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SYNTHESIS AND ANTIMICROBIAL ACTIVITY OF SOME CHALCONES AND FLAVONES HAVING 2-HYDROXY ACETOPHENONE MOIETY.

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ABSTRACT

Chalcones (3a-e) on reaction with catalytic amount of I_2 in DMSO gives Flavones (4a-e) in high yield. The corresponding chalcones (3a-e) were obtained by Claisen-Schimdt condensation of aromatic aldehydes with o-hydroxy acetophenone. The flavones obtained were further studied for antifungal activity.

KEYWORDS : 2-Hydroxy acetophenone, 2-Hydroxy chalcones, Flavones, Antifungal and Antibacterial activity.

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INTRODUCTION

The significance of chalcones as an important starting material for the synthesis of heterocyclic compounds and their wide range of applications are well known. Flava means yellow in Greek and the collective name of flavonoids for this group of compounds was proposed by Geissman[1-5] in 1952. This is a very large group of compounds showing extraordinary diversity and variation and as the Greek root for the group suggests, as many of these compounds are yellow in colour.

Anthony C. Dweck[6], et al studied the internal and external use of medicinal plantscontaining flavones.

Lei zou, et al[7] studied Synthesis of 4'-Methoxy Flavone. The flavones are obtained mainly from nature, secondly by biological synthesis. The chemical synthesis is carried out mostly by cyclisation and condensation of o-hydroxyacetophenone[8-10], or by dehydrogenation of flavanones[11-13]. Here, we report a new synthetic method of flavones by the reaction of enamine and heptanedioyl chloride. Acylation of 2 mole enamine with 1 mole chloride of dicarboxylic acid has been used to prepare bis-(1,3-diketone) compound[14], but we discovered that 4'-methoxy-5,6,7,8-tetrahydroflavone prepared by reaction of 4-methoxy acetophenone enamine with heptanedioyl chloride in 80% can be easily transformed to 4'-methoxy flavone.

In the Baker-Venkataraman process,[15] 2-hydroxyl acetophenones are converted into benzoylesters, which are rearranged with bases to form 1,3 diphenylpropane- 1,3-diones, followed by cyclization with sodium acetate or sulfuric acid in acetic acid, I2-DMSO,[16] and CoIII(salpr)(OH)[17] to yield flavones in three steps.

An intramolecular Wittig reaction[18] of 2- acetoxyphenacyl bromides and benzoyl chlorides also gives flavones, a four step process from 2'-hydroxyacetophenones.

Jae In Lee, et al[19] studied An Efficient Synthesis of Flavones from 2-Hydroxy benzoic Acids. Flavones can be efficiently synthesized in two steps *via* 1-(2-hydroxyphenyl)-3-phenylpropane-1,3-diones from 2'-hydroxyacetophenones in high yields.

S.S.Mokle, et al[20]studied synthesis and antibacterial activity of some new chalcones and flavones having 2-chloro-8-methoxyquinolinyl moiety.

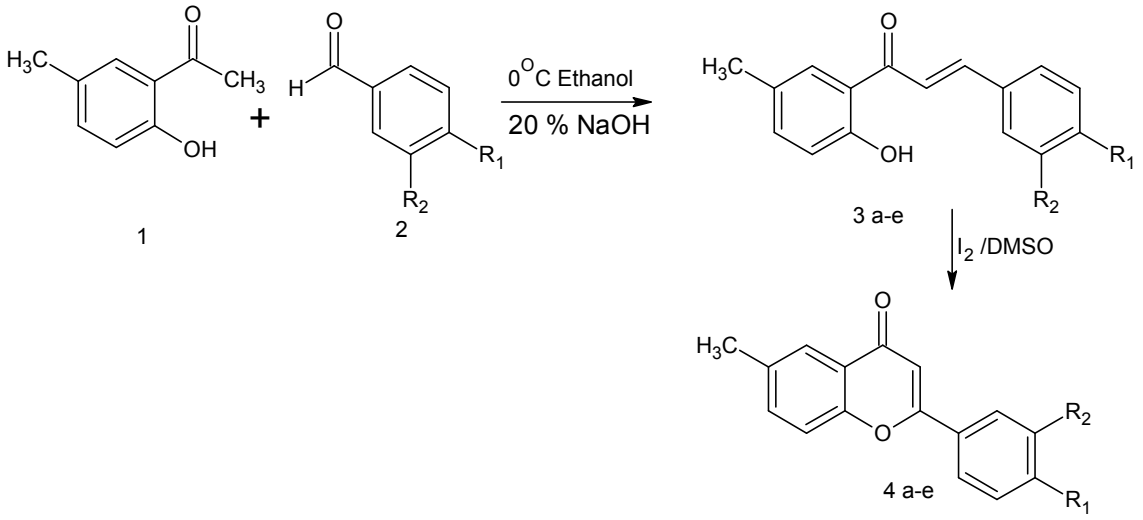
Chalcones of plant origin are known[21].Chalcones present great interest as compounds exhibiting antimalaria[22], antibactrial[23], antifibrogenic[24], anticancer[25], antitrichomonal[26], antiinflammatory[27], antileishmanial[28], cytotoxic and antitrypanosoma cruzi[29] activities. While the flavonoids compounds are a group of natural products found in fruits, vegetables, nuts, seeds and flowers as well as in teas and are important constituent of human diet. They have been demonstrated to possess antioxdidant[30], antihypertensive[31], antiallergic[32], Antinocicepative[33], trypsin inhibitors[34], plant growth regulator[35], antibacterial and Antifungal [36-37] activities.

MATERIALS AND METHODS

^{1}H NMR Spectra were recorded on a varian-NMR –mercury 300 in $CDCl_3$. The IR Spectra were recorded on a shimadzu FTIR Presige-21.

General procedure for the preparation of chalcones (3a-e)

To a well suspension of powdered NaOH in ethanol (1:10) at 0^{o}C were added 2-hydroxy-5-methyl acetophenone (1.5g,1 mole) and aromatic aldehydes (1 mole) in 1:2 proportion. The reaction mixture become deep red in colour after 0.5 hour was stirred further for 3 hour. After it was pour over ice and neutralised with dilute HCl and ethanol is added for crystallisation.



Scheme 1

(E)-1-(2-Hydroxy-5-methylphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one.
Yellow solid; m.p. =74^{0}C; yield= 74% ; Elemental analysis: **C** 74.11/73.89, **O** 17.91/19.03, **H** 5.97/6.08; IR (KBr): 1691.63(C=O stret.), 1639.55 (C=C stret.), 3738.17(-OH stret.), 2933.83(C-H(CH_3) stret.), 2999.41 (-OCH_3 stret.); ^{1}H NMR: δ 2.3 (s,3H,Ar-C**H**$_3$), δ 3.7 (s,3H,Ar-O-C**H**$_3$), δ 12.79 (s,1H,Ar-O**H**), δ 6.91 (d,1H,-CO-CH=C**H**-(Ph)-CH_3), δ 6.97 (d,1H,-CO-C**H**=CH-),δ 7.47(M,7h,Ar-**H**).

(E)-1-(2-Hydroxy-5-methylphenyl)-3-phenylprop-2-en-1-one.
Yellow solid; m.p. =104^{0}C; yield= 72% ; Elemental analysis: **C** 80.67/82.58, **O** 13.44/12.4, **H** 5.88/4.11; IR (KBr): 1768.78(C=O stret.), 1448.59 (C=C stret.), 3728.53(-OH stret.), 2916.48(C-H(CH_3) stret.), 806.27 (-OCH_3 stret.); ^{1}H NMR: δ 2.28 (s,3H,Ar-C**H**$_3$)δ 12.62 (s,1H,Ar-O**H(**H-bonded)),δ 7.28 (d,1H,-CO-CH=C**H**-Ph),δ 7.31 (d,1H,-CO-C**H**=CH-),δ 6.9- 7.92 (M,8h,Ar-**H**).

(E)-1-(2-Hydroxy-5-methylphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one.
Red solid; m.p. =117^{0}C; yield= 69% ; Elemental analysis: **C** 75.59/75.29, **O** 18.89/16.46, **H** 5.51/7.25; IR (KBr): 1510.31(C=O stret.), 1448.59 (C=C stret.), 3440 (-OH stret.), 2910 (C-H(CH_3) stret.), 806.27 (-OCH_3 stret.); ^{1}H NMR: δ 2.34 (s,3H,Ar-C**H**$_3$), δ 6.84 (d,1H,-CO-CH=C**H**-#-OH), δ 6.9 (d,1H,-CO-C**H**=CH-), δ 7.4 (m,7H,Ar-**H**), δ 12.1 (s,1H,Ar-O**H(**H-bonded**)**), δ 2.61 (s,1H,Ar-OH(without H- bonding).

(E)-1-(2-Hydroxy-5-methylphenyl)-3-(3-nitrophenyl)prop-2-en-1-one.:
Brown solid; mp=130^{0}C; yield= 71% ; Elemental analysis: **C** 69.84/68.7, **O** 22.61/23.02, **H** 4.59/4.11,**N** 4.94/4.89; IR (KBr): 1690 (C=O stret.), 1452.45 (C=C stret.), 3469 (-OH stret.), 2942 (C-H(CH_3) stret.), 1382 (-NO_2 stret.); ^{1}H NMR: δ 2.33 (s,3H,Ar-C**H**$_3$), δ 12.1 (s,1H,Ar-O**H**), δ 6.93(d,1H,-CO-C**H**=CH-), δ 6.89 (d,1H,-CO-CH=C**H**-Ph), δ 8.25 (m,7H,Ar-**H**).

(E)-3-(4-Chlorophenyl)-1-(2-hydroxy-5-methylphenyl)prop-2-en-1-one.:
Yellow solid; mp=134^{0}C; yield= 76% ; Elemental analysis: **C** 70.58/71.98, **O** 11.78/11.37, **H** 4.77/5.48,**Cl** 12.86/11.15; IR (KBr): 1648 (C=O stret.), 1489.10 (C=C stret.), 3440 (-OH stret.), 2880 (C-H(CH_3) stret.), 775 (-C-Cl stret.); ^{1}H NMR: δ 2.29 (s,3H,Ar-C**H**$_3$), δ 12.49 (s,1H,Ar-O**H(**H-bonded**)**), δ 7.26-8.14 (m,7H,Ar-**H**), δ 6.96 (d,1H,-CO-C**H**=CH-), δ 6.94 (d,1H,-CO-CH=C**H**-Ar).

General procedure for the preparation of flavones (4a-e):

Chalcones 3a-e (1 m mole) on reaction with catalytic amount of I_2 (0.1 m mole) in DMSO on refluxing for 20-40 minute, yields flavones (4a-e) after dilution with excess of cold water was filtered, washed with 20% Sodium thiosulphate solution to remove the colour of Iodine.

Table 1
Synthesis of 2-hydroxy chalcones and flavones

Entry	R_1	R_2	Yield %	Elemental Analysis				
				C	O	H	N	Cl
3a	OCH_3	-H	74	76.11/73.89	17.91/19.03	5.87/6.08	--	-
4a	OCH_3	-H	63	76.69/77.11	18.04/19.63	5.26/3.19	-	-
3b	-H	-H	72	80.67/82.58	13.44/12.4	5.88/4.11	-	-
4b	-H	-H	62	81.35/79.87	13.55/14.04	5.08/6.06	-	-
3c	-OH	-H	69	75.59/75.29	18.89/16.46	5.51/7.25	-	-
4c	-OH	-H-	60	76.19/78.02	19.04/18.78	4.76/3.19	-	-
3d	-H	-NO_2	71	69.84/68.7	22.61/23.02	4.59/4.11	4.94/4.89	-
4d	-H	-NO_2	63	68.32/68.13	22.77/21.01	3.91/4.09	4.98/4.76	-
3e	-Cl	-H	76	70.58/71.98	11.78/11.37	4.77/5.48	-	12.86/11.15
4e	-Cl	-H	65	71.11/70.69	11.85/11.19	4.07/5.03	-	12.86/13.08

2-(4-Methoxyphenyl)-6-methyl-4H-chromen-4-one

Yellow solid; mp=67^{0}C; yield= 63% ; Elemental analysis: **C** 76.69/77.11, **O** 18.04/19.69, **H** 5.26/3.19; IR (KBr): 1640 (C=O stret.), 1610 (C=C stret.), 2865(C-H(CH_3) stret.),2980 (-OCH_3 stret.), 1119 (C-O-C stret.); ^{1}H NMR: δ 2.35 (s,3H,Ar-C**H**$_3$), δ 3.9 (s,3H,Ar-O-C**H**$_3$), δ 6.95 (s,1H,-CO-C**H**=C<), δ 6.94-7.92 (m,7H,Ar-**H**).

6-Methyl-2-phenyl-4H-chromen-4-one

Yellow solid; mp=95^{0}C; yield= 62% ; Elemental analysis: **C** 81.35/79.87, **O** 13.55/14.04, **H** 5.08/6.06; IR (KBr): 1687 (C=O stret.), 1566.25 (C=C stret.), 2916.47(-C-H(CH_3)stret.), 785.05 (mono substituted benzene), 11.32 (C-O-C stret.); ^{1}H NMR: δ 2.3 (s,3H,Ar-C**H**$_3$), δ 6.80 (s,1H,-CO-C**H**=C<), δ 6.85-8.00 (m,8H,Ar-**H**).

2-(4-Hydroxyphenyl)-6-methyl-4H-chromen-4-one

Brown solid; mp=48^{0}C; yield= 60% ; Elemental analysis: **C** 76.19/78.02, **O** 19.04/18.78, **H** 4.76/3.19; IR (KBr): 1719 (C=O stret.), 1642 (C=C stret.), 2860 (-C-H(CH_3)stret.), 1118.75 (C-O-C stret.); ^{1}H NMR: δ 2.3 (s,3H,Ar-C**H**$_3$), δ 1.6 (s,1H,Ar-OH), δ 7.32 (m,7H,Ar-**H**), δ 6.9 (s,1H,-CO-C**H**=C<).

6-Methyl-2-(3-nitrophenyl)-4H-chromen-4-one

Black solid; mp=92^{0}C; yield= 63% ; Elemental analysis: **C** 68.32/68.13, **O** 22.77/21.01, **H** 3.91/4.09; **N** 4.98/4.76; IR (KBr): 1690 (C=O stret.), 1643 (C=C stret.), 2864 (-C-H(CH_3)stret.), 1182.40 (C-O-C stret.); 1529.60 (-NO_2 stret.) ^{1}H NMR: δ 2.6 (s,3H,Ar-C**H**$_3$), δ 7.6 (m,7H,Ar-**H**), δ 6.22 (s,1H,-CO-C**H**=C<).

2-(4-Chlorophenyl)-6-methyl-4H-chromen-4-one

Yellow solid; mp=128^{0}C; yield= 65% ; Elemental analysis: **C** 71.11/70.69, **O** 11.85/11.19, **H** 4.07/5.03; **Cl** 12.86/13.08 IR (KBr): 1678 (C=O stret.), 1642 (C=C stret.), 2880 (-C-H(CH_3)stret.), 1138.64 (C-O-C stret.), 790 (-C-Cl stret.); ^{1}H NMR: δ 2.6 (s,3H,Ar-C**H**$_3$), δ 6.96 (m,7H,Ar-**H**), δ 6.76 (s,1H,-CO-C**H**=C<).

ANTIMICROBIAL SCREENING

The antifungal activities of compounds 4 a-e have been assayed at the concentration of 200 µg/disc assays against four plants pathogenic and moulds fungi. The inhibitory effects of compounds 4 a-e against these organisms are given in Table 1a.The screening results indicate that the compound 4a-e shows good to moderate antifungal activities to the tested fungi against *Curvularia eryostides, Drecheslera tetrameda, Fusarium cicerg, and Bipolaris sorokenia.*

Table 1a
Antifungal activity of flavones

S.N	Name of compounds	Zone of inhibition in mm.			
		Curvularia eryostides	*Drecheslera tetrameda*	*Fusarium cicerg*	*Bipolaris sorokenia*
1	4a	10	12	14	07
2	4b	08	14	10	14
3	4c	09	12	11	06
4	4d	07	06	10	14
5	4e	16	02	12	16

RESULTS AND DISCUSSION

The substituted 2-hydroxy acetophenone was condensed with aromatic aldehydes to obtain curresponding 2-hydroxy chalcones (3a-e). the structure of this compound were established from their physical and spectral data. The IR spectrum of 3a-e shows absorption band in the region 1630-1650 cm^{-1} (C=O) and 3295-3480 cm^{-1} (2'-OH).

The flavones (4a-e) were obtained by oxidative cyclization of 2-hydroxy chalcones (3a-e). The IR spectrum shows absence of band in the region 2995-3080 cm^{-1} (2'-OH). ^{1}HNMR spectra shows singlet at δ 6.9 due to $-COCH_3$ proton.

All synthesised flavone were evaluated for in vitro antifungal screening. It is observed that the all flavone shows good to moderate antifungal activity. The results are shown in table (table 1a).

CONCLUSION

In summary, we have synthesised some chalcones having 2-hydroxy acetophenone moiety and convert them into flavones. The antifungal screening of flavones 4a-e were found to be active and due to presence of chlorine on phenyl ring increases the activity of the compound.

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