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An efficient Synthesis and Characterization of New Bis-chalcones

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Abstract: A series of new bis-chalcones (2a-e) were synthesized by Claisen-Schmidt condensation of 2,4-diacyl phenol with variously substituted aryl aldehydes in good to excellent yield. Starting material 2,4-diacyl phenol(1a) was prepared by reacting 2-hydroxyacetophenone with acetyl chloride. The structure of the synthesized compounds were characterized by IR, ^{1}H-NMR, and mass spectral analysis.
Keywords: Chalcones, bis-chalcones, condensation, biological activities.

1. Introduction:

Chalcone is the trivial name given to the α,β-unsaturated carbonyl compounds and their IUPAC name is 1,3-Diphenyl-2-propene-1-one. They are designated structurally as Ar-CH=CH-CO-Ar'. The substituents attached to the benzene rings of chalcone are numbered as shown in the figure 1 below[1].

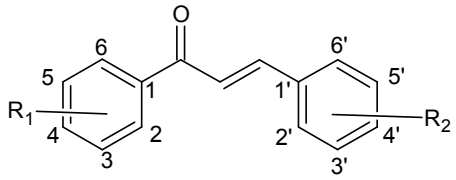


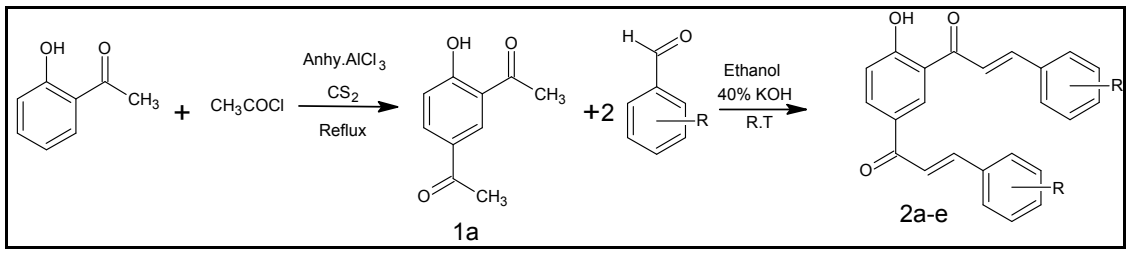
Figure 1: **Structure of chalcones**

Chalcones contain the reactive ketoethylenic group –CO-CH=CH-. These are coloured and biologically active compounds because of the presence of the chromophoric unit –CO-CH=CH-. In chalcones two aromatic rings are linked by an aliphatic three carbon chain. All the α,β-unsaturated ketones are not necessarily the chalcones but all the chalcones are the α,β-unsaturated ketones. If the groups attached to –CH=CH-CO- moiety do not possess aromaticity, then the resulting compound is just the α,β-unsaturated ketone and not the chalcone.

Chalcones are abundantly present in nature from ferns to higher plants. Chalcones have been reported to be isolated from various parts of plants, buds, leaves, blossoms, heart wood, roots, seeds, flowers and inflorescence. Chalcone is the major classes of natural products which are widely distributed in spices, tea, beer, fruits and vegetables. Chalcones considered as the precursors of flavonoids and isoflavonoids and structurally chalcones are open-chain flavonoids in which the two aromatic rings are joined by a three carbon α,β-unsaturated carbonyl system. Chalcones are either natural or synthetic and known to exhibit a broad spectrum of various biological activities[2]. The presence of α,β-unsaturated carbonyl moiety as well as substituted aromatic rings render the chalcones biologically active. Chalcones have been reported to possess anticaner[3], anti-inflammatory[4], antioxidant[5], antitumour[6], antifungal[7], antibacterial[8], anti-microbial[9] and antitubercular[10] activities. Extensive literature study reveals that majority of scientists have been synthesized chalcones containing only one α,β-unsaturated site, a very little attention is given on the synthesis of bis-chalcones containing two α,β-unsaturated groups[11]. Claisen-Schmidt condensation of 2,4-diacyl phenol with two

equivalents of aryl aldehydes yields bis-chalcones containing two reactive α,β-unsaturated keto-ethylenic groups at ortho and para position of –OH group of 2,4-diacyl phenol. In continuation of our work on "Synthetic studies in 1,5-benzothiazepines" we have prepared bis-chalcones from 2,4-diacyl phenol and planned to synthesize bis-1,5-benzothiazepines, pyrazolines, pyrimidines, isoxazolines using these bis-chalcones, so that different heterocyclic compounds with good pharmaceutical profile can be synthesized.

1.1.Scheme: Synthesis of bis-chalcones.



2. Experimental:

Melting points were determined in an open capillary and are uncorrected. Purity of the compounds was checked by TLC. IR spectra were recorded on Perkin-Elmer FT-IR spectrometer using KBr pellets. ^{1}H NMR, spectra were recorded on Bruker varian 300 MHz instrument using $CDCl_3$ as solvent and TMS as internal reference. Mass spectra were recorded on an EI-MS, DIP-300 spectrometer.

3.1. Result and Discussion:

We describe herein the synthesis of bis–chalcones from 2,4-diacyl phenol. For the synthesis of target compound first 2,4-diacyl phenol (1a) was prepared by the reaction of 2-hydroxyacetophenone with acetyl chloride in presence of anhydrous $AlCl_3$ in CS_2. Bis-chalcones (2a-e) were prepared by reacting 2,4-diacyl phenol with variously substituted aromatic aldehydes in the presence of base by conventional Claisen-Schmidt condensation method. IR spectra of compound (2b) shows absorption bands in the region 3251 cm^{-1} (-OH stretch, intramolecular hydrogen bonded), 1676 cm^{-1} (=C=O, stretch, aromatic and conjugated carbonyl), 1632 cm^{-1} (=C=O, stretch, aromatic and conjugated carbonyl), ^{1}H NMR spectra of compound (2b) displays signals at δ 7.11-8.73 (m,15H, aromatic and vinylic protons), δ 13.34 (s, 1H, -OH phenolic). The structure of all the synthesized compounds were confirmed by FT-IR, ^{1}H NMR and mass spectral analysis.

3.2.General procedure for the synthesis of bis-chalcone:

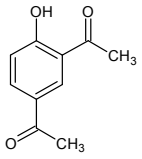
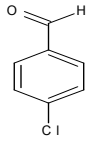
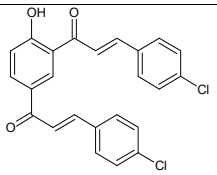
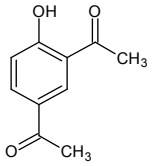
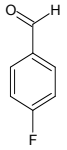
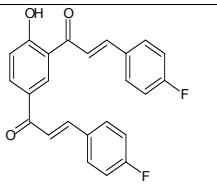
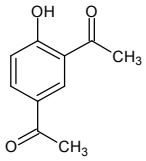
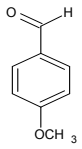
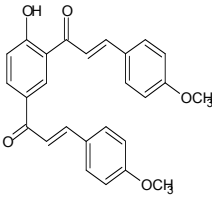
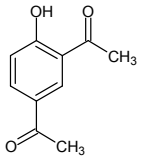
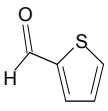
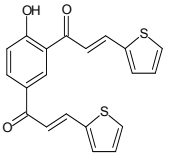
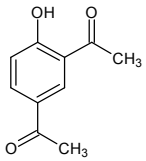
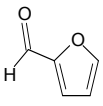
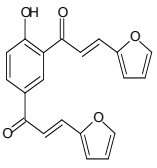
A mixture of 2,4-diacyl phenol (0.01 mole) and aromatic aldehyde (0.02 mole) was dissolved in ethanol (50 ml) and was added a solution of 40% KOH (15 ml) in portions, keeping the temperature below 10^{0}C.The reaction flask was corked and stirred at room temperature for 48 hours. The content of the flask was then poured over ice containing acetic acid. The solid thus obtained was filtered, washed with water and crystallized from acetic acid. Purity of the product was checked by TLC, Melting point, yields are recorded in table1. All the products were characterized by FT-IR, ^{1}H NMR, and Mass spectral analysis.

3.3. Spectral data of synthesized bis-chalcones(2a-e):

1. (2E,2'E)-1,1'-(4-hydroxybenzene-1,3-diyl)bis[3-(4-chlorophenyl)prop-2-en-1-one].(2a): Yellow solid; m.p: 202^{0}C; IR (KBr) cm^{-1}: 3352, 1660, 1640; ^{1}H NMR ($CDCl_3$), 300 MHz): δ 7.11-8.72 (m, 15H, ArH and -CH=CH-), δ 13.33 (s, 1H, Ar-OH); MS (ES^+) m/z; 424 (M^+ +1), 425 (M^+ +2).
2. (2E,2'E)-1,1'-(4-hydroxybenzene-1,3-diyl)bis[3-(4-Flurophenyl)prop-2-en-1-one].(2b): Yellow solid; m.p: 190^{0}C; IR (KBr) cm^{-1}: 3281, 1676,1632; ^{1}H NMR($CDCl_3$, 300 MHz): δ 7.11-8.73 (m, 15H, ArH and -CH=CH-), δ 13.34 (s, 1H, Ar-OH); MS(ES^+) m/z; 391 (M^+ +1).
3. (2E,2'E)-1,1'-(4-hydroxybenzene-1,3-diyl)bis[3-(4-methoxyphenyl)prop-2-en-1-one].(2c):Yellow solid; m.p: 144^{0}C; IR (KBr) cm^{-1}: 1657, 1633; ^{1}H NMR ($CDCl_3$,300 MHz): δ 3.89 (s, 3H, OCH_3), δ 3.90 (s, 3H, OCH_3), δ 6.96-8.20 (m, 15H, ArH and -CH=CH-), δ 13.51 (s, 1H, Ar-OH); MS(ES^+) m/z; 415 (M^+ +1), 416 (M^+ +2).

4. (2E,2'E)-1,1'-(4-hydroxybenzene-1,3-diyl)bis[3-(thiophene-2-yl)prop-2-en-1-one].(2d): Brown solid; m.p: 180^0C; IR (KBr) cm^{-1}: 3100, 1656, 1626; ^{1}H NMR ($CDCl_3$,300 MHz): δ 7.11-8.68 (m, 13H, ArH and -CH=CH-), δ 13.34 (s, 1H, Ar-OH); MS(ES^+) m/z; 367 (M^+ +1).
5. (2E,2'E)-1,1'-(4-hydroxybenzene-1,3-diyl)bis[3-(Furn-2-yl)prop-2-en-1-one].(2e): Yellow solid; m.p: 174^0C; IR (KBr) cm^{-1}: 3129, 1666, 1637; ^{1}H NMR ($CDCl_3$,300 MHz): δ 7.21-8.67 (m, 13H, ArH and -CH=CH-), δ 13.42 (s, 1H, Ar-OH); MS(ES^+) m/z; 335(M^+ +1).

3.4. Table 1: Physical data of bis-chalcones(2a-e)

2,4-diacyl phenol	Ar-aldehyde	Product	Yield %	Molecular Formula	Molecular Weight	Melting point ^{o}c
 1a		 2a	80	$C_{24}H_{16}Cl_2O_3$	423	202
 1a		 2b	84	$C_{24}H_{16}F_2O_3$	390	190
 1a		 2c	90	$C_{26}H_{22}O_5$	414	144
 1a		 2d	92	$C_{20}H_{14}O_3S_2$	366	180
 1a		 2e	90	$C_{20}H_{14}O_5$	334	174

4. Acknowledgement:

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