 1.30 and 2.12 respectively; Mol. Wt.: 576.5.

(2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-ethyl-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIb)

Yellow solid, C₂₈H₂₅N₄O₃S₂Cl, Yield-83%, M.P.-168^oC, Composition-found(calculated) C-59.64 (59.51), H-4.94 (4.46), N-9.91 (9.91), S-11.16 (11.35) and Cl-6.74 (6.27); **FTIR (KBr) v cm⁻¹**-3075.11 (ArC-H stretching), 3366.53 (N-H stretching), 1674.55 (C=O stretching), 1135.86 (C=S stretching), 1033.66 (C-O-C stretching) and 1216.45 (C-N stretching); **¹H NMR (400 MHz CDCl₃ δ)** singlet of 6H of -OCH₃ at δ 3.39ppm, singlet of 2H of -CH=CH- at δ 3.65-3.78ppm, multiplet of 11H of Ph at δ 6.67-8.12ppm, singlet of 1H of -NH at δ 8.88ppm and quartet of 2H and triplet of 3H of ethyl at δ 1.47 and δ 1.39 respectively; Mol. Wt.: 564.5.

(2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(2-methylprop-2-yl)-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIc)

Yellow solid, C₃₀H₂₉N₄O₃S₂Cl, Yield-79%, M.P.-181^oC, Composition-found(calculated) C-60.61 (60.75), H-4.16 (4.93), N-9.45 (9.45), S-10.58 (10.81) and Cl-5.99 (5.98); **FTIR (KBr) v cm⁻¹**-3074.85 (ArC-H stretching), 3356.06 (N-H stretching), 1684.9 (C=O stretching), 1141.78 (C=S stretching), 1067.68 (C-O-C stretching) and 1219.44 (C-N stretching); **¹H NMR (400 MHz CDCl₃ δ)** singlet of 6H of -OCH₃ at δ 3.38ppm, singlet of 2H of -CH=CH- at δ 3.62-3.81ppm, multiplet of 11H of Ph at δ 6.65-8.09ppm and singlet of 1H of -NH at δ 9.841ppm; Mol. Wt.: 592.5.

(2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-phenyl-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIId)

Yellow solid, C₃₂H₂₅N₄O₃S₂Cl, Yield-76%, M.P.-167^oC, Composition-found(calculated) C-62.34 (62.68), H-4.26 (4.11), N-9.14 (9.14), S-10.63 (10.46) and Cl-6.76 (5.78); **FTIR (KBr) v cm⁻¹**-3056.81 (ArC-H stretching), 3360.89 (N-H stretching), 1685.80 (C=O stretching), 1131.50 (C=S stretching), 1029.16 (C-O-C stretching) and 1220.03 (C-N stretching); **¹H NMR (400 MHz CDCl₃ δ)** singlet of 1H of -NH at δ 9.841ppm, singlet of 9H, CH₃ at δ 1.32ppm, singlet of 6H of -OCH₃ at δ 3.40ppm, singlet of 2H of -CH=CH- at δ 3.63-3.74ppm, multiplet of 16H of Ph at δ 6.64-8.12ppm and singlet of 1H of -NH at δ 9.84ppm; Mol. Wt.: 612.5.

(2E)-1-{4-[2,4-dithio-3-(4-chlorophenyl)-5-(4-chlorophenyl)-1,3,5-triazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IXee)

Yellow solid, C₃₁H₂₃N₄O₃S₂Cl₂, Yield-80%, M.P.-184^oC, Composition-found(calculated) C-58.91 (58.67), H-4.13 (3.65), N-8.83 (8.83), S-10.22 (10.11) and Cl-11.42 (11.17); **FTIR (KBr) v cm⁻¹**-3069.36 (ArC-H stretching), 3365.91 (N-H stretching), 1684.52 (C=O stretching), 1138.33 (C=S stretching), 1032.46 (C-O-C stretching) and 1220.19 (C-N stretching); **¹H NMR (400 MHz CDCl₃ δppm)** singlet of 6H of -OCH₃ at δ 3.40ppm, singlet of 2H of -CH=CH- at δ 3.64-87ppm, multiplet of 15H of Ph at δ 6.58-8.14ppm and singlet of 1H of -NH at δ 9.84ppm; Mol. Wt.: 634.

CONCLUSION

The entire synthesized compound were verified, and confirmed by their chemical tests, elemental study, IR spectra and PMR

spectra. Similar method and procedure can be adopted for the synthesis of variety of derivatives of 1,3,5-triazines.

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