MICROWAVE ASSISTED SYNTHESIS OF SUBSTITUTED 1,2,3,4-TETRAHYDRO-2-PYRIMIDINONES AND 1,2,3,4-TETRAHYDRO-2-PYRIMIDINETHIONES FROM QUINOLINE CHALCONES

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Abstract :

The synthesis of heterocyclic compounds 1,2,3,4-tetrahydro-2-pyrimidinones **(3a-f)** and 1,2,3,4-tetrahydro-2-pyrimidinethiones **(4a-f)** from quinoline chalcones under solvent free conditions using microwaves and by conventional methods are described.

Introduction:

Quinoline derivatives are associated with immense biological activities[1-5], likewise dihydropyrimidines and -tetrahydro-2-pyrimidinones also found to be associated with wide range of biological activities[6,7]. Moreover quinoline drugs are considered very effective for the treatment of malaria and amoebic infections[8,9]. In view of the biological activities of these nitrogen heterocyclics, we developed novel route for synthesis of quinoline substituted tetrahydro-2-pyrimidinones and tetrahydro-2-pyrimidinethiones from quinoline chalcones.

Microwave irradiation using commercial domestic ovens has been used to accelerate organic reactions, the high heating efficiency giving remarkable rate enhancement and dramatic reduction of reaction times[10,11].In order to avoid the experimental difficulties arising from the use of homogeneous solvent we described here a novel synthesis of quinoline substituted pyrimidinones and 2-pyrimidinethiones by microwave irradiation on solid inorganic materials in the absence of solvent. It has been reported that synthesis of various biodynamic heterocycles pyrimidones, pyrazoline and Isoxazoline derivatives from simple chalcones with microwave technology[12].

The required synthons substituted 2-chloro-3-formyl quinolines were prepared by literature methods[13-15]. 2-chloro-3-formyl quinolines were condensed with substituted acetophenones. The resulting products Quinoline chalcones on reaction with urea and thiourea under Microwave irradiation on solid neutral alumina support afforded 1,2,3,4-tetrahydro-2-pyrimidinones and 1,2,3,4-tetrahydro-2-pyrimidinethiones All these prepared compounds structures were established by spectral data.

Results and discussion:

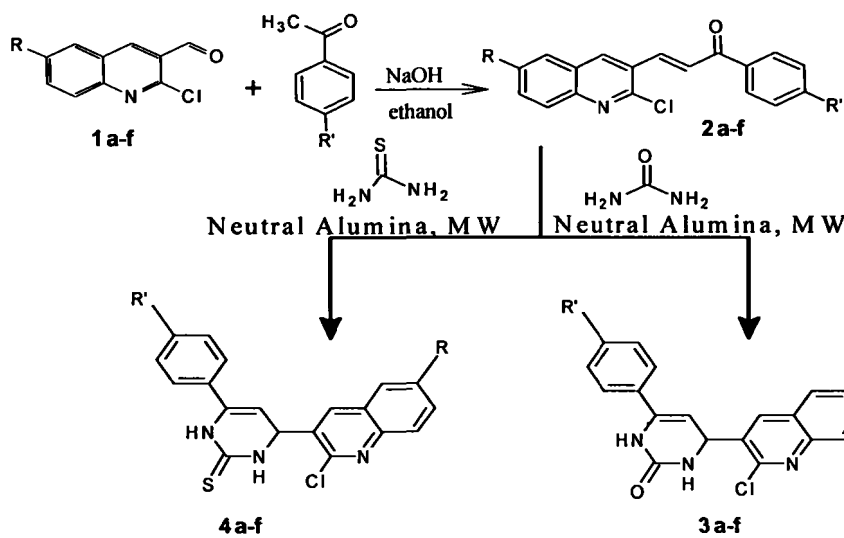
2-chloro quinoline-3-aldehyde**(1a)** condensed with acetophenone in alkali to obtain corresponding chalcones E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one **(2a)**. Similarly, **2b-f** were prepared. In it's IR **2a** shows 1669 cm^{-1} carbonyl and in it's ^{1}H-NMR **2a** shown signals corresponding to olefinic protons δ 7.41, d, H-4, 1H, J=16.8Hz; δ 8.20, d, H-3, 1H, J=16.8Hz; and other aromatic protons appeared at δ 7.64, m, 5H, C_1-Phenyl protons; δ 8.42, s, H-5 (quinoline); δ 7.82, m, 4H(quinoline). The analytical and spectral data of **2a-2f** are given in experimental section.

Compound **2a** treated with urea and adsorbed on neutral alumina, on Microwave irradiation afforded 6-(phenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone **3a**. In it's IR **3a** shows peaks shows at 3250cm^{-1}(N-H),1680cm^{-1} (C=O) 1640 cm^{-1} (C=N) and in its ^{1}H-NMR **3a** shows peaks at δ 5.10, d, H-4, 1H, J=5.2Hz; δ5.25, d, H-5, 1H, J=5.2Hz, indicates tetrahydro pyrimidinone formation, δ 7.22,m, 5H(Phenyl); δ 7.78, m, 4H(quinonyl); δ 7.88, s, H-4(quinonyl), 1H; δ 9.50, 2H(bs) N-H. Similarly, Compound **2a** treated with thiourea and adsorbed on neutral alumina, on Microwave irradiation afforded 6-(4-chlorophenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinethione **4a**. In IR **4a** shows peaks 3330cm^{-1} (N-H), 1640cm^{-1} (C=N), 1130cm^{-1} (C=S) and in it's ^{1}H-NMR **4a** has shown δ 5.00, d, H-4, 1H, J=5.2Hz; δ5.15, d, H-5, 1H, J=5.2Hz, (tertahydro-2-pyrimidinethione protons) δ 7.20,m, 5H(Phenyl); δ 7.58, m,4H(quinonyl); δ 7.80, s, H-4(quinonyl), 1H; δ 9.30, 2H(bs) N-H. Similarly **3a-f** , **4a-f** were prepared, the analytical and spectral data of **3a-f** and **4a-f** are given in experimental section.The reaction periods for the formation of the compounds **(3a-f)** and **(4a-f)** are given in the **Table-1**.

Table-1

compound No	REACTION PERIOD (hrs / mins)		compound No	REACTION PERIOD (hrs / mins)	
	Method-1(hrs)	Method-2 (mins)		Method-1(hrs)	Method-2 (mins)
3a	6	30	4a	8	35
3b	65	30	4b	75	40
3c	70	35	4c	8.5	35
3d	65	25	4d	75	35
3e	6	3.0	4e	80	30
3f	65	35	4f	80	30

Scheme



a) R=H & R'= H b) R=H & R'= Cl c) R=H & R'= Br d) R=H & R'= CH_3
e) R=H & R'= OCH_3 f) R= OCH_3 & R'=H

EXPERIMENTAL:

Melting points were determined in a sulphuric acid bath and are uncorrected. IR spectra are recorded in KBr on a Shimadzu-435 spectrometer, H^1-NMR spectra were obtained on Varian Gemini-200MHz spectrometer with TMS as an internal standard. Mass spectra were recorded on Perkin-Elmer Hitichi RDO-62 instrument. MWI were carried out in Padmini Essentia oven, Model Brownie at 2459MHz

General procedure for the synthesis of E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-ones (2a-2f):

Aectophenone (0.01ml), 2-chloro quinoline-3-carboxaldehyde(**1a**) (0.01ml), are dissolved in 20ml of methanol and 40% NaOH was added. The reaction mixture was then stirred for about 10hrs at room temperature. The product isolated was crystallized from ethanol.

E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one- 2a: yield - 78%, m.p 105°c;IR (KBr) 1669 cm^{-1} (C=O); ^{1}H-NMR δ 7.41, d, H-4, 1H, J=16.8Hz; δ 8.20, d, H-3, 1H, J=16.8Hz; δ 7.64, m, 5H, C_1-Phenyl protons; δ 8.42, s, H-5 (quinoline); δ 7.82, m, 4H(quinoline); M^+=293.

(*E*)-1-(4-chlorophenyl)-3-(2-chloro-3-quinolyl)-2-propen-1-one -2b: yield - 80%, m.p. 176°c IR (KBr) 1668 cm^{-1} (C=O); ^{1}H-NMR δ 7.42, d, H-2, 1H, J=16.9Hz; δ 8.21, d, H-3, 1H, J=16.9Hz; δ 7.65, d, H-2′, H-6′, 2H, J=8.1Hz; δ 7.63,d, H-3′, H-5′, 2H, J= 8.1Hz); δ 8.44, s, H-5 (quinoline); δ 7.83, m, 4H(quinoline) ; M^+=327.

(*E*)-1-(4-bromophenyl)-3-(2-chloro-3-quinolyl)-2-propen-1-one -2c: yield - 75%, m.p.180°c IR (KBr) 1667 cm^{-1} (C=O); ^{1}H-NMR δ 7.40, d, H-2, 1H, J=16.7Hz; δ 8.20, d, H-3, 1H, J=16.7Hz; δ 7.63, d, H-2′, H-6′, 2H, J= 7.8Hz; δ 7.60, d, H-3′, H-5′, 2H, J= 7.8Hz); δ 8.41, s, H-5 (quinoline); δ 7.80, m, 4H(quinoline); δ2.26, s, H-4′, 3H, CH_3; M^+=371.

(*E*)-3-(2-chloro-3-quinolyl)-1-(4-methylphenyl)-2-propen-1-one -2d: yield - 78%, m.p 150°c IR (KBr) 1665 cm^{-1} (C=O); ^{1}H-NMR δ 7.43, d, H-2, 1H, J=17.0Hz; δ 8.22, d, H-3, 1H, J=17.0Hz; δ 7.66, d, H-2′, H-6′, 2H, J=8.1Hz; δ 7.64, d, H-3′, H-5′, 2H, J= 8.0Hz); δ 8.45, s, H-5 (quinoline); δ 7.84, m, 4H(quinoline) ; M^+=307.

(*E*)-3-(2-chloro-3-quinolyl)-1-(4-methoxyphenyl)-2-propen-1-one- 2e: yield- 77%, m.p 165°c IR (KBr) 1662 cm^{-1} (C=O); ^{1}H-NMR δ 7.41, d, H-2, 1H, J=17.0Hz; δ 8.21, d, H-3, 1H, J=17.0Hz; δ 7.66, d, H-2′, H-6′, 2H, J=8.0Hz; δ 7.63, d, H-3′, H-5′, 2H, J= 8.0Hz); δ 8.47, s, H-5 (quinoline); δ 7.84, m, 4H(quinoline); δ 3.83, s, 3H, OCH_3; M^+=323.

(*E*)-3-(2-chloro-6-methoxy-3-quinolyl)-1-phenyl-2-propen-1-one -2f : yield - 76%, m.p 165°c IR (KBr) 1660 cm^{-1} (C=O); ^{1}H-NMR δ 7.40, d, H-2, 1H, J=17.0Hz; δ 8.20, d, H-3, 1H, J=17.0Hz; δ 7.30, m, 5H(phenyl), δ 8.44, s, H-5 (quinoline); δ 7.82, m, 3H(quinoline), δ 3.82,s,OCH_3; M^+=323.

General procedure for the synthesis of 6-(phenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinones (3a-3f):

Method-1

A mixture of chalcone E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one **2a** (0.005 mol) and urea (0.005mol) in AcOH(10ml) was refluxed for 6 hrs. Reaction mixture was concentrated under reduced pressure then poured into cold water, stirred well the resulting product 6-(phenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone **3a** was chromatographed on silica gel eluting with chloroform.

Method-2

Chalcone E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one **2a** (0.005 mol) and urea (0.005mol) are dissolved in ethanol(10ml) and neutral alumina was added, the reaction mixture was mixed thoroughly and dried, the adsorbed material was placed in an alumina bath inside the microwave oven and then irradiated for 3 minutes, the reaction was monitored by TLC, the mixture was cooled to room temperature and then the product 6-(phenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone **3a** was extracted with $CHCl_3$, evaporation of solvent under reduced pressure afforded crude product **3a** was chromatographed on silica gel eluting with $CHCl_3$

6-(phenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone-3a: yield-79%, m.p 120°c; IR (KBr) 3250cm^{-1}(N-H), 1680cm^{-1} (C=O) 1640 cm^{-1} (C=N); ^{1}H-NMR δ 5.10, d, H-4, 1H, J=5.2Hz; δ5.25, d, H-5, 1H, J=5.2Hz; δ 7.22,m, 5H(Phenyl); δ 7.78, m, 4H(quinonyl); δ 7.88, s, H-4(quinonyl), 1H; δ 9.50, 2H(bs) N-H; M$^+$=335.

6-(4-chlorophenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone-3b: yield-77%, m.p 160°c; IR (KBr) 3246cm^{-1}(N-H),1678cm^{-1} (C=O) 1638 cm^{-1} (C=N); ^{1}H-NMR δ 5.12, d, H-4, 1H, J=5.3Hz; δ 6.26, d, H-5, 1H, J=5.3Hz; δ 7.25,d, H-3′,5′, 2H, J= 7.8Hz; δ 7.33,d, H-2′,6′, 2H, J= 7.8Hz); δ 7.79, m, 4H(quinonyl); δ 7.90, s, H-4(quinonyl), 1H; δ 9.60, 2H(bs) N-H; M$^+$=369.

6-(4-bromophenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinone-3c: yield-78%, m.p 165°c; IR (KBr) 3248cm^{-1}(N-H),1681cm^{-1} (C=O) 1639 cm^{-1} (C=N); ^{1}H-NMR δ 5.11, d, H-4, 1H, J=5.2Hz; δ 6.25, d, H-5, 1H, J=5.2Hz; δ 7.25,d, H-3′,5′, 2H, J= 7.7Hz; δ 7.32,d, H-2′,6′, 2H, J= 7.7Hz); δ 7.78, m, 4H(quinonyl); δ 7.89, s, H-4(quinonyl), 1H; δ 9.59, 2H(bs) N-H; M$^+$=413.

4-(2-chloro-3-quinolyl)-6-(4-methylphenyl)-1,2,3,4-tetrahydro-2-pyrimidinone-3d: yield-75%, m.p 171°c; IR (KBr) 3252cm^{-1}(N-H),1681cm^{-1} (C=O) 1641 cm^{-1} (C=N); ^{1}H-NMR δ 5.80, d, H-4, 1H, J=5.1Hz; δ 6.23, d, H-5, 1H, J=5.1Hz; δ 7.22,d, H-3′,5′, 2H, J= 7.4Hz; δ 7.32,d, H-2′,6′, 2H, J= 7.4Hz); δ 7.76, m, 4H(quinonyl); δ 7.88, s, H-4(quinonyl), 1H; δ 9.56, 2H(bs) N-H; δ2.3, s, 3H,CH_3; M$^+$=349.

4-(2-chloro-3-quinolyl)-6-(4-methoxyphenyl)-1,2,3,4-tetrahydro-2-pyrimidinone-3e: yield- 76%, m.p 128°c IR (KBr) 3252cm^{-1}(N-H),1682cm^{-1} (C=O) 1642 cm^{-1} (C=N); ^{1}H-NMR δ 5.87, d, H-4, 1H, J=5.4Hz; δ 6.27, d, H-5, 1H, J=5.4Hz; δ 7.25,d, H-3′,5′, 2H, J= 7.5Hz; δ 7.35,d, H-2′,6′, 2H, J= 7.5Hz); δ 7.78, m, 4H(quinonyl), 1H; δ 7.91, s, H-4(quinonyl), 1H; δ 9.58, 2H, (bs) N-H; δ3.84, s, 3H, OCH_3; M$^+$=365.

4-(2-chloro-6-methoxy-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinone-3f:yield- 77%, m.p 131°c; IR (KBr) 3248cm^{-1}(N-H),1678cm^{-1} (C=O) 1637 cm^{-1} (C=N); ^{1}H-NMR δ 5.82, d, H-4, 1H, J=5.2Hz; δ 6.24, d, H-5, 1H, J=5.2Hz; δ 7.22, m, 5H(phenyl); δ 7.70, m, 3H(quinonyl); δ 7.92, s, H-4(quinonyl), 1H; δ 9.60, 2H, (bs) N-H; δ3.86, s, 3H, OCH_3. ; M$^+$=365.

General procedure for the synthesis of 4-(2-chloro-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinethiones (4a-4f):

Method-1

A mixture of chalcone E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one **2a** (0.005 mol) and thiourea (0.005mol) in AcOH(10ml) was refluxed for 8 hrs. Reaction mixture was concentrated under reduced pressure then poured into cold water, stirred well the resulting product 4-(2-chloro-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinethione **4a** was chromatographed on silica gel eluting with chloroform.

Method-2

Chalcone E-3-(2-chloro-3-quinolyl)-1-phenyl-2-propen-1-one **2a** (0.005 mol) and thiourea (0.005mol) are dissolved in ethanol(10ml) and neutral alumina was added, the reaction mixture was mixed thoroughly and dried, the adsorbed material was placed in an alumina bath inside the microwave oven and then irradiated for 3.5 minutes, the reaction was monitored by TLC, the mixture was cooled to room temperature and then the product was extracted with $CHCl_3$, evaporation of solvent under reduced pressure afforded crude 4-(2-chloro-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinethione **4a** was chromatographed on silica gel eluting with $CHCl_3$.

4-(2-chloro-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinethione-4a: yield- 77%, m.p. 124°c; IR (KBr) 3330cm^{-1}(N-H), 1640 cm^{-1} (C=N),1130 cm^{-1} (C=S); ^{1}H-NMR δ 5.00, d, H-4, 1H, J=5.2Hz; δ5.15, d, H-5, 1H, J=5.2Hz; δ 7.20,m, 5H(Phenyl); δ 7.58, m, 4H(quinonyl); δ 7.80, s, H-4(quinonyl), 1H; δ 9.30, 2H(bs) N-H; M$^+$=351.

6-(4-chlorophenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinethione-4b: yield- 77%, m.p. 165°c; IR (KBr) 3240cm^{-1}(N-H), 1635 cm^{-1} (C=N), 1130 cm^{-1} (C=S); ^{1}H-NMR δ 5.11, d, H-4, 1H, J=5.2Hz; δ 6.22, d, H-5, 1H, J=5.2Hz; δ 7.25,d, H-3′,5′, 2H, J= 7.8Hz;δ 7.31,d, H-2′,6′, 2H, J= 6.0Hz); δ 7.77, m, 4H(quinonyl); δ 7.91, s, H-4(quinonyl), 1H;δ 9.50, 2H(bs) N-H; M$^+$=385.

6-(4-bromophenyl)-4-(2-chloro-3-quinolyl)-1,2,3,4-tetrahydro-2-pyrimidinethione-4c: yield- 77%, m.p. 170°c; IR (KBr) 3246cm^{-1}(N-H),1638 cm^{-1} (C=N), 1130 cm^{-1} (C=S); ^{1}H-NMR δ 5.10, d, H-4, 1H, J=5.2Hz; δ 6.24, d, H-5, 1H, J=5.2Hz; δ 7.24,d, H-3′,5′, 2H, J= 7.6Hz; δ 7.31,d, H-2′,6′, 2H, J= 7.6Hz); δ 7.76, m, 4H(quinonyl); δ 7.87, s, H-4(quinonyl), 1H; δ 9.57, 2H(bs) N-H; M$^+$=429.

4-(2-chloro-3-quinolyl)-6-(4-methylphenyl)-1,2,3,4-tetrahydro-2-pyrimidinethione-4d: yield-77%, m.p. 176°c; IR (KBr) 3251cm^{-1}(N-H),1641 cm^{-1} (C=N), 1131 cm^{-1} (C=S); ^{1}H-NMR δ 5.75, d, H-4, 1H, J=5.1Hz; δ 6.21, d, H-5, 1H, J=5.1Hz; δ 7.21,d, H-3′,5′, 2H, J= 7.3Hz; δ 7.31,d, H-2′,6′, 2H, J= 7.3Hz); δ 7.76, m, 4H(quinonyl); δ 7.87, s, H-4(quinonyl), 1H; δ 9.56, 2H(bs) N-H; δ2.30, s, 3H,CH_3; M$^+$=365.

4-(2-chloro-3-quinolyl)-6-(4-methoxyphenyl)-1,2,3,4-tetrahydro-2-pyrimidinethione-4e: yield-77%, m.p. 132°c; IR (KBr) 3250cm^{-1}(N-H),1642 cm^{-1} (C=N), 1129 cm^{-1} (C=S); ^{1}H-NMR δ 5.85, d, H-4, 1H, J=5.4Hz; δ 6.28, d, H-5, 1H, J=5.4Hz; δ 7.24,d, H-3′,5′, 2H, J= 7.5Hz; δ 7.34,d, H-2′,6′, 2H, J=7.5Hz; δ 7.76, m, 4H(quinonyl), 1H; δ 7.90, s, H-4(quinonyl), 1H; δ 9.56, 2H, (bs) N-H; δ3.84, s, 3H, OCH_3; M$^+$=381.

4-(2-chloro-6-methoxy-3-quinolyl)-6-phenyl-1,2,3,4-tetrahydro-2-pyrimidinethione-4f: yield-77%, m.p. 135°c; IR (KBr) 3245cm^{-1}(N-H),1637 cm^{-1} (C=N), 1130 cm^{-1} (C=S); ^{1}H-NMR δ 5.81, d, H-4, 1H, J=5.2Hz; δ 6.23, d, H-5, 1H, J=5.1Hz; δ 7.21, m, 5H(phenyl); δ 7.68, m, 3H(quinonyl); δ 7.90, s, H-4(quinonyl), 1H; δ 9.50, 2H, (bs) N-H; δ3.84, s, 3H, OCH_3; M$^+$=381.

Conclusions:

The salient features of this microwave assisted synthesis are modernization and simplification of classical procedures avoiding volatile and toxic organic solvents, corrosive mineral acids, which make it a clean, efficient and cheap technology to obtain 1,2,3,4-tetrahydro-2-pyrimidinones **(3a-f)** and 1,2,3,4-tetrahydro-2-pyrimidinethiones **(4a-f)** compounds.

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