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SYNTHESIS, ANTIMICROBIAL AND ANTIFUNGAL STUDY OF 2-(5–ARYL-4,5–DIHYDRO-1-SUBSTITUTED-PYRAZOL-3-YL)-SUBSTITUTED NAPHTHALENE-1-OL

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ABSTRACT

The present studies are a part of the continuing interest on the synthesis of new dihydropyrazole derivatives. dihydropyrazole derivatives shows anti-inflammatory and analgesics properties. In view of various pharmological and biological activities of dihydropyrazole derivatives it was proposed to synthesize and microbial screening of titled dihydropyrazole derivatives. The titled dihydropyrazole derivatives were obtained by treating 1-(substituted-1-hydroxynaphthalen-2-yl)-3-aryl-prop-2-en-1-one with semicarbazide in DMF solvent. The synthesized compounds are characterized by elemental analysis, ^{1}H NMR, IR spectroscopy. Newly synthesized compound shows an excellent antimicrobial and antifungal activities.

Key words: Synthesis, Dihydropyrazole, Antimicrobial and antifungal study.

INTRODUCTION

Dihydropyrazoles have played a crucial part in the development of heterocyclic chemistry The literature survey reveals that dihydropyrazole derivatives have been studied extensively because of their ready accessibility, diverse chemical reactivity, broad spectrum of biological activity[1-4] and variety of industrial applications[5-6]. Dihydropyrazoles with sulphonamidoaryl substituent at 3-position show cerebroprotective[7], antidepressant activity[8], anti-implantation activity[9], hypoglycemic activity[10]. Due to this vital biological roll of dihydropyrazole derivatives, it was planned to synthesize titled dihydropyrazole derivatives. Thus we present herein the synthesis of the titled compounds.

It has been observed that substituted chalcones are the best starting compounds for the preparation of derivatives of dihydropyrazole. Present work deals with the synthesis and

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biological study of some new dihydropyrazoles and their characterization by spectral analysis (IR, ^{1}H NMR).

EXPERIMENTAL

All the melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer ^{1}H NMR spectra were recorded on a Brucker AC300 FNMR spectrometer (300 MHz), using TMS as an internal standard. Microanalysis of nitrogen was obtained by Kjeldahal's method. Thin layer chromatography on silica gel-G, was used to check the purity of the compounds.

Procedure for the synthesis of 2-acetyl-substituted-1-naphthol (2-3)

In hot glacial acetic acid, fused $ZnCl_2$ was added and refluxed till dissolved, then powdered substituted 1-naphthol was added and the mixture was refluxed for about 8 hours then cooled and poured in acidulated water. The solid obtained was filtered, washed, dried and recrystallized from rectified spirit to obtain compound (2-3). Physical data of the compounds are given in Table 1.

2: IR (KBr) cm^{-1}: 1650 (C=O), 3412 (-OH)

NMR ($CDCl_3$ + DMSO-d6) : δ 2.35 (s, 3H, CH_3), δ 7.11-6.88 (m, 6H, Ar-H), δ 9.83 (s, 1H, -OH).

Synthesis of 1-(substituted-1-hydroxynaphthalen-2-yl)-3-aryl-prop-2-en-1-one (3-14)

2-acetyl-substituted-1-naphthol and aromatic aldehydes were added in ethanol solvent. To this mixture KOH (10%) solution was added dropwise with constant stirring. The reaction mixture was kept overnight. Then the mixture was poured over crushed ice and little HCl. The product was filtered and recrystallized from ethanol to obtain the compounds (3-14). The physical data is given in Table 1.

3: IR (KBr) (cm^{-1}): 1650 (C=O), 3412 (-OH), 1520 (-NO_2)

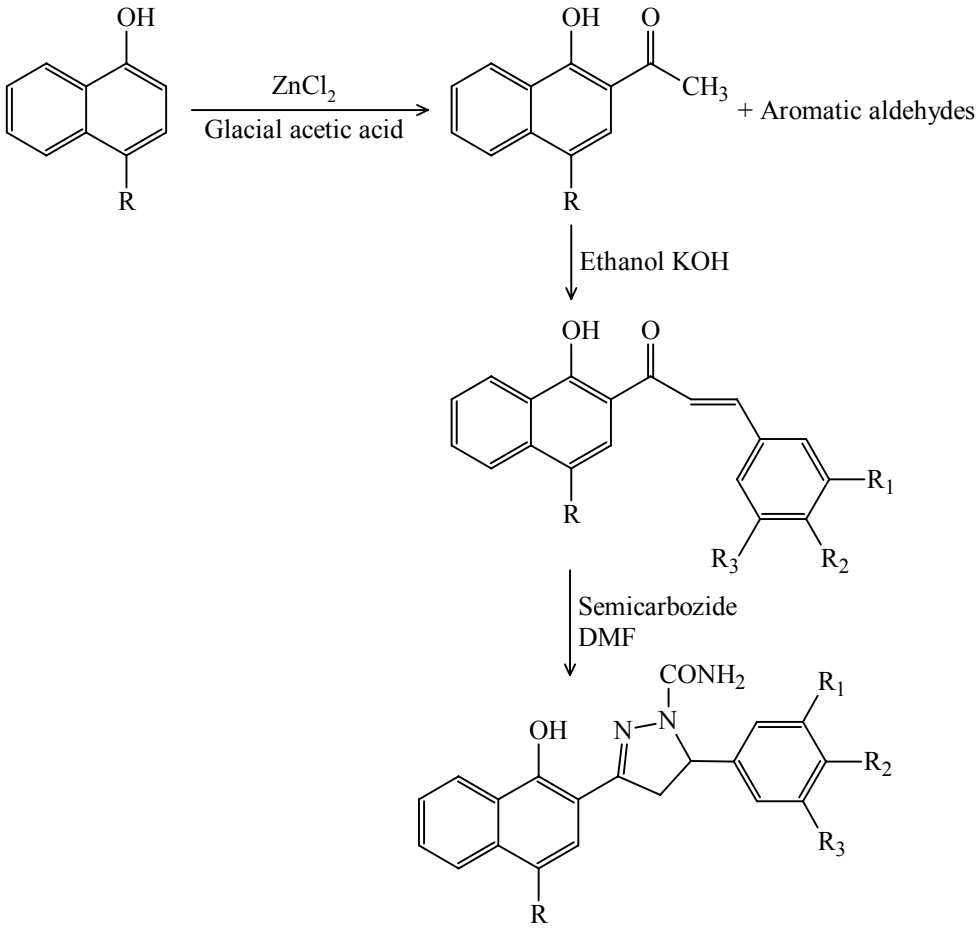
NMR ($CDCl_3$ + DMSO-d6): δ 6.98-7.46 (m, 10H, Ar-H), δ 8.059 (d, 1H =CH), δ 8.111 (d, 1H =CH) δ 9.55 (s, 1H, -OH).

14: IR (KBr) cm^{-1}: 1655 (C=O), 3417 (-OH).

NMR ($CDCl_3$ + DMSO-d6) : δ 7.12-7.32 (m, 9H, Ar-H), δ 8.054 (d, 1H =CH), δ 8.096 (d, 1H =CH) δ 9.55 (s, 1H, -OH).

Table 1: Physical and analytical characterization data of newly synthesized compounds

Compd.	R	R_1	R_2	R_3	Melting point (^{0}C)	% Yield	% Nitrogen		R_f value
							Found	Calculated	
1	H	--	--	--	98	62	--	--	--
2	Cl	--	--	--	105	67	--	--	--
3	H	H	NO_2	H	154	58	--	--	--
4	H	NO_2	H	H	158	52	--	--	--
5	H	H	H	NO_2	145	51	--	--	--
6	H	H	F	H	123	40	--	--	--
7	H	Cl	H	H	106	50	--	--	--
8	H	H	H	Cl	109	58	--	--	--
9	Cl	H	NO_2	H	131	60	--	--	--
10	Cl	NO_2	H	H	138	68	--	--	--
11	Cl	H	H	NO_2	125	62	--	--	--
12	Cl	H	F	H	101	71	--	--	--
13	Cl	Cl	H	H	117	56	--	--	--
14	Cl	H	H	Cl	140	55	--	--	--
15	H	H	NO_2	H	265	40	14.88	14.89	0.68
16	H	NO_2	H	H	287	38	14.86	14.89	0.62
17	H	H	H	NO_2	270	32	14.82	14.89	0.58
18	H	H	F	H	253	37	12.31	12.38	0.54
19	H	Cl	H	H	250	35	11.41	11.49	0.61
20	H	H	H	Cl	245	37	11.43	11.49	0.63
21	Cl	H	NO_2	H	241	39	13.62	13.64	0.56
22	Cl	NO_2	H	H	248	41	13.60	13.64	0.68
23	Cl	H	H	NO_2	238	42	13.59	13.64	0.63
24	Cl	H	F	H	287	35	11.20	11.24	0.58
25	Cl	Cl	H	H	298	33	10.47	10.50	0.64
26	Cl	H	H	Cl	268	44	10.43	10.50	0.57



Where R = H, Cl; R_1 = H, Cl, NO_2; R_2 = H, F, NO_2; R_3 = H, Cl, NO_2

Synthesis of 2-(5-aryl-4,5-dihydro-1-substituted-pyrazol-3-yl)-substituted naphthalene-1-ol. (15-26)

1-(substituted-1-hydroxynaphthalen-2-yl)-3-aryl-prop-2-en-1-one and semicarbazide were added to DMF and refluxed for 2 hours. The cooled reaction mixture was diluted with water and the semisolid so obtained was triturated with ethanol to get a solid, which was recrystallised from ethanol-acetic acid mixture to get titled dihydropyrazole derivatives in 32-44% yield. The physical data is given in Table 1.

15: IR (KBr) cm^{-1}: 3200 (NH_2), 3402 (-OH), 1515 (-NO_2).

NMR ($CDCl_3$ + DMSO-d6): δ 6.55 (s, 2H, -NH_2), δ 6.95-7.17 (m, 10H, Ar-H), δ 8.051 (d, 1H =CH) , δ 8.091 (d, 1H =CH) δ 9.62 (s, 1H, -OH).

20: IR (KBr) cm^{-1}: 3210 (NH_2), 3397 (-OH).

NMR ($CDCl_3$ + DMSO-d6) : δ 6.49 (s, 2H, -NH_2), δ 6.67-7.14 (m, 10H, Ar-H), δ 8.047 (d, 1H =CH), δ 8.086 (d, 1H =CH) δ 9.57 (s, 1H, -OH).

Antimicrobial studies

All above dihydropyrazoles have been studied for their antimicrobial activity against *Escherichia coli*, *Proteus mirabilis*, *Staphylococcus aureas* and *Pseudomonas aeruginosa*. The culture of each species was incubated at 37°C and the zone of inhibition was measured after 24 hr. Most of these compounds were found active.

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