ejpmr, 2016,3(7), 337-339.

SJIF Impact Factor 3.628

EUROPEAN JOURNAL OF PHARMACEUTICAL AND MEDICAL RESEARCH

www.ejpmr.com

Research Article

ISSN 2394-3211

EJPMR

SYNTHESIS AND SPECTRAL CHARACTERIZATION OF (2*E*)-1-[4-(2-PHENYLIMINO-4-SUBSTITUTEDIMINO-1,3,5-DITHIAZINO-6-YL) AMINOPHENYL]-3-(3,4-DIMET-HOXYPHENYL)PROP-2-EN-1-ONE

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Article Received on 27/04/2016 **Article Revised on 17/05/2016** **Article Accepted on 07/06/2016**

ABSTRACT

Recently, novel series of (2*E*)-1-[4-(2-phenylimino-4-substitutedimino-1,3,5-dithiazino)-aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIIa-e)** had been synthesized in this laboratory by refluxing (2*E*)-1-[4-(5-phenyl-2,4-dithiobiureto)phenyl]-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(I)** with substituted alkyl/aryl isocyanodichlorides **(IIa-e)** in acetone medium in 1:1 molar proportion for two hours. The structures of all the synthesized compounds were justified on the basis of chemical tests, elemental analysis and spectral studies.

KEYWORDS: alkyl/aryl isocyanodichlorides **(IIa-e).**

INTRODUCTION

Heterocycles are the unit parts of the drugs. All the properties of the heterocycles are well influenced by the nature of heterocycle present in the drug structure. Activities of all the drugs are specific to the specific disease; heterocycles also have the specific activity to the variety of diseases. Sometimes the activities of the drugs also get varied due to the substituent attached to the basic nucleus of the heterocycles. Now-a-days bacteria are becoming resistant to the existing drugs. Need of the today's age is to develop the new variety of drugs in the laboratory.

1,3,5-dithiazine are special class of the heterocycles. Every 1,3,5-dithiazino nucleus has diverse significance relative to the substituent linked to the fundamental nucleus of the 1,3,5-dithiazine. It was also observed during literature survey that, variety of applications of 1,3,5-dithiazines are due to the structural arrangement of the six member sulphur and nitrogen containing ability. Every 1,3,5-derivative is unique to its activity. Some of the 1,3,5-dithazines are also reported for their useful role in the automobile engineering and mechanical engineering.

Keeping all the referenced ideas in mind, it was planned to synthesize some derivatives of 1,3,5-dithiazine in laboratory by one step cyclisation of (2*E*)-1-[4-5-phenyl-2,4-dithio-biureto) phenyl]-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(I)** with N-substitutedisocyanodichlorides **(IIa-e)** in acetone medium to isolate (2*E*)-1-[4-(2-phenylimino-4-substitutedimino-1,3,5-dithiazino)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIIa-e).**

MATERIAL AND METHOD

Materials

All the chemicals used in this method are MERCKS (India Made). Compounds (I) is synthesized using reference method.[7-9]

Method

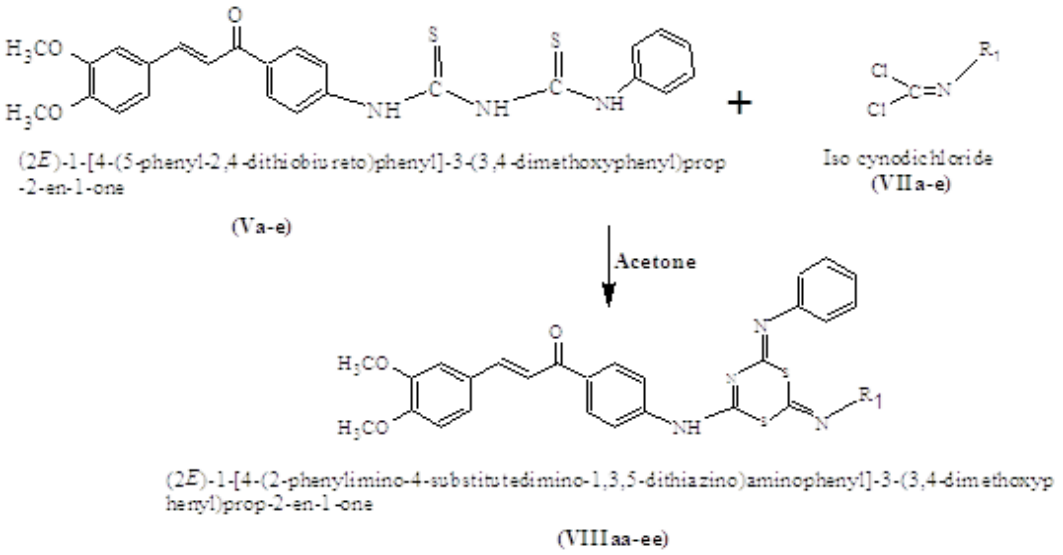
Method used in the present research is conventional refluxing on water bath at stable temperature.

EXPERIMENTAL

General Procedure

The interaction of (2*E*)-1-[4-(5-phenyl-2,4-dithiobiureto)phenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(I)** with alkyl/aryl isocyanodichloride **(IIa-e)** in 1:1 molar ratio refluxed on water bath in acetone medium for 2 hours. During heating evolution of hydrochloride gas was clearly noticed. Product obtained was basified with dilute ammonium hydroxide and recrystalised from ethanol.

Similar, procedure was adopted for the synthesis of all the derivatives in the series. The probable reaction for the formation of products is depicted below.



Reaction Scheme

Similarly, (2*E*)-1-{4-[5-phenyl-2,4-dithiobiureto] phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one**(I),** were interacted with N-allyl isocyanodichloride **(IIa),** N-ethyl isocyanodichloride **(IIb)** N-t-butyl isocyanodichloride **(IIc)**, N-phenyl Isocyanodichloride **(IId),** N-(4-chlorophenyl) Isocyanodichloride **(IIe)** by above mentioned method to (2*E*)-1-{4-[2-phenylimino-4- (prop-2-en-1-yl) imino-1,3,5-dithiazino-6-yl] aminophen yl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(IIIa),** (2*E*)-1-[4-(2-phenyl imino-4-ethylimino)-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(IIIb),** (2*E*)-1-{4-[2-phenylimino-4-(2-methylprop-2-yl) imino-1,3,5-dithiazino-6-yl] aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(IIIc),** (2*E*)-1-[4-(2-phenylimino-4-phenylimino) -1,3,5-dithia-zino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIId)** and (2*E*)-1-{4-[2-phenylimino-4-(4-chlorophenyl) imino-1,3,5-dithiazino-6-yl] amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIIe)** respectively.

RESULT AND DISCUSSION

Reaction data obtained and spectral characterization of all the synthesized compounds **(IIIa-e)** are given below,

Spectral Analysis

(2*E*)-1-{4-[2-phenylimino-4-(prop-2-en-1-yl)imino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIa)

Cream yellow solid, $C_{28}H_{25}N_4O_3S_2$, Yield-68%, M.P.-175^{0}C Composition-found(calculated) C-63.79 (63.49), H-4.88 (4.76), N-10.58 (10.58) and S-11.65 (12.11); **FTIR (KBr) ν cm^{-1}**-302.55-3016.89 (ArC-H stretching), 1581.26 (S-C=N stretching), 731.38 (C-S stretching), 1658.66 (C=O stretching), 1027.65 (C-O-C stretching) and 3321.26 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.41ppm, doublet of 2H, -CH=CH- at δ 2.92-3.76ppm, multiplet of 7H of Ph at δ 6.77-8.24ppm, Singlet of 1H of NH at δ 8.31ppm, multiplet of 5H, Ph at δ 6.79-8.02ppm and pentate of 1H, doublet 2H and doublet of 2H of allyl at δ2.22, 1.31 and 2.12respectively; Mol. Wt.: 529.

(2*E*)-1-[4-(2-phenylimino-4-ethylimino)-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIb)

Dark yellow solid, $C_{27}H_{25}N_4O_3S_2$, Yield-76%, M.P.-156^{0}C, Composition-found(calculated) C-64.89 (62.65), H-4.91 (4.87), N-10.82 (10.82) and S-11.73 (12.39); **FTIR (KBr) ν cm^{-1}**-3022.99-3019.21 (ArC-H stretching), 1579.46 (S-C=N stretching), 732.78 (C-S stretching), 1658.27 (C=O stretching), 1028.91 (C-O-C stretching) and 3321.26 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.43ppm, doublet of 2H, -CH=CH- at δ 2.81-3.46ppm, multiplet of 7H of Ph at δ 6.67-8.32ppm, Singlet of 1H of NH at δ 8.46ppm, multiplet of 5H, Ph at δ 6.77-8.02ppm and quartet of 2H and triplet of 3H of ethyl at δ1.41 and δ 1.32respectively; Mol. Wt.: 517.

(2*E*)-1-{4-[2-phenylimino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino- 6-yl] aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIc)

Yellow solid, $C_{30}H_{30}N_4O_3S_2$, Yield-68%, M.P.-157^{0}C, Composition-found(calculated) C-65.21 (64.49), H-5.9 (5.41), N-10.03 (10.03) and S-11.32 (11.48); **FTIR (KBr) ν cm^{-1}**-3024.26-3018.24 (ArC-H stretching), 1584.24 (S-C=N stretching), 727.16 (C-S stretching), 1658.56 (C=O stretching), 1030.19 (C-O-C stretching) and 3327.34 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.41ppm, doublet

of 2H, -CH=CH- at δ 2.68-3.76ppm, multiplet of 7H of Ph at δ 6.65-8.01ppm, Singlet of 1H of NH at δ 8.34ppm, multiplet of 5H, Ph at δ 6.77-8.15ppm and singlet of 9H, CH_3 at δ 1.296ppm; Mol. Wt.: 558.

(2*E*)-1-[4-(2-phenylimino-4-phenylimino-1,3,5-dithiazino- 6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIc)

Yellow solid, $C_{32}H_{26}N_4O_3S_2$, Yield-74%, M.P.- 166^{0}C ^{0}C, Composition-found(calculated):C-65.02(64.41), H-4.87(4.53), N-9.98(9.68) and S-10.98 (11.08); **FTIR (KBr) ν cm^{-1}**-3091.66-3006.82 (ArC-H stretching), 1591.16 (S-C=N stretching), 730.97 (C-S stretching), 1656.74 (C=O stretching), 1026.06 (C-O-C stretching) and 3373.27 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** singlet of 6H of two –OCH_3 at δ3.89ppm, doublet of 2H of –CH=CH- at δ 3.46ppm- δ 3.63ppm, multiplets of 17H of ph at δ 6.60ppm-8.81ppm and singlet of 1H of –NH at δ 9.68ppm.; MASS Spectra: base peak at m/z= 288.06 ($C_{15}H_{13}NO_3Cl$)and fragments at 93.99 (C_6H_7N), 136.03 (C_7H_7NS), 289.08 ($C_{15}H_{14}NO_3Cl$) and 302.99($C_{17}H_{16}O_3Cl$).

(2*E*)-1-{4-[2-phenylimino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIe)

Yellow solid, $C_{32}H_{25}N_4O_3S_2Cl$, Yield-74%, M.P.-179^{0}C, Composition-found(calculated) C-61.87 (62.68), H-4.26 (4.11), N-9.14 (9.14), S-10.54 (10.46) and Cl-6.45(5.78); **FTIR (KBr) ν cm^{-1}**-3034.164-3019.46 (ArC-H stretching), 1578.21 (S-C=N stretching), 736.65 (C-S stretching), 1645.04 (C=O stretching), 1028.16 (C-O-C stretching) and 3324.25 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.41ppm, doublet of 2H, -CH=CH- at δ 2.62-3.76ppm, multiplet of 7H of Ph at δ 6.67-8.15ppm, Singlet of 1H of NH at δ 8.21ppm, multiplet of 5H, Ph at δ 6.46-7.62ppm and multiplet of 4H, Ph at δ 6.37-7.92ppm; Mol. Wt.: 612.5.

CONCLUSION

All the synthesized compounds in the present reaction scheme **(IIIa-e)** are verified and confirmed by making use of several chemical test**s**. Spectral data analysis of the synthesized compounds **(IIIa-e)** also support and confirm the desired product moieties. A variety of chalcone based 1,3,5-dithiazine derivatives can be synthesized for their antimicrobial activities adopting the method. This method is cheaper, convenient and less time consumable.

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