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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF THIAZOLIDINONE COMPOUND CONDENSED WITH AZO MOIETY

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ABSTRACT

Thiazolidine is a five membered ring with one nitrogen and one sulphur atoms. Hydrazides and their hetrocyclized derivatives also found to passes an important role in biological activities. Thiazolidinone derivatives showed good pharmacological properties. On the basis of literature study the objective of the present work was to prepare new derivatives of hydrazide containing thiazolidine moiety. In the present work we have prepared some of novel thiazolidinone derivatives condensed with azo moiety. The newly synthesized compounds were analysed by IR, 1H-NMRand ^{13}C- NMR spectral analysis.

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INTRODUCTION

Heterocyclic composites are an important class of organic compounds possessing comprehensive applications in various fields of learning[1–7]. One of the most prominent heterocyclic motifs, thiazolidine, is a five-membered heterocycle system having the formula C3H7NS containing one nitrogen and one sulfur atom, and which exhibits notable medicinal and pharmaceutical properties. In the thiazolidine nucleus, a large number of substitutions are possible on 2, 4 and 5 positions responsible for enhancing the compound's pharmaceutical importance.Thiazolidine and its composites arekey components of many natural products and drugs. The synthesis and the biological activities of thiazolidinone derivatives have been the subject of substantial research. The thiazolidinone derivatives are privileged heterocyclic compounds owing to their contribution as biologically active chromophores along with pharmaceutical application in disease treatment such as anticancer (Zhou et al., 2020, Sigalapalli et al., 2021, Rani et al., 2020), anti-inflammatory (Shawky et al., 2019), antimicrobial activities (Arshad and Ahmad, 2020, Beniwal and Jain, 2019), antioxidant activities (Zhang et al., 2018) and antileishmanial (Bhat et al., 2020). Also, thiazolidinone derivatives have been utilized as a hybrid drug in medicines, and their activities were compared with marketed drugs. Medicinal Chemistry has its pedigrees in several outlets of chemistry andbiology. It is alarmed with the study, identification and synthesis of the metabolicproducts of drugs and related compounds. It also attempts to establish relationshipbetween structure and function and to link biodynamic behaviour with chemicalreactivity and physical properties. However, essentially it concerns with theunderstanding of mechanisms of action of drugs. Besides this, medicinal chemistryalso involves the isolation, characterization and synthesis of compounds that can beused in medicine for the prevention, treatment and cure of diseases. Thus, it provideschemical basis for the interdisciplinary field of therapeutics.

EXPERIMENTAL

Solvents were employed as commercial anhydrous grade. The column chromatography was done over the silica gel (100-120 mesh). Melting points were determined in open capillary tube and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on Bruker advance II-400 MHz spectrometer.

MATERIAL AND METHODS

All solvents were labouring as commercial anhydrous mark without further Refining. The column chromatography was carried out over silica gel (100-120esh). Melting points determined by open capillary tube. ^{1}H NMR spectra were

recorded on a Bruker 400 MHz spectrometer in DCl3solvent TMS as internal standard. The crude product was recrystallizing from 80 percentage ethanol.

Present Work: In the present work, first aromatic substituted Schiff bases were synthesized by condensing substituted amine, the synthesized Schiff base was reacting with thioglycolic acid to form Thiazolidinone, this product further diazotise to form a final product.

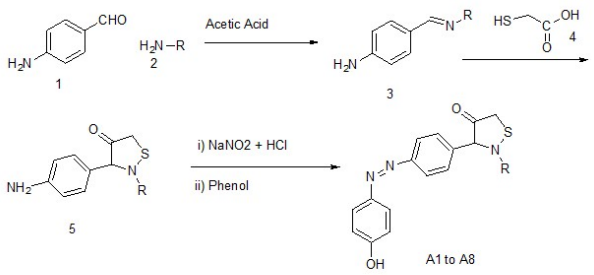
Step I: General Procedure for the synthesis of Schiff base: The compound 1(0.01mmole) was dissolve in 5 to 10 ml of ethanol in a round-bottom flask. The to this 0.01mmole of Aromatic amine (2) was added slowly with constant stirring, then to the mixture in trace amount of acetic acid was added to act as a catalyst and promote imine formation. The reaction mixture was heated nearby 40 to 60^0C (Depending on Substrate) for 3 to 5 hours. reaction progress was monitor via TLC (Thin Layer Chromatography) using an applicable solvent system.After completion, cool the reaction mixture to room temperature.the solid product was filtered, and wash with cold ethanol or water to remove impurities. the crude product was dried under vacuum or in a desiccator. Recrystallize the Schiff base from ethanol or an ethanol-water mixture to obtain a pure compound.

Step II: General Procedure for the synthesis ofThiazolidinone: In a round-bottom flask, dissolve 0.1 mmole of the Schiff base in 5–10 mL of dry ethanol.Add 0.1 mmol of thioglycolic acid to the reaction mixture slowly drop by drop Add a few drops of (catalytic amount) of $ZnCl_2$ to promote cyclization.Reflux the mixture for 4–6 hours.Monitor the reaction progress using TLC (e.g., ethyl acetate/n-hexane as solvent system).After accomplishment, cool the reaction mixture to normal temperature. the dense product was filtered, and wash-down with cold ethanol or water to remove impurities. the rudimentary product was dried out under vacuum or in a desiccator.Recrystallize the product from ethanol or an ethanol-water blend to obtain a pure compound.

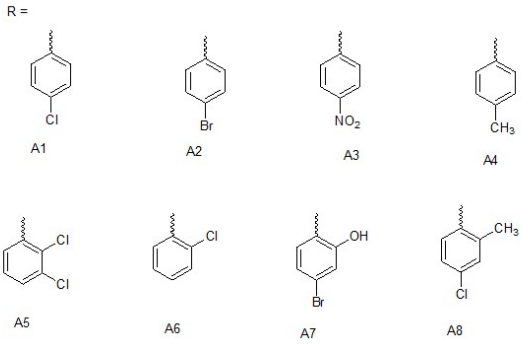
Step II: General Procedure for the synthesis Diazotization: 0.1 mmol of the aromatic amine was dissolve in 5–10 ml of 2 M HCl in a flask.The reaction mixture in an ice bath and cool the solution to 0–5 °C. the temperature of the reaction was maintainingat about 0–5 °C throughout the reaction to prevent decomposition of the Diaz onium salt.Separately, 0.1 mmol of sodium nitrite ($NaNO_2$) was dissolve in 2–3 mL of cold distilled water, sodium nitrite solution was added dropwise to cold amine/HCl solution while stirring. By maintain the temperature below 5 °C. inthe reaction generate nitrous acid in situ, which reacts with the amine to form the Diazonium salt. The excess nitritewas destroyed by adding small amount urea. Then to this solution 0.1 mmole phenol was added then coloured product was isolated, recrystallizes from ethanol to get compound A_1-$_8$.

Spectral Data

1) (A_1): Dark Red solid, 50%,M.P.284 °C, FT-IR (KBr, cm−1): 735 For C-Cl str, 1450 For C=C Ar str, 1515 for N=N str, 1634 for Carbonyl str, 2924 for C-H str, 3350 for Ar–O-H Str. 1H-NMR ($CDCl_3$, 400 MHz) δ: 8.27 (s, 1H, OH), 7.41–7.45 (m, 4H, Ar–H), 7.33 (d, 2H, Ar–H, J = 8.4 Hz), 6.96 (d, 2H, Ar–H, J = 8.4 Hz), 6.79–6.88 (m, 5H, Ar–H) ppm; 4.79(S1H) 5.79(S, 2H) ppm.



Scheme 1



13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 45.2 (Aliphatic Alpha Carbon), 85.9(Benzylic Carbon), 110.4(Ar Carbon), 122.3ppm,123.0 (Ar Carbon),129.5(ArCarbon),137.0(Ar Carbon),141.7(Ar Carbon),150.8(Ar Carbon),160.6(Ar-C), 206.7(Carbonyl), 116.4 (Ar Carbon),ppm;

2) (A_2): Red solid, 53%, M.P. 284 °C, FT-IR (KBr, cm^{-1}): 1634 (C=O Str), 751 for C-Br Str, 1460 for C=N Ar str, 1670 for Carbonyl str, 2824 for C-H str, 3350 for Ar–O-H Str . 1H-NMR ($CDCl_3$, 400 MHz) δ7.25–7.35 (m, 4H, Ar–H), 7.40(d, 2H, Ar–H, J = 8.4 Hz), 6.9 (d, 2H, Ar–H, J = 8.4 Hz), 6.79–6.88 (m, 5H, Ar–H) ppm; 3.37(s1H)ppm, ppm; 5.08(s2H) ppm ^{13}C-NMR (DMSO-d_6, 100 MHz) *δ*:) δ: 55.3 (Aliphatic Alpha Carbon), 90.1 (Benzylic Carbon), 112.1(Ar Carbon), 120.2ppm,125.6 (Ar Carbon), 130.1(Ar Carbon),136.2(Ar Carbon),141.7(Ar Carbon), 150.8(Ar Carbon), 160.6(Ar-C), 206.7(Carbonyl), 116.4 (Ar Carbon),ppm;

(A_3): Red solid, 54%, 1.85 g. mp 133–135°C. FT-IR (KBr, cm−1): 1275 for –NO2 Str, 1480 for C=N Ar str, 1630 for Carbonyl str, 2950 for C-H str, 3190 for Ar–O-H Str . 1H-NMR ($CDCl_3$, 400 MHz) δ7.85–8.10 (m, 4H, Ar–H), 8.40(d, 2H, Ar–H, J = 8.4 Hz), 8.9 (d, 2H, Ar–H, J = 8.4 Hz), 6.65–6.78 (m, 5H, Ar–H) ppm; 3.21 (s1H) ppm, ppm; 5.91(s2H) ppm 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 45.2 (Aliphatic Alpha Carbon), 85.9 (Benzylic Carbon), 110.4(Ar Carbon), 122.3ppm,123.0 (Ar Carbon), 129.5(Ar Carbon),137.0(Ar Carbon),141.7(Ar Carbon), 150.8(Ar Carbon), 160.6(Ar-C), 206.7(Carbonyl), 116.4 (Ar Carbon),ppm; **(A_4):**Redish solid, 53%, 1.50 g. mp 79–81 °C. 1480 for N=N Ar str, 1550 for C=C str, 1670 for Carbonyl str, 2850 for C-H str, 3120 for Ar–O-H Str .1H-NMR ($CDCl_3$, 400 MHz) δ: 9.27 (s, 1H, OH), 7.43–7.48 (m, 4H, Ar–H), 7.35 (d, 2H, Ar–H, J = 8.4 Hz), 6.99 (d, 2H, Ar–H, J = 8.4 Hz), 6.80–6.89 (m, 5H, Ar–H) ppm; 4.80(S1H) 4.90 (S, 2H) ppm. 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 43.2 (Aliphatic Alpha Carbon), 90.5 (Benzylic Carbon), 112.4(Ar Carbon), 125.3ppm,126.0 (Ar Carbon), 130.5(Ar Carbon),139.0(Ar Carbon),144.7(Ar Carbon), 155.8(Ar Carbon), 160.6(Ar-C), 214.7(Carbonyl), 117.4 (Ar Carbon),ppm; **(A_5):**Red solid, 54%, 1.44 g. mp 133–135 °C. 730 C-Cl str, 1430 for N=N Str, 1480 for C=C Ar str, 1630 for Carbonyl str, 2800 for C-H str, 3250 for Ar–O-H Str . 1H-

NMR ($CDCl_3$, 400 MHz) δ: 8.28 (s, 1H, OH), 7.42–7.46 (m, 4H, Ar–H), 7.35 (d, 2H, Ar–H, J = 8.4 Hz), 6.97 (d, 2H, Ar–H, J = 8.4 Hz), 6.25–7.10 (m, 5H, Ar–H) ppm;4.79(S1H) 5.79 (S, 2H) ppm. 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 47.2 (Aliphatic Alpha Carbon), 86.9 (Benzylic Carbon), 115.4(Ar Carbon), 124.3ppm,125.0 (Ar Carbon), 127.5(Ar Carbon),139.0(Ar Carbon),143.7(Ar Carbon), 155.8(Ar Carbon), 161.6(Ar-C), 208.7(Carbonyl), 119.4 (Ar Carbon),ppm;

(A_6):: Yellow solid, 85%, 0.45 g. mp 243–245 °C. FT-IR (KBr, cm−1): 720 for C-Cl str 1360 for –NO2 Str, 1520 for N=N str, 1630 for Carbonyl str, 2860 for C-H str, 3210 for Ar–O-H Str .1H-NMR ($CDCl_3$, 400 MHz) δ: 8.29 (s, 1H, OH), 7.42–7.46 (m, 4H, Ar–H), 7.35 (d, 2H, Ar–H, J = 8.4 Hz), 6.98(d, 2H, Ar–H, J = 8.4 Hz), 7.10–7.20 (m, 5H, Ar–H) ppm; 4.25(S1H) 5.80 (S, 2H) ppm. 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 45.2 (Aliphatic Alpha Carbon), 85.9 (Benzylic Carbon), 110.4(Ar Carbon), 122.3ppm,123.0 (Ar Carbon), 129.5(Ar Carbon),137.0(Ar Carbon),141.7(Ar Carbon), 150.8(Ar Carbon), 160.6(Ar-C), 206.7(Carbonyl), 116.4 (Ar Carbon),ppm;

(A_7): Yellow solid, 74%, 0.41 g. mp 219–221°C. FT-IR (KBr, cm−1): 825 For C-Br str, 1390 for –NO2 Str, 1530 for C=N Ar str, 1650 for Carbonyl str, 2960 for C-H str, 3230 for Ar–O-H Str **.** 1H-NMR ($CDCl_3$, 400 MHz) δ: 8.29 (s, 1H, OH), 7.43–7.46 (m, 4H, Ar–H), 7.35 (d, 2H, Ar–H, J = 8.4 Hz), 6.98 (d, 2H, Ar–H, J = 8.4 Hz), 6.81–6.88 (m, 5H, Ar–H) ppm; 4.80(S1H) 5.81 (S, 2H) ppm. 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 47.2 (Aliphatic Alpha Carbon), 88.9 (Benzylic Carbon), 113.4(Ar Carbon), 125.3ppm,127.0 (Ar Carbon), 131.5(Ar Carbon),139.0(Ar Carbon),146.7(Ar Carbon), 153.8(Ar Carbon), 163.6(Ar-C), 208.7(Carbonyl), 118(Ar Carbon),ppm;

(A_8): Yellow solid, 85%, 0.50 g. mp 208–210 °C. FT-IR (KBr, cm−1): 1275 for –NO2 Str, 740 fro C-Cl Str, 1390 for C=N Ar str, 1450 for C=C Ar Str, 1700 for C=O str, 2890 for C-H str, 3390 for Ar–O-H Str . 1H-NMR ($CDCl_3$, 400 MHz) δ: 8.29 (s, 1H, OH), 7.43–7.46 (m, 4H, Ar–H), 7.36 (d, 2H, Ar–H, J = 8.4 Hz), 6.99 (d, 2H, Ar–H, J = 8.4 Hz), 6.80–6.89 (m, 5H, Ar–H) ppm; 4.80(S1H) 5.82 (S, 2H) ppm. 13C-NMR ($CDCl_3$-d6, 100 MHz) δ: 57.2 (Aliphatic Alpha Carbon), 95.9 (Benzylic Carbon), 112.4(Ar Carbon), 125.3ppm,126.0 (Ar Carbon), 130.5(Ar Carbon),139.0(Ar Carbon),143.7(Ar Carbon), 155.8(Ar Carbon), 163.6(Ar-C), 215.8(Carbonyl), 118.4 (Ar Carbon),ppm;

RESULT AND DISCUSSION

In the present work eight thiazolidinone derivative condensed with azo moiety was synthesized in good yield. In this article we summarize newscaffolds for the synthesis of Thiazolidinonederivatives condensed with azo moiety with simple solvent and catalyst. It is likely that in the future, many more powerful and benign methodologieswill emerge in this field. Furthermore, attention should be paid to modificationand substitution in thiazolidine nuclei and also toward SAR and dockingstudies for improving the design strategies for drug targets. On a global scale, theopportunity in the field of thiazolidine is broad for synthetic and medical discovery,and this knowledge has been applied in the synthesis of multi-target hybrid drugs,which control diseases such as diabetes, cancer, multiple sclerosis and lupus. will serve as an update for researchers focused on the synthesisof thiazolidine derivatives condensed with azo moiety and will encourage further growth in this field. The present study may be useful in pharmaceutical industries and agriculture field.

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