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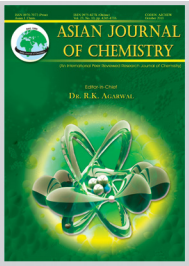

Design and Synthesis of 2(3*H*)-furanone Derivatives as γ-Aminobutyric Acid Receptor Modulator

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Eleven new 3-(substituted-arylidene)-5-(4-chloro-3-methyl-phenyl)-)-3*H*-furan-2-one were synthesized from appropriate 4-(substituted -phenyl)-4-oxo-butyric acid and isatin derivatives. Molecular docking calculations experiments were performed using EXHZ version 1.4 and Auto Dock 4.0 to identify potential γ-aminobutyric acid receptor modulator among all synthesized furanones. The anticonvulsant activities of selected compounds were evaluated in mice by PTZ induced seizure method. Result indicate that all tested compounds possess anti-convulsant activity after oral administration and that the compounds 1-acetyl-3-[5-(4-bromo-phenyl) -2-oxo -furan-3-ylidene]-1,3-dihydro-indol-2-one (C-6) and 1-acetyl-3-[5-(4-ethyl-phenyl)-2-oxo- furan-3-ylidene]-5-ethyl-1,3-dihydro-indol-2-one (C-8) possess significant anticonvulsant activity could be comparable to that of diazepam a standard.

Key Words: Furanones, Docking, γ-Aminobutyric acid, Anticonvulsant.

INTRODUCTION

Epilepsy is the most frequent neurologic affection characterized by excessive temporary neuronal discharge[1]. γ-Aminobutyric acid (GABA) is the major inhibitory neurotransmitter in the mammalian brain[2,3]. Fast synaptic inhibition in the fore brain is mediated mostly by the neutral aminoacid GABA intracting with post-synaptic GABAA receptors (GABARs). GABARs are heteromeric protein complexes composed of multiple subunit that form ligand-gated anion-selective channels that are modulated by barbiturates, benzodiazepines, ethanol, volatile anesthetics and the anesthetic steroids[4,5]. As GABA itself does not cross the blood-brain barrier, there is considerable interest in the development of systematically active GABA-mimetic agents[6]. The furanones ring system is also known as butyrolactone or butenolide, is a recognizable component of natural product[7]. The simpler butyrolactone, 3,3-diethylbutyrolactone, shows anticonvulsant activity[8]. While the furanones also reported to have cardiotonic[9,10], antiinflammatory[11,12], antimicrobial[13-15], antiviral[16] and anticancer activity[17], platelet inhibitory activity[18]. Recently synthesized compounds 4,5-diaryl-3-hydroxy-2(5*H*)-furanone also reported to exhibits good antioxidant activity[19].

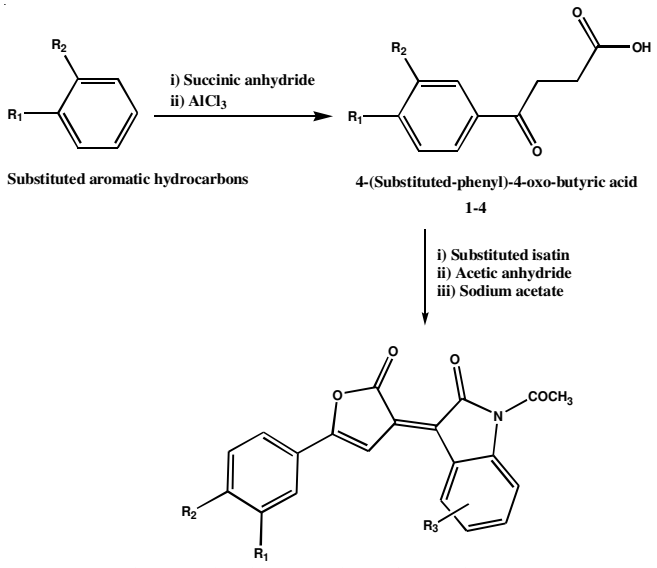
EXPERIMENTAL

The purity of synthesized compounds were ascertained by thin layer chromatography on silica gel G in TEF(5:4:1) solvent systems using iodine vapours and UV lamps for visualization of spots. Melting points of the compounds were determined in open glass capillaries using Kjeldahl flask containing liquid paraffin and are uncorrected. The elemental analysis was carried out on Vario-EL III CHNOS- Elemantar analyzer and the values were found within ± 0.4 % of the theoretical values.

The IR spectra of compounds were recorded in KBr on Perkin Elmer BX-II FTIR spectrophotometer. The proton magnetic resonance spectra (^{1}H NMR) were recorded on Bruker 300 MHz instrument in $CDCl_3$ using tetramethylsilane [$(CH_3)_4Si$] (TMS) as internal standard.

Synthesis of 4-(substituted-phenyl)-4-oxo-butyric acid (1-4): A mixture of succinic anhydride (0.1 mol; 10 g) and dry appropriate substituted benzene (50 mL) was taken in a round bottom flask. Anhydrous aluminium chloride (0.1125 mol; 15 g) was added at once and refluxed under anhydrous conditions for 2 h. After completion of reaction dilute hydrochloric acid (2.5 % v/v, 50 mL) was added slowly. Excess of solvent was removed by steam distillation and the hot solution was poured into beaker containing cold water (100 mL) when a solid mass separated out. It was purified by dissolving in sodium hydroxide solution (5 % w/v), filtered followed by addition of hydrochloric acid. The compound so obtained was filtered, washed with cold water, dried and crystallized from methanol.

General procedure for the synthesis of 1-acetyl- 3-[5-(4-fluoro-phenyl)-2-oxo-furan-3-ylidene]- 1,3-dihydro- (5-substituted-indol) -2-one (5-15): To a solution of 4-(substituted-phenyl)-4-oxo-butyric acid (1 mmol) and appropriate isatin (1 mmol) in acetic anhydride (10 mL) was added fused sodium acetate (0.03 mol) under Perkin condition. The reaction mixture was refluxed for 4 h. After completion of reaction the mixture was poured onto crushed ice. Reddish brown precipitate was obtained, which was separated out, filtered, washed, dried and recrystalized from acetic acid (**Scheme-I**).



Scheme-I

Molecular docking: To identify potential anticonvulsant lead compounds among all synthesized 2(3*H*)-furanones, docking calculations were performed using EXHZ Version 1.4 and Auto Dock 4.0 on Fedora Linux WS 3.0 into the 3D structure of the catalytic site of GABA receptor (pdb code: 3IP9). The conversion of available ligands to 3-dimensional format carried out with the help of Marvin view and binding site analysis carried out for the identification and visualization of possible binding sites and the distribution of surrounding residues in the active sites. As a result, 4 active sites have been predicted; it also gives residues in active site and center of active site with X, Y, Z coordinates of all the active sites.

Out of which, one active site has been selected (center of active site: 23.357, 19.489, 18.247), which is present closer to the ligand selected (ABU) from the chosen receptor.

Parameters have been chosen for docking of ligands with the chosen receptor (PDB Id: 3IP9). Gridcenter as: 23.357 19.489 18.247 (xyz-coordinates), npts 110 110 110 (num.grid points in xyz), spacing 0.375 (spacing-Å), dielectric constant -0.1465 (< 0, AD4 distance-dep.diel; > 0) and LGA algorithm (Lamarckian genetic algorithm).

Protein-Ligand Docking has been carried out: using 3IP9 as a receptor with bound ligand (ABU) and obtained results were evaluated in terms of binding energy.

Anticonvulsant evaluation: Male albino mice weighing between 25-30 g were used for grandmal type of epileptic condition. They were housed under standard laboratory conditions maintained at 25 ºC and humidity at 45-60 % for one week before anticonvulsant activity was carried out by using PTZ animal models. Food and water were withdrawn prior to the experiment. All the result was statistically analyzed and result expressed as mean ± SEM.

RESULTS AND DISCUSSION

The present studies report eleven new 2(3*H*)-furanones and their structure were established on the basis of spectral studies and elemental analysis. The substitutions on synthesized compounds and physical data are given in Table-1.

TABLE-1
PHYSICAL DATA AND SUBSTITUTION ON SYNTHESIZED COMPOUNDS

S. No.	Compound	R_1	R_2	R_3	m.f.	m.w.
1	C-5	-F	-H	-H	$C_{20}H_{12}NO_4F$	349.31
2	C-6	-Br	-H	-H	$C_{20}H_{12}NO_4Br$	410.22
3	C-7	- C_2H_5	-H	-H	$C_{22}H_{17}NO_4$	359.37
4	C-8	-Cl	-CH_3	-H	$C_{21}H_{14}NO_4Cl$	379.79
5	C-9	-F	-H	-CH_3	$C_{21}H_{14}NO_4F$	363.34
6	C-10	-Br	-H	-CH_3	$C_{21}H_{14}NO_4Br$	424.24
7	C-11	-C_2H_5	-H	-CH_3	$C_{23}H_{19}NO_4$	373.40
8	C-12	-Cl	-CH_3	-CH_3	$C_{22}H_{16}NO_4Cl$	393.82
9	C-13	-F	-H	-Br	$C_{20}H_{11}NO_4BrF$	428.21
10	C-14	-C_2H_5	-H	-Br	$C_{22}H_{16}NO_4Br$	438.27
11	C-15	-Cl	-CH_3	-Br	$C_{21}H_{13}NO_4BrCl$	458.69

4-(4-Chloro-3-methyl-phenyl)-4-oxo-butyric acid (1) White cream coloured flakes, m.p. 96-98 ºC, R_f: 0.78, yield: 67 %, IR (KBr, ν_{max}, cm^{-1}) : 3367(O-H), 2932(C-H), 1726 (C=O), 1679 (ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.30 (s,3H,-CH_3), 2.80, 2.52, (t, each, 4H, 2 × CH_2), 6.90, 7.31 (m, each, 2H, Ar-H), 7.61 (s,1H, Ar-H), 10.61 (s, 1H, -OH), m.f.: $C_{11}H_{11}O_3Cl$.

4-(4-Fluoro-phenyl)-4-oxo-butyric acid (2): Cream coloured flakes, m.p.: 102-104 ºC, R_f: 0.80, Yield: 64 %, IR (KBr, ν_{max}, cm^{-1}): 3378(O-H), 2934(C-H), 1735 (C=O), 1671 (ArC = C), ^1H NMR ($CDCl_3$): δ, ppm 2.80, 3.26 (t, each, 4H, 2 × CH_2), 7.28, 7.61(d, each, 4H, Ar-H), 10.61(s, 1H,-OH) m. f.: $C_{10}H_9O_3F$

4-(4-Bromo-phenyl)-4-oxo-butyric acid (3): Cream coloured flakes, m.p.: 150-152 ºC, R_f: 0.74, Yield: 67 %, IR (KBr, ν_{max}, cm^{-1}): 3400(O-H), 2933(C-H), 1733(C=O), 1661(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.83, 3.20 (t, each, 4H, 2 × CH_2), 7.50, 7.69 (d, each, 4H, Ar-H), 10.71(s, 1H, -OH), m.f.: $C_{10}H_9O_3Br$.

4-(4-Ethyl-phenyl)-4-oxo-butyric acid (4): White coloured flakes m.p.: 112-114 ºC, R_f: 0.70, yield: 67 % IR (KBr, ν_{max}, cm^{-1}): 3382(O-H), 2935(C-H), 1729(C=O), 1663(ArC = C), ^1H NMR ($CDCl_3$): δ, ppm 1.26 (t, 3H, -CH_3), 2.65 (q, 2H, -CH_2-) 2.83, 3.28 (t, each, 4H, 2 × CH_2), 7.37, 7.89 (d, each, 4H, Ar-H), 10.68 (s, 1H, -OH), m.f.: $C_{12}H_{14}O_3$

1-Acetyl-3-[5-(4-fluoro-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro-indol-2-one (5): Reddish brown solid, m.p.: 152-155 ºC, R_f: 0.80, Yield: 63 %, IR (KBr, ν_{max}, cm^{-1}): 2958(C-H), 1721(C=O), 1597(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm, 2.40(s, 3H, -$COCH_3$), 6.71(s, 1H, furanone ring), 6.89,

7.49(m, 2H, Ar-H), 7.03, 7.32, 7.63, (d, each, 6H, Ar-H), calcd. for $C_{20}H_{12}NO_4F$: C, 68.77; H, 3.46; N, 4.01, found: C, 68.74; H, 3.42; N, 4.00

1-Acetyl-3-[5-(4-bromo-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro-indol-2-one (6): Reddish brown solid, m.p: 163-166 ºC, R_f: 0.76, Yield: 64 %, IR (KBr, ν_{max}, cm^{-1}): 2960 (C-H), 1760 (C=O), 1612 (ArC=C), ^1H NMR ($CDCl_3$): δ, ppm, 2.45(s, 3H, -$COCH_3$), 6.80(s, 1H, furanone ring), 6.42, 7.23(d, each, 4H, Ar-H), 7.00, 7.10(m, 2H, Ar-H), 7.33, 7.62(d, 2H, Ar-H), calcd for $C_{20}H_{12}NO_4Br$: C, 58.56; H, 2.95; N, 3.41, found: C, 58.54; H, 2.92; N, 3.40.

1-Acetyl-3-[5-(4-ethyl-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro-indol-2-one (7): Reddish brown solid, m.p.: 143-146 ºC, R_f: 0.70, yield: 55 %, IR (KBr, ν_{max}, cm^{-1}): 2946(C-H), 1762(C=O), 1610(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 1.27(t, 3H, -CH_3), 1.94(s, 3H, -$COCH_3$), 2.70(m, 2H, -CH_2), 6.74(s, 1H, furanone ring), 6.92, 7.23(d, each, 4H, Ar-H), 7.02, 7.14(m, 2H, Ar-H), 7.33, 7.62(d, each, 2H, Ar-H), calcd for $C_{22}H_{17}NO_4$: C, 73.53; H, 4.77; N, 3.90, found: C, 73.50; H, 4.72; N, 3.88.

1-Acetyl-3-[5-(4-chloro-3-methyl-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro -indol-2-one (8): Brown solid, m.p: 135-137ºC, R_f: 0.78, yield: 62 %, IR (KBr, ν_{max}, cm^{-1}): 2951(C-H), 1756(C=O), 1610(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.36(s, 3H, -CH_3), 2.44(s, 3H, -$COCH_3$), 6.76(s, 1H, furanone ring), 6.97, 7.10(m, 2H, Ar-H), 7.08,(s, 1H, Ar-H), 7.31, 7.62(d, each, 2H, Ar-H), 7.27(m, 2H, Ar-H).

1-Acetyl-3-[5-(4-fluoro-phenyl)-2-oxo-furan-3-ylidene]-5-methyl-1,3-dihydro-indol-2-one (9): Brown crystals, m.p.: 141-145 ºC, R_f: 0.77, Yield: 65 %, IR (KBr, ν_{max}, cm^{-1}): 2954(C-H), 1776(C=O), 1600(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.36(s, 3H, -CH_3), 2.41(s, 3H, -$COCH_3$), 6.78(s, 1H, furanone ring), 6.90, 7.20(d, each, 4H, Ar-H), 7.08,(s, 1H, Ar-H), 7.18, 7.52(m, 2H, Ar-H).

1-Acetyl-3-[5-(4-bromo-phenyl)-2-oxo-furan-3-ylidene]-5-methyl-1,3-dihydro- indol-2-one (10): Brown crystals, m.p.: 163-166 ºC, R_f: 0.75, Yield: 57 %, IR (KBr, ν_{max}, cm^{-1}) : 2950(C-H), 1776(C=O), 1600(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.36(s, 3H, -CH_3), 2.41(s, 3H, -$COCH_3$), 6.78(s, 1H, furanone ring), 7.08,(s, 1H, Ar-H), 7.22, 7.41(d, each, 4H, Ar-H), 7.14, 7.51(d, each, 2H, Ar-H).

1-Acetyl-3-[5-(4-ethyl-phenyl)-2-oxo-furan-3-ylidene]-5-ethyl-1,3-dihydro-indol-2-one (11): Reddish brown crystals, m.p.: 143-146 ºC, R_f: 0.71, Yield: 61 %, IR (KBr, ν_{max}, cm^{-1}): 2956(C-H), 1760(C=O), 1600(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 1.27(t, 3H, -CH_3), 1.98(s, 3H, -$COCH_3$), 2.70(m, 2H, -CH_2), 6.74(s, 1H, furanone ring), 6.92, 7.23 (d, each, 4H, Ar-H), 7.06,(s, 1H, Ar-H), 7.29, 7.62 (d, each, 2H, Ar-H).

1-Acetyl-3-[5-(4-chloro-3-methyl-phenyl)-2-oxo-furan-3-ylidene]-5-methyl-1,3 -dihydro-indol-2-one (12): Reddish brown crystals, m.p.: 135-137 ºC, R_f: 0.73, Yield: 71 %, IR (KBr, ν_{max}, cm^{-1}): 2961(C-H), 1770(C=O), 1597(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 2.27(s, 6H, -CH_3), 2.44(s, 3H, -$COCH_3$), 6.80(s, 1H, furanone ring), 6.99, 7.13(d, each, 2H, Ar-H), 7.06,(s, 1H, Ar-H), 7.09(s,1H, Ar-H), 7.29, 7.62(d, each, 2H, Ar-H).

1-Acetyl-5-bromo-3-[5-(4-fluoro-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro- indol-2-one (13): Brown solid, m.p.: 169-171 ºC, R_f: 0.79, Yield: 59 %, IR (KBr, ν_{max}, cm^{-1}): 2959 (C-H), 1760 (C=O), 1610 (ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 1.98(s, 3H, -COCH3), 6.80(s, 1H, furanone ring), 6.92, 7.23 (d, each, 4H, Ar-H), 7.36,(s, 1H, Ar-H), 7.29, 7.62(d, each, 2H, Ar-H).

1-Acetyl-5-bromo-3-[5-(4-ethyl-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro-indol-2-one (14): Brown solid, m.p.: 129-132 ºC, R_f: 0.72, Yield: 59 %, IR (KBr, ν_{max}, cm^{-1}): 2952 (C-H), 1762(C=O), 1600(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 1.27(t, 3H, -CH_3), 1.94(s, 3H, -$COCH_3$), 2.70(m, 2H, -CH_2), 6.76(s, 1H, furanone ring), 6.97, 7.23(d, each, 4H, Ar-H), 7.32, 7.64(d, each, 2H, Ar-H), 7.48(s, 2H, Ar-H).

1-Acetyl-5-bromo-3-[5-(4-chloro-3-methyl-phenyl)-2-oxo-furan-3-ylidene]-1,3-dihydro-indol-2-one (15): Greenish brown solid, m.p.: 155-157 ºC, Rf: 0.76, Yield: 63 %, IR (KBr, ν_{max}, cm^{-1}): 2953(C-H), 1770(C=O), 1610(ArC=C), ^1H NMR ($CDCl_3$): δ, ppm 1.98(s, 3H, -$COCH_3$), 2.35(s, 3H, -CH_3) 6.80(s, 1H, furanone ring), 6.98, 7.13(d, each, 2H, Ar-H), 7.08,(s, 1H, Ar-H), 7.29, 7.62(m, each, 2H, Ar-H), 7.49 (s,1H, Ar-H).

Docking result: Best ten compounds were selected on the basis of least mean binding energies. Among the all synthesized compounds the c-6 (cyan), was found in best conformation with least mean binding energy -8.26, other compounds c-11(magenta), c-8(orange) and c-12(red) were also found comparable in their conformation with least mean binding energies -8.14, -7.69 and -7.60 respectively (Fig.-1).

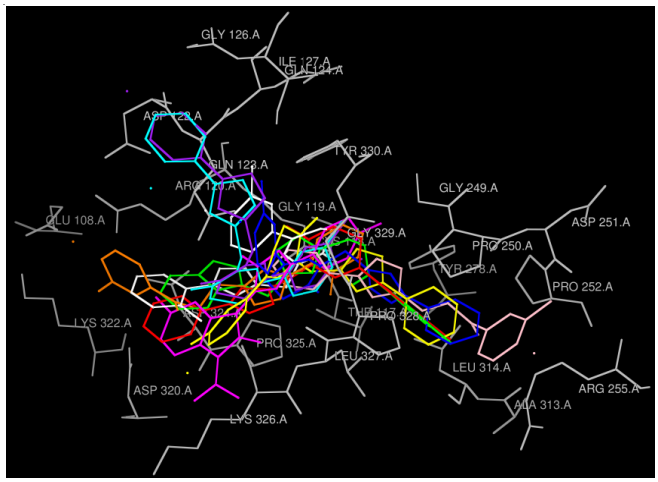


Fig. 1. 3IP9 receptor with atoms interacting with individual best ten compounds

Anticonvulsant result: It was observed that the test agents decreases the pentylene tetrazole induce convulsion. Moreover the effect of test drugs c-6 and c-11 were found to be comparable to that of diazepam as standard drug. The test drugs could suppress the onset and duration of the chronic seizure in PTZ model. As we could observe that the test agent's c-6, reduced the convulsion rate and percentage of mortality protection. The chronic seizure is induced by γ-aminobutyric acid (GABA) transmission blocker PTZ and may be by decreased GABA-ergic action of the test drugs (Table-2).

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TABLE-2
EFFECT OF TEST DRUGS AND DIAZEPAM ON PENTYLENETETRAZOLE (PTZ) INDUCE CONVULSION

	Control	c-6	c-8	c-11	c-12	Diazepam (8 mg/kg)
Onset of convulsion	51.83	214.15	172.48	201.52	98.21	390.42
(sec)	47.22	189.15	165.31	178.42	151.72	430.27
	62.45	193.56	142.75	186.92	138.12	-
	55.60	217.22	201.88	201.42	107.58	-
	78.23	202.78	131.52	145.48	103.11	389.42
No. of convulsion	25	10	19	17	20	02
	21	16	20	14	19	01
	20	13	18	16	17	00
	21	12	15	16	18	00
	27	11	17	18	20	03
Mean ± S.E.	22.8 ± 1.35	12.4 ± 2.30	17.8 ± 1.94	16.2 ± 1.66	18.8 ± 1.5	1.2 ± 1.33
P value	-	< 0.01	< 0.05	< 0.05	< 0.05	< 0.01
% of mortality protection	-	60	20	40	20	100

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