SYNTHESIS AND CHARACTERIZATION OF SOME NOVEL HETEROCYCLIC CHALCONE DERIVATIVES

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***ABSTRACT*:**

Synthesis of chalcones from acetylthiophene with substituted aromatic aldehyde in dilute ethonolic sodium hydroxide at cool condition reaction followed by Claisen – Schmidt condensation method. In this study of heterocyclic chalcones has generated intensive scientific interest due to their biological and industrial applications. A new series of heterocyclic compounds containing isoxazole, pyrazoles ring systems were prepared from chalcones. The synthesized heterocyclic Chalcone derivatives undergo characterized by melting point, TLC, IR and ^{1}H.NMR and ^{13}C-NMRSpectroscopy.

Keywords: heterocyclic compound, isoxazole, pyrazoles, Chalcone.

1. INTRODUCTION:

Chalcones are belonging to the flavonoid families which are isolated from natural sources. They can be used as an initial compound for synthesis of a lot of compounds. In recent years, Chalcones have found a wide range of applications in the pharmacological activities such as, potential cytotoxic agents, antiviral, anesthetics, mydriatics, antimicrobial, and antipyretic properties etc., [1-10].Chalcones are synthesized by condensing ketones with aromatic aldehydes in the presence of suitable bases [11, 12]. They undergo a variety of chemical reactions and are found to be useful in the synthesis of variety of heterocyclic compounds[13]They are very useful intermediates for the synthesis of five, six and seven-membered heterocyclic compounds. Based on the above observation, we synthesized some new heterocyclic derivatives of Chalcones.

Table 1: Physicochemical characterization data for synthesized heterocyclic Chalcone derivatives:

Compounds	Melting points(^{0}C)	Yield (%)	Molecular Formula	Molecular weight
CPH	160	74	$C_{25}H_{23}N_3O_2$	397
CHA	205	62	$C_{19}H_{18}N_2O_3$	322
CEA	141	82	$C_{25}H_{25}NO_5$	419

Table 2: Synthesized heterocyclic Chalcone derivatives, its molecular name and molecular formula:

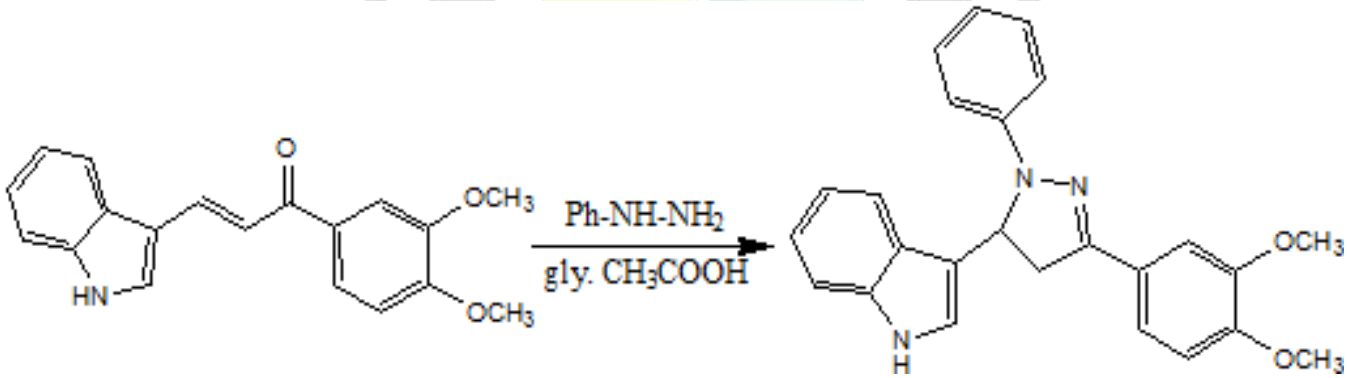
S.NO	COMPOUND	MOLECULAR NAME	MOLECULAR FORMULA
1	CPH	3-[5-(3,4-dimethoxyphenyl)-2-phenyl-3,4-dihydro-2H-pyrazol-3-yl]-1H-indole	$C_{25}H_{23}N_3O_2$
2	CHA	3-[3-(3,4-dimethoxyphenyl)-4,5-dihydro-isoxazol-5yl]-1H-indole	$C_{19}H_{18}N_2O_3$
3	CEA	4-(3,4-dimethoxyphenyl)-6-(1H-indol-3-yl)-2-oxo-cyclohex-3-ene- carboxylic acid ethyl ester	$C_{25}H_{25}NO_5$

2. Material and Methods:

The melting point of the compounds was determined in open capillaries, using Eligo digital melting point apparatus and expressed in degree Celsius and the values were uncorrected. The compound contain IR spectral data were recorded on Shimadzu 8201 spectrophotometer using KBr and the values are expressed in 4000-400 cm^{-1}.The compound contain ^1H and ^{13}C NMR spectral data were recorded on Bruker AV 400 MHz Spectrophotometer using TMS as an internal standard and the values are expressed in δ ppm. All the solvents used were analytical grade. The purity of the compound was checked by TLC using silica gel plates.

2.1.Preparation 1-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone (CPH):

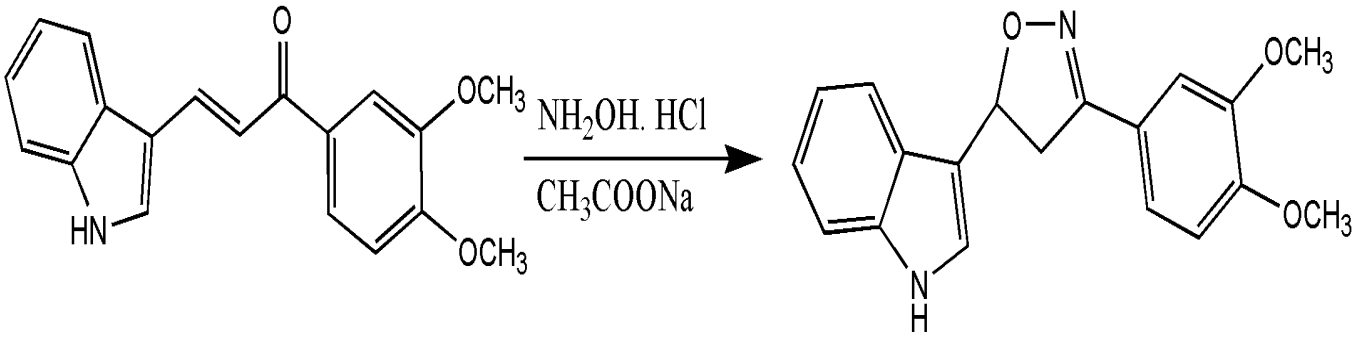
The solution of 1-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone (0.001 mol) was refluxed with phenyl hydrazine (0.001mol) in dry EtOH (30 ml) and catalytic amount of glacial acetic acid for 80^0C for 8h. The progress of the reaction was monitored by TLC. After completion of the reaction, the solvent was removed under reduced pressure, distillation. The residue obtained was recrystallized from EtOH giving brown solid. The yield of the product was 72%. The melting point of the solid was noted as 160^0 C.



1-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone 1-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone

2.2. Preparation of 3-[3-(3, 4-dimethoxyphenyl)-4, 5-dihydro-isoxazol-5yl]-1H-indole (CHA)

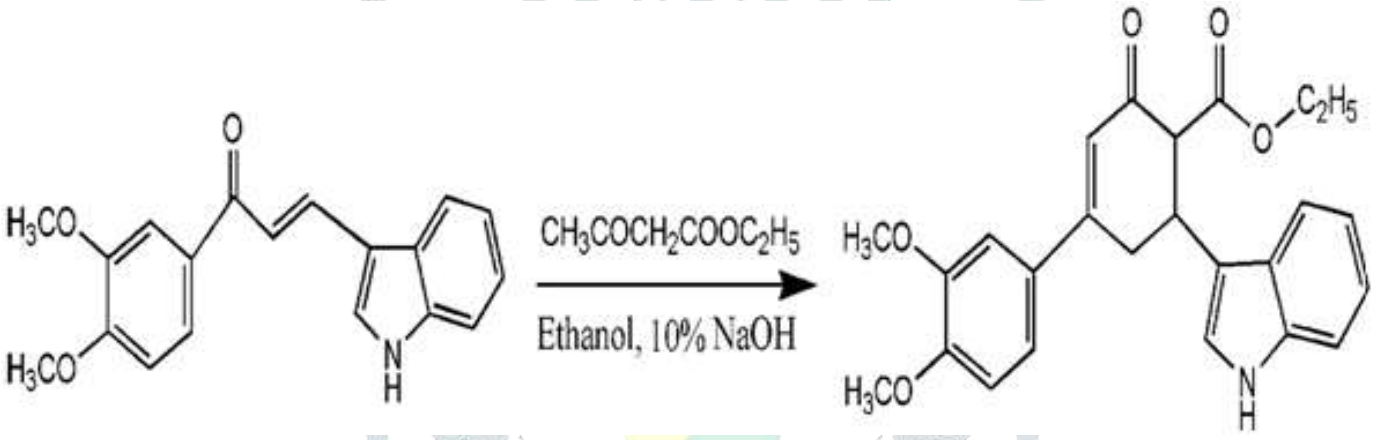
A solution of 1-(3, 4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone (0.001 mol) hydroxylamine hydrochloride (0.001mol) and sodium acetate in ethanol (25ml) was refluxed for 6 h. The mixture was concentrated by distillation out the solvent under reduced pressure and poured into 50ml ice-cold water. The precipitate was collected by filtration and purified by recrystallization from ethanol giving yellow solid. The yield of the product was 62%. The melting point of the solid was noted as 205^0C.



1-(3, 4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone 3-[3-(3, 4-dimethoxyphenyl)-4, 5-dihydro-isoxazol-5yl]-1H-indole (CHA)

2.3. Preparation of 4-(3, 4-dimethoxyphenyl)-6-(1H-indol-3-yl)-2-oxo-cyclohex-3-ene- carboxylic acid ethyl ester (CEA):

A mixture of 1-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone (0.001mol) and ethyl acetoacetate (0.001 mol) in ethanol was refluxed for 6h in 10-15 mL of ethanol in the presence of 10% 10 mL NaOH. The reaction mixture was cooled and poured into 50 mL of ice-cold water. The precipitate was collected by filtration, washed with water and dried in air. The solid obtained was purified by recrystallization from ethanol giving brown solid. The yield of the product was 82%. The melting point of the solid was noted as 141^0C.



1-(3, 4-dimethoxyphenyl)-3-(1H-indol-3-yl)-propenone 4-(3, 4-dimethoxyphenyl)-6-(1H-indol-3-yl)-2-oxo-cyclohex-3-ene-carboxylic acid ethyl ester (CEA)

3. Result and Discussion:

3.1 Spectral data:

The results are obtained from various spectral data are results discussed below.

3-[5-(3, 4-dimethoxyphenyl)-2-phenyl-3,4-dihydro-2H-pyrazol-3-yl]-1H-indole (CPH):

IR (KBr) cm-1: 3404.36(NHStr), 1575.84(NHbend), 1388.75(C-NStr), 1442.75(Aromatic C=CStr), 2926(Aromatic C-HStr), 883-638(C-Hdef),1242(C-OStr) .^{1}H NMR (DMSO)ppm: 7-8(Aromatic), 3.3(C-OCH3), 2.5(CH2) and 9.9(N-H).^{13}HNMR (DMSO)ppm: 118-137(Aromatic),), 38(CH2),and 40(C-OCH3).

3-[3-(3, 4-dimethoxyphenyl)-4, 5-dihydro-isoxazol-5yl]-1H-indole (CHA):

IR (KBr) cm-1: 3170.97(N-HStr), 1442.75(Ar C=C Str), 1633.71(C=NStr), 2924(C-HStr), 1525.69(N-H bend), 1388(Aromatic) and 640-848(C-Hdef). ^{1}H NMR (DMSO) ppm: 7-8(Aromatic), 3.2(C-OCH3), 2.5(CH2) and 9.9(N-H). ^{13}HNMR (DMSO) ppm: 111-138(Aromatic), 39(CH2) and 40(C-OCH3).

4-(3, 4-dimethoxyphenyl)-6-(1H-indol-3-yl)-2-oxo-cyclohex-3-ene- carboxylic acid ethyl ester (CEA):

IR (KBr) cm-1: 3387(N-HStr), 1448(N-HBend), 1340(C-NStr), 1517(C=CStr), 2926(C-HStr), 642-862(C-Hdef), 1637(C=NStr), 1H NMR (DMSO)ppm: 71-8.3(Aromatic), 3.3(C-OCH3), 2.5(CH2) and 11(N-H).13HNMR(DMSO) ppm: 120-137(Aromatic), 155(C=N), 39(CH2) and 40(C-OCH3).

4. ***CONCLUSION***:

The newly heterocyclic Chalcone derivatives were synthesized and characterized by physical method (i.e., melting point, molecular weight, molecular formula) and the compounds were characterized by spectral data (IR, 1H-NMR &13C-NMR,). The Spectral result confirmed that the product has formed Among the heterocyclic chalcones derivatives 4-(3, 4-dimethoxyphenyl)-6-(1H-indol-3-yl)-2-oxo-cyclohex-3-ene- carboxylic acid ethyl ester gives high percentage of yield.

5. ***ACKNOWLEDGEMEN***T:

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