Synthesis Characterization Antioxidant Activity of Some Novel Chalcone Derivatives

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Abstract: Present work deals with synthesizing novel chalcones using 4-aminoacetanilide with different types of benzaldehyde derivatives using Claisen-Schmidt condensation reaction. The synthesized chalcones were purified by recrystallization with ethanol. The characterization of the purified chalcones was made by IR, $^1$H NMR and $^{13}$C NMR data. The compounds were further subjected to antioxidant and antibacterial activities.

Key words: Chalcone, Claisen-Schmidt condensation, Antioxidant and Antibacterial activity.

I. Introduction
Diversely substituted chalcones widely prepared by condensing aromatic aldehyde with 4-aminoacetanilides in presence of base. A classical synthesis of chalcones Claisen-Schmidt condensation reaction to give α,β-unsaturated carbonyl system which undergoes cyclization reaction with hydrazine hydrate and malononitrile. Many chalcones have been widely reported anti-inflammatory[1], antifungal [2,3], antibacterial [4,5], antimalarial [5] and antitumor activities [6] and so on. Most of the reactions of chalcones are the cyclization reaction with hydrazine hydrate, phenyl hydrazine, urea, thiourea, malononitrile it gives heterocyclic derivatives of chalcone. The compounds with pyrazole and cyanopyridine structures are known to possess antimicrobial, antibacterial, antifungal, antitubercular, anti-inflammatory. In the present study some novel pyrazole and cyanopyridine derivatives have been synthesized by the reaction of chalcones. Chalcone and its derivatives have a variety of applications in biological probs, such as analgesic [7,8], antidiabetic [9], anticonvulsant [10], antimicrobial [11], anti-inflammatory [12], antiviral [13] and anticancer activities. [14] In this view, we synthesised chalcone and their derivatives of compound 6 and 7 having good antioxidant activity.

II. Materials And Methods
All the reagents were purchased from Sigma-Aldrich and used as received. Ethanol solvent was supplied by Spectrochem, India. The solvents and reagents used for the synthesis were of reagent grade and were purified by standard methods. TLC was performed using glass plates coated with silica gel-G containing 13% calcium sulphate as binder. Benzene, ethyl acetate, methanol, chloroform were used as developing solvents. A chamber containing iodine vapour was used to located the spots. Melting points were determined by Vision Instrument. $^1$H NMR were taken on Bruker 400MHz using CDCl$_3$ as the solvent with TMS as an internal standard. FTIR spectra were of the synthesized organic compounds were recorded using a Jasco-400 spectrometer instruments.

III. Experimental
Preparation of N-((Z)-4-(3-methyl-2,6-diphenylpiperidin-4-ylideneamino)phenyl)acetamide (3).
Equimolar amount of 3-methyl-2,6-diphenylpiperidin-4-one (0.01 mol) and 4-aminoacetanilide (0.01 mol) in 30ml alcohol, and 10% NaOH solution was refluxed for 8 hours. Then the reaction mixture was cooled and poured into crushed ice. The crude sample was recrystallised from ethanol. Sample Dark brown color powder, Yield 53% , m.p. 110-113°C IR (KBr) (NH-C=O) 1633.41 cm$^{-1}$, (C=N) 1383.68 cm$^{-1}$. 

Preparation of (2E)-N-((Z)-4-(3-methyl-2,6-diphenylpiperidin-4-ylideneamino)phenyl)-3-(benzo[d][1,3]dioxol-5-yl)acrylamide (5).
Equimolar amount of N-((Z)-4-(3-methyl-2,6-diphenylpiperidine-4-ylideneamino)phenyl)acetamide (0.01mol) and piperanol (0.01mol) 25ml of ethanol equipped with stirring bar. 10% solution of NaOH solution are added slowly and refluxed 5 hours. Then it was poured into 400ml water with constant stirring and left overnight in refrigerator. The precipitated obtained was filter washed and air – drying. The crude sample was
recrystallised from ethanol. The Yellow color powder, Yield 70.45%, m.p. 130-133°C; IR (KBr) (NH-C=O) 1672.28 cm⁻¹, (C=C) 1595.13 cm⁻¹, (N-H) 3294.42 cm⁻¹, (C=N) 1333.20 cm⁻¹, (C-N) 1251.50 cm⁻¹.

Preparation of (Z)-N1-(5-(benzo[d][1,3]dioxol-5-yl)-1H-pyrazol-3-yl)-N4-(3-methyl-2,6-diphenylpiperidin-4-ylidene)benzene-1,4-diamine (6).

A mixture of (2E)-N-((Z)-4-(3-methyl-2,6-diphenylpiperidin-4-ylideneamino)phenyl)-3-(benzo[d][1,3]dioxol-5-yl)acrylamide (0.0006mol) in 30ml alcohol, hydrazine hydrate (0.0006mol) and 10% KOH solution was refluxed for 6 hr. Then the reaction mixture was cooled and poured into crushed ice. The product separated out was filtered, washed with and recrystallised from alcohol to give compound 6. The Chocolate Brown color powder, Yield 78.68%, m.p. 138°C. IR, (N-H) 3365.78 cm⁻¹, (C=C) 1566.20 cm⁻¹, (C=N) 1506.20 cm⁻¹. ¹H NMR, 2.7-(s, 3H, Aliphatic CH₃), 6.8-7.9- (m, Ar-H), 11.0 (s, 1H, Pyrazol -NH). ¹³C NMR, 45-(Aliphatic CH₃), 114-(Ar- C), 122-(Pyrazol CH).

Preparation of 4-((Z)-4-(3-methyl-2,6-diphenylpiperidin-4-ylideneamino)phenylamino)-2-amino-6-(benzo[d][1,3]dioxol-5-yl)-1,4-dihydropyridine-3-carbonitrile(7).

A mixture of (2E)-N-(4-(3-methyl-2,6-diphenylpiperidin-4-ylideneamino)phenyl)-3-(4-hydroxyphenyl)acrylamide (0.0007mol) in 30 ml alcohol, malononitrile (0.0007mol) and ammonium acetate was refluxed for 8 hours. Then the reaction mixture was cooled and poured into crushed ice. The product separated out was filtered, washed with and recrystallised from alcohol to give compound. IR, (Ar-NH) 3336.78 cm⁻¹, (C=C) 1566.20 cm⁻¹, (NH) 3273 cm⁻¹, (C=N) 1506.20 cm⁻¹. ¹H NMR, 1.28 (s, 3H, Aliphatic CH₃), 6.8-8.0-(m, Ar-H), 11.0 (s, 2H, Ar- -NH₂), 2.6-8.1H, Ar-NH). ¹³C NMR, 35-(Aliphatic CH₃), 129-(Ar- C), 130-(C=N), 157(Ar-CN).

TABLE 1: Schematic Representation of titled Compounds

![Diagram](image-url)
TABLE 2: Anti-oxidant activity of chalcone derivatives by DPPH scavenging method

<table>
<thead>
<tr>
<th>S.NO</th>
<th>Compound</th>
<th>% of DPPH scavenging activity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>100µgml⁻¹</td>
</tr>
<tr>
<td>1.</td>
<td>Standard (Ascorbic acid)</td>
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</tr>
<tr>
<td>2.</td>
<td>6</td>
<td>56.3</td>
</tr>
<tr>
<td>3.</td>
<td>7</td>
<td>48</td>
</tr>
</tbody>
</table>

IV. Result And Conclusion
The substituted chalcone derivatives of compound exhibits interesting antioxidant activity for various concentration inhibition of compound 6 and 7 show in the Table 2.

Acknowledgement
I have great pleasure in acknowledging my deep indebtedness and gratitude to my authors are thankful to Pondicherry University to carrying out NMR and also thankful to Thiruvalluvar university for recording antioxidant activity.

References


