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SYNTHESIS AND ANTIBACTERIAL ACTIVITY OF SOME NEW CHALCONES AND FLAVONES HAVING 2-CHLORO-8-METHOXYQUINOLINYL MOIETY

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ABSTRACT:

New chalcones (3a-h) were synthesized from 2-chloro-8-methoxy-quinoline-3-carbaldehyde (2) and halohydroxysubstitued acetophenones (1a-h) via Claisen-Schmidt condensation. Further new flavones (4a-h) were synthesized by oxidative cyclisation of chalcones (3a-h) using microwave as well as by conventional method. The structures of synthetic compounds were established on the basis of analytical and spectral data. These compounds were screened for their antibacterial activity.

KEYWORDS:

2-chloro-8-methoxy-quinoline-3-carbaldehyde, halohydroxysubstitued acetophenones, chalcones, flavones, antibacterial activity.

INTRODUCTION:

Chalcones, analogs of 1,3-diarylprop-2-en-1-one, form a wide class of compounds containing two aromatic rings bound with vinyl ketone fragment. They are useful in synthesis of various heterocyclic compounds.Chalcones of plant origin are known[1].Chalcones present great interest as compounds exhibiting antimalarial[2], antibactrial[3], antifibrogenic[4], anticancer[5], antitrichomonal[6], anti-inflammatory[7], antileishmanial[8], cytotoxic and anti-trypanosoma cruzi[9] activities. While the flavonoid 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[10], antihypertensive[11], antiallergic[12], antinocicepative[13], trypsin inhibitors[14], plant growth regulator[15], antibacterial and antifungal[16,17] activities.

In the last few years microwave induced organic reaction enhancement (MORE) chemistry has gained popularity as a non-conventional technique for rapid organic synthesis[18] and many researchers have described accelerated organic reactions, and a large number of papers has appeared. Proving the synthetic utility of SMORE chemistry in routine organic synthesis[19]. It has been termed as ‘e-chemistry’ because it is easy, effective, economical and ecofriendly and is belived to be a step toward green chemistry. In view of these observations and in continuation of our work on

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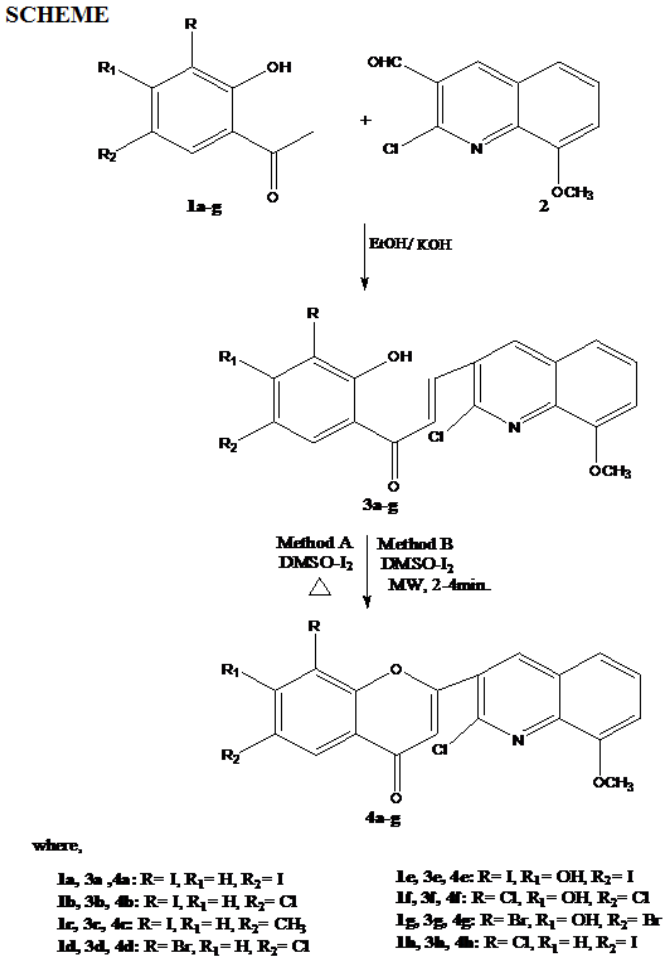
biologically active chalcones and their heterocycles[20], we have been planned to synthesize the new flavones (4a-i) and 1,5-benzothiazepines (5a-i) from chalcones (3a-i) and also studied their antibacterial activity against *Xanthomanas citri* (Xc), *Ervinia carotovara* (Ec), *Escherichia coli* (E. coli) and *Bacillus subtilis* (Bs) using *Ampicillin* as a standard drug.

MATERIALS AND METHODS:

All melting points are taken in open glass capillaries and were found uncorrected. The purity of compounds has been checked by TLC on silica gel G. The IR spectra in KBr were recorded on Shimadzu spectrophotometer and ^{1}HNMR spectra were recorded in DMSO on Varian Inova 300 FT MHz spectrophotometer using TMS as internal standard (δ ppm). Elemental analyses were performed on a Perkin-Elmer 240 CHN elemental analyzer.

SCHEME



where,

1a, 3a ,4a: R= I, R_1= H, R_2= I
1b, 3b, 4b: R= I, R_1= H, R_2= Cl
1c, 3c, 4c: R= I, R_1= H, R_2= CH_3
1d, 3d, 4d: R= Br, R_1= H, R_2= Cl
1e, 3e, 4e: R= I, R_1= OH, R_2= I
1f, 3f, 4f: R= Cl, R_1= OH, R_2= Cl
1g, 3g, 4g: R= Br, R_1= OH, R_2= Br
1h, 3h, 4h: R= Cl, R_1= H, R_2= I

General procedure for synthesis of Chalcones (3a-h):

Equimolar quantities of halo substituted 2-hydroxyacetophenone (0.01mol) and 2-chloro-6-methyl-quinoline-3-carbaldehyde (0.01 mol)

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were dissolved in ethanol (15 ml), under stirring and aqueous KOH (50%, 10 ml) was added dropwise. The reaction mixture was stirred at room temperature and kept for 14-16 hr.The reaction mixture was diluted with water and acidified with 10% HCl.The separated solid was filtered and cryststallised from acetic acid to give compounds (3a-h).

1-(2'-hydroxy-3',5'-diiodophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2- propen-1-one (3a): m.p. 146^{0}C. IR (KBr) cm^{-1}: 2995(-OH), 1635(C=O), 1569, 1492(ring C=C), 1051(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.93 (s, 3H, OCH_3), 6.93 (d, 1H, H_α), 7.35 (d, 1H, H_β), 7.38- δ8.47 (m, 6H, Ar-H), 12.91 (s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_3NI_2Cl$ (591.5): C, 38.54; H, 2.02; N, 2.36. Found: C, 38.41; H, 1.91; N, 2.39.

1-(2'-hydroxy-3'-iodo-5'-chlorophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3b): m.p. 163^{0}C. IR (KBr) cm^{-1}: 3032(-OH), 1630(C=O), 1569, 1496(ring C=C), 1052(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.95 (s, 3H, OCH_3), 6.98 (d, 1H, H_α), 7.29 (d, 1H, H_β), 7.35- δ8.44 (m, 6H, Ar-H), 13.05 (s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_3NICl_2$ (500): C, 45.60; H, 2.40; N, 2.80. Found: C, 45.50; H, 2.39; N, 2.82.

1-(2'-hydroxy-3'-iodo-5'-methylphenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3c): m.p.170^{0}C. IR (KBr) cm^{-1}: 3066(-OH), 1632(C=O), 1569, 1487(ring C=C), 1045(C-O). ^{1}HNMR (300 MHz, DMSO): δ δ2.40 (s, 3H, CH_3), 3.93 (s, 3H, OCH_3), 6.91 (d, 1H, H_α), 7.32 (d, 1H, H_β), 7.34- δ8.39 (m, 6H, Ar-H), 13.23(s, 1H, Ar-OH). Anal. Calcd. for $C_{20}H_{15}O_3NICl$ (479.5): C, 50.05; H, 3.12; N, 2.91. Found: C, 50.14; H, 3.07; N, 2.99.

1-(2'-hydroxy-3'-bromo-5'-chlorophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3d): m.p.156^{0}C. IR (KBr) cm^{-1}: 3078(-OH), 1637(C=O), 1571, 1490(ring C=C), 1055(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.97 (s, 3H, OCH_3), 7.02 (d, 1H, H_α), 7.35 (d, 1H, H_β), 7.39- δ8.21 (m, 6H, Ar-H), 12.95(s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_3NBrCl_2$ (453): C, 50.33; H, 2.64; N, 3.09. Found: C, 50.45; H, 2.60; N, 3.03.

1-(2'4'-dihydroxy-3',5'-diiodophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3e): m.p.183^{0}C. IR (KBr) cm^{-1}: 3393(-OH), 1635(C=O), 1574, 1485 (ring C=C), 1050(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.89 (s, 3H, OCH_3), 6.93 (d, 1H, H_α), 7.30 (d, 1H, H_β), 7.39- δ8.21 (m, 5H, Ar-H), 10.83(s, 1H, 4'Ar-OH), 13.32(s, 1H, 2'Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_4NI_2Cl$ (607.5): C, 37.53; H, 1.97; N, 2.30. Found: C, 37.64; H, 1.99; N, 2.39.

1-(2'4'-dihydroxy-3',5'-dichlorophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3f): m.p.150^{0}C. IR (KBr) cm^{-1}: 3405(-OH), 1635(C=O), 1575, 1485 (ring C=C), 1056(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.94 (s, 3H, OCH_3), 6.98 (d, 1H, H_α), 7.32 (d, 1H, H_β), 7.42- δ8.30 (m, 5H, Ar-H), 10.95(s, 1H, 2'Ar-OH), 13.25(s, 1H, 4'Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_4NCl_3$ (424.5): C, 53.71; H, 2.82; N, 3.29. Found: C, 53.58; H, 2.90; N, 3.36.

1-(2'4'-dihydroxy-3',5'-dibromophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3g): m.p.175^{0}C. IR (KBr) cm^{-1}: 3396(-OH), 1637(C=O), 1570, 1480 (ring C=C), 1050(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.93 (s, 3H, OCH_3), 6.95 (d, 1H, H_α), 7.30 (d, 1H, H_β), 7.40- δ8.35 (m, 5H, Ar-H), 10.89(s, 1H, 2'Ar-OH), 13.27(s, 1H, 4'Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_4NBr_2Cl$ (513.5): C, 44.40; H, 2.33; N, 2.72. Found: C, 44.56; H, 2.34; N, 2.79.

1-(2'-hydroxy-3'-chloro-5'-iodophenyl)-3-(2-chloro-8-methoxy-quinolin-3-yl)-2-propen- 1-one (3h): m.p.158^{0}C. IR (KBr) cm^{-1}: 3039(-OH), 1639(C=O), 1567, 1496(ring C=C), 1052(C-O). ^{1}HNMR (300 MHz, DMSO): δ 3.96 (s, 3H, OCH_3), 6.97 (d, 1H, H_α), 7.31 (d, 1H, H_β), 7.31- δ8.49 (m, 6H, Ar-H), 13.05 (s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{12}O_3NICl_2$ (500): C, 45.60; H, 2.40; N, 2.80. Found: C, 45.50; H, 2.37; N, 2.81.

Synthesis of Flavones (4a-h):-

Method A:-

Chalcone (0.01 mol) was suspended in DMSO (10 ml) and a crystal of iodine was added to it. The

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mixture was refluxed for 2h. and diluted with water. The solid obtained was filtered off, washed with 20% sodium thiosulfate and crystallized from ethyl alcohol to give compounds (4a-h). It gave positive Mg/HCl test (yellow colouration).

Method B:-

Chalcone (0.01 mol) was suspended in DMSO (10 ml) and a crystal of iodine was added to it. The mixture was irradiated in microwave oven for the appropriate time (Table-1) at 650 W. After completion of reaction as followed by TLC examination, the solid product was washed with 20% sodium thiosulfate and crystallized from ethyl alcohol to give compounds (4a-h). It gave positive Mg/HCl test (yellow colouration).

2-(2-Chloro-8-methoxy-quinolin-3-yl)-6,8-diiodo-chromen-4-one (4a):

m.p.169^{0}C. IR (KBr) cm^{-1}: 1645(C=O), 1570, 1495 (ring C=C). ^{1}HNMR (300 MHz, DMSO): δ 3.93 (s, 3H, OCH_3), 7.09 (s, 1H, COCH), 7.40- δ8.22 (m, 6H, Ar-H). Anal. Calcd. for $C_{19}H_{10}O_3NI_2Cl$ (589.5): C, 38.67; H, 1.69; N, 2.37. Found: C, 38.52; H, 1.83; N, 2.32.

6-Chloro-2-(2-Chloro-8-methoxy-quinolin-3-yl)-8-iodo-chromen-4-one (4b):

m.p.186^{0}C. IR (KBr) cm^{-1}: 1643(C=O), 1571, 1493(ring C=C). ^{1}HNMR (300 MHz, DMSO): δ 3.95 (s, 3H, OCH_3), 6.98 (s, 1H, COCH), 7.30- δ8.15 (m, 6H, Ar-H). Anal. Calcd. for $C_{19}H_{10}O_3NICl_2$ (498): C, 45.78; H, 2.0; N, 2.81. Found: C, 45.85; H, 2.03; N, 2.82.

2-(2-Chloro-8-methoxy-quinolin-3-yl)-8-iodo-6-methyl-chromen-4-one (4c): m.p.157^{0}C. IR (KBr) cm^{-1}: 1632(C=O), 1565, 1490(ring C=C). ^{1}HNMR (300 MHz, DMSO): δ δ2.45 (s, 3H, CH_3), 3.93 (s, 3H, OCH_3), 6.99 (s, 1H, COCH), 7.37- δ8.20 (m, 6H, Ar-H). Anal. Calcd. For $C_{20}H_{13}O_3NICl$ (477.5): C, 50.26; H, 2.72; N, 2.93. Found: C, 50.18; H, 2.79; N, 2.88.

8-Bromo-6-chloro-2-(2-Chloro-8-methoxy-quinolin-3-yl)-chromen-4-one (4d):

m.p.171^{0}C. IR (KBr) cm^{-1}: 1645(C=O), 1568, 1495(ring C=C). ^{1}HNMR (300 MHz, DMSO): δ 3.95 (s, 3H, OCH_3), 7.06 (s, 1H, CHOH), 7.40- δ8.10 (m, 6H, Ar-H). Anal. Calcd. for $C_{19}H_{10}O_3NBrCl_2$ (451): C, 50.55; H, 2.21; N, 3.10. Found: C, 50.50; H, 2.29; N, 3.18.

2-(2-Chloro-8-methoxy-quinolin-3-yl)-7-hydroxy-6,8-diiodo-chromen-4-one (4e):

m.p.196^{0}C.IR (KBr) cm^{-1}: 3405(-OH), 1639(C=O), 1570, 1489 (ring C=C).

^{1}HNMR (300 MHz, DMSO): δ 3.93 (s, 3H, OCH_3), 7.02 (s, 1H, CHOH), 7.35- δ8.17 (m, 5H, Ar-H), 10.92(s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{10}O_4NI_2Cl$ (605.5): C, 37.65; H, 1.65; N, 2.31. Found: C, 37.78; H, 1.71; N, 2.28.

6,8-Dichloro-2--(2-Chloro-8-methoxy-quinolin-3-yl)-7-hydroxy-chromen-4-one(4f):

m.p. 164^{0}C. IR (KBr) cm^{-1}: 3405(-OH), 1644(C=O), 1579, 1489 (ring C=C).

^{1}HNMR (300 MHz, DMSO): δ 3.97 (s, 3H, OCH_3), 7.06 (s, 1H, CHOH), 7.45- δ8.25 (m, 5H, Ar-H), 10.97(s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{10}O_4NCl_3$ (422.5): C, 53.96; H, 2.36; N, 3.31. Found: C, 54.08; H, 2.30; N, 2.37.

6,8-Dibromo-2--(2-Chloro-8-methoxy-quinolin-3-yl)-7-hydroxy-chromen-4-one(4g):

m.p.162^{0}C. IR (KBr) cm^{-1}: 3400(-OH), 1647(C=O), 1567, 1485 (ring C=C).

^{1}HNMR (300 MHz, DMSO): δ 3.95 (s, 3H, OCH_3), 7.01 (s, 1H, CHOH), 7.43- δ8.33 (m, 5H, Ar-H), 10.95(s, 1H, Ar-OH). Anal. Calcd. for $C_{19}H_{10}O_4NBr_2Cl$ (511.5): C, 44.57; H, 1.95; N, 2.73. Found: C, 44.44; H, 2.03; N, 2.80.

8-Chloro-2--(2-Chloro-8-methoxy-quinolin-3-yl)-6-iodo-chromen-4-one (4h):

m.p.178^{0}C. IR (KBr) cm^{-1}: 1643(C=O), 1571, 1493(ring C=C). ^{1}HNMR (300 MHz, DMSO): δ 3.97 (s, 3H, OCH_3), 7.01 (s, 1H, COCH), 7.31- δ8.23 (m, 6H, Ar-H). Anal. Calcd. for $C_{19}H_{10}O_3NICl_2$ (498): C, 45.78; H, 2.0; N, 2.81. Found: C, 45.88; H, 2.03; N, 2.84.

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Table 1

Physical data of Flavones (4a-h)

Compound code	Reaction Period (h/ min.)		Yield (%)	
	Method A (h)	Method B (min.)	Method A	Method B
4a	2	4	70	83
4b	2	4	64	86
4c	1.5	2.5	59	92
4d	1.5	3	69	81
4e	2	3.5	73	90
4f	1.5	2	66	87
4g	2	3.5	71	89
4h	1.5	2.5	67	93

***Antibacterial screening*:**

The antibacterial activity of newly synthesized compounds (**3a-h** and **4a-h**) was determined by agar diffusion method.[21]. The compounds were evaluated for antibacterial activity was against *Xanthomanas citri* (Xc), *Ervinia carotovara* (Ec), *Escherichia coli* (E. coli) and *Bacillus subtilis* (Bs).The antibiotic *Ampicillin* (25µg/mL) was used as standard antibiotic and 1% DMSO was used as solvent control.

The culture strains of bacteria were maintained on nutrient agar slant at 37±0.5°C for 24 h. The antibacterial activity was evaluated using nutrient agar plate seeded with 0.1 mL of respective bacterial culture strain suspension prepared in sterial saline (0.85%) of 10^5 CUF/ mL dilution. The wells of 6mm diameter were filled with 0.1 mL of solution at fixed concentration 25µg/mL separately for each bacterial strain. All the plates were incubated at 37±0.5°C for 24 h. The zone of inhibition of compounds was measured using mm scale.

Table 2: Antibacterial activity data of synthesized compounds

Compound code	Zone of inhibition (mm)			
	Xc	Ec	E. coli	Bs
3a	15	13	16	10
3b	18	20	20	22
3c	12	14	09	11
3d	14	11	13	08
3e	25	27	24	27
3f	23	25	22	29
3g	13	16	11	14
3h	18	17	11	15
4a	12	14	11	10
4b	14	12	14	11
4c	13	11	08	09
4d	11	14	10	12
4e	20	27	24	22
4f	23	24	20	26
4g	12	10	12	15
4h	24	18	20	25
Control	----	----	----	----
***Ampicillin* 25µg/mL**	25	28	22	27

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RESULTS AND DISCUSSION

2-Chloro-8-methoxy-quinoline-3-carbaldehyde (2) was condensed with halohydroxysubstitued acetophenones (1a-h) to obtain the corresponding 2'-hydroxychalcones (3a-h).The structure of these products were established from their physical and spectral data. The IR spectrum of 3a-h showed absorption bands in the region 1630-1640 cm^{-1}(C=O) and 2995-3080 cm^{-1}(2'-OH). The ^{1}HNMR further support for their structure and showed doublet of doublet in the region δ 6.91-7.35 due to olefinic protons and multiplet in the region δ 7.39-8.47 due to aromatic protons and also show singlet in the region δ 12.91-13.27 due to proton of ortho hydroxyl group.

Flavones (4a-h) were obtained by oxidative cyclisation of 2'-hydroxychalcones (1a-h).The IR spectra of these compound showed absences of absorption band in the region 2995-3080 cm^{-1}(2'-OH). Its ^{1}HNMR spectra showed singlet at δ 6.98-7.10 due to -COCH proton and absence of singlet in the region δ 12.91-13.27 due to proton of ortho hydroxyl group.

All the newly synthesized compounds were evaluated for *in vitro* antibacterial activity. The results are showed in Table-2. It has been observed that compounds **3e, 3f, 4e, 4f** and **4h** indicated better activity while **3b** and **3h** showed good activity than standard *Ampicillin*. The remaining compounds were less active than the reference drug.

CONCLUSION

In summery, we have synthesized some bioactive chalcones and convert them into flavones by using conventional method as well as microwave irradiation. Short reaction time, clean reaction with high yield than conventional method is the advantages of microwave irradiation method. The antibacterial study show that compound **3e, 3f, 4e, 4f** and **4h** were found to be active. Due to presence of iodine/ chlorine along with hydroxyl group/s on phenyl ring increase the activity of these compounds.

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