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SYNTHESIS AND CHARACTERIZATION OF NEW CHALCONES OF 4-FLUORO-3-METHYL ACETOPHENONE

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Abstract:
In the present investigation it has been considered worthwhile to synthesize some new chalcones from 4-fluoro-3-methyl-acetophenone and different aromatic aldehydes by Claisen-Schimdt condensation. The synthesized compounds were characterized by means of Chemical analysis, IR and ^{1}H NMR spectroscopic data.

Key words: *Flavonoids, Chalcone, IR and ^{1}H NMR spectroscopy.*

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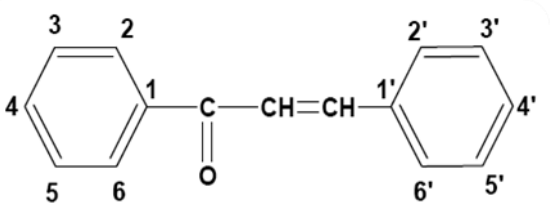
INTRODUCTION:

The natural compounds are generally divided into various classes on the basis of their molecular structures including chalcones, flavones, flavanones and anthocyanidins. Flavonoids have several pharmacological benefits (e.g. anticancer, anti-inflammatory, anti-allergic, etc.) and are considered as effective antioxidants, metal chelators and free radical scavengers. Natural and synthetic flavonoids are therefore of considerable interest in the development of novel therapeutic agents for various diseases and are generally believed to be non-toxic compounds since they are widely distributed in the human diet. Chalcones (*trans*-1,3-diaryl-2-propen-1-ones), which belong to the flavanoid family are precursors of open chain flavonoids and isoflavonoids, and are abundant in edible plants. They are also key precursors in the synthesis of many biologically important heterocycles such as benzothiazepine, pyrazolines, 1,4-diketones, and flavones.`

Chemistry of chalcones:



Chalcones are α,β-unsaturated ketones consisting of two aromatic rings with substituents. They contain the keto-ethylenic group (-CO-CH=CH-). These are coloured compounds because of the presence of the chromophore (-CO-CH=CH-). Two aromatic rings are interconnected by a highly electrophonic three carbon α, β unsaturated carbonyl system that assumes linear or nearly planar structure. Chalcones possess conjugated double bonds and a completely delocalized π-electron system on both benzene rings. Chalcones exist as either E or Z isomers. E isomer is the most stable form.

Modification of chalcone structures by substitution with a prenyl side chain also affects their biological activities. Prenylation as protein post-translational modification results in higher protein lipophilicity and targets the modified protein to cell membrane

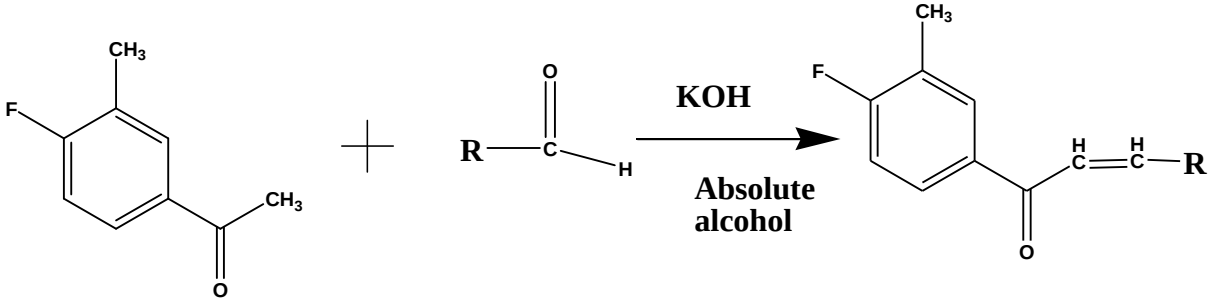
EXPERIMENT:

All the reagents and chemicals used were of laboratory grade. Completion of the reaction was monitored by thin layer chromatography (TLC) using E-Merk 0.25 mm silica gel plates. Visualization was accomplished with UV light (256nm). All the solvents were distilled and dried using appropriate drying agents before use. Melting points were determined on ANALAB melting point apparatus and were uncorrected. All the ^{1}H NMR spectra were recorded in DMSO-d_6 solvent. Chemical shifts are reported on AVANCE 300 MHz and INNOVA 500 MHz relative to TMS internal standard on the δ (ppm)-scale. The IR spectra were recorded on SCHIMADZU FT-IR SPECTROPHOTOMETER by using 1% Potasium bromide discs.

Procedure for Synthesis of chalcones of 4-fluoro-3-methyl acetophenone:

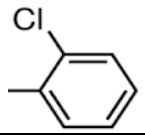
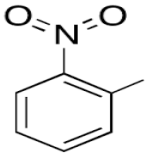
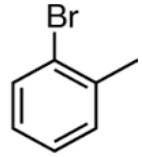
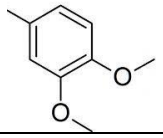
A mixture of 4-flouro-3-methyl acetophenone (0.01 mol) and aromatic aldehydes(0.01 mol) was stirred in ethanol (30ml) and then aqueous potassium hydroxide (40%) was added to it dropwise with constant stirring. The mixture was kept overnight at room temperature and then poured into crushed ice. It was acidify with 1:1 dilute hydrochloric acid and water till a precipitate is obtained, then it was filtered. The so obtained compound was identified and where ever necessary compound was recrystallized and the pure compound was also obtained by separating with the help of column chromatography.

SCHEME



R = 2-Cl Phenyl, 2-Nitro phenyl, 2-Br phenyl, 3,4-dimethoxy phenyl,

RESULTS AND DISCUSSION:

NAME OF THE COMPOUND	R=	MELTING POINT	%YIELD	ELEMENTAL ANALYSIS(%)
A_1		160^{0}C	79	C-69.95, H-4.40 F-6.92, Cl-12.92 O-5.82
A_2		170^{0}C	85	C- 67.36,H- 4.24 F-6.66, N- 4.91 O-16.83
A_3		150^{0}C	92	C- 60.21,H-3.97 F-5.91, Br-25.04 O-5.01
A_4		200^{0}C	73	C-71.99,H-5.71 F-6.33,O-15.98

Interpreted ^{1}HNMR & IR spectra data of synthesized compound:

(2E)-3(2-chlorophenyl)-1-(4-fluoro-3-methylphenyl)prop-2-en-1-one:(A_1)

^{1}HNMR(DMSO)(δppm) C_6H_5: 2.360, C=0 : 3.363, CH-F: 3.932, Ar-H : 7.885, 7.876,7.870. -C=C- : 7.757, 7.730, 7.587, CH-Cl : 2.363 IR -CH-F:1179 nm, -C=O:1717 nm, p-disub:880 nm, -CH-Cl:820 nm tri substituted benzene :1620-1550nm, Adjacent H's:880-790nm

(2E)-1-(4-fluoro-3-methylphenyl)-3-(3-nitrophenyl)prop-2-en-1-one: (A_2)

^{1}HNMR(DMSO)(δppm) C_6H_5: 2.383, C=0 : 3.363, CH-F: 4.932,Ar-H : 7.886, 7.879,7.851. -C=C- : 7.645,7,625,7.605 IR -CH-F:1152 nm, -C=O:1720 nm, p-disub:870 nm, N=0:1524nm tri substituted benzene:1620-1550nm, Adjacent H's:880-790nm

(2E)-3(3bromophenyl)-1-(4-fluoro-3-methylphenyl)prop-2-en-1-one: (A_3)

^{1}HNMR(DMSO)(δppm) C_6H_5: 2.359, C=0 : 3.338, CH-F: 4.504, Ar-H : 7.856, 7.837,7.819. -C=C- : 7.561,7.541,7.401, C-Br : 3.48 IR -CH-F:1175 nm, -C=O:1725 nm, -CH-Br:560 nm, p-disub:872 nm, tri substituted benzene :1620-1550nm, Adjacent H's :880-790nm

(2E)-3-(3,4dimethoxyphenyl)-1-(4-fluoro-3-methylphenyl)prop-2-en-1-one: (A_4)

^{1}HNMR(DMSO)(δppm) C_6H_5: 2.358, C=0 : 3.953, CH-F: 3.932, CH-O-R: 3.60, Ar-H : 7.868, 7.855,7.848., -C=C- : 7.757,7.758,7.736. IR -CH-F:1150 nm, -C=O:1720 nm, p-disub:890 nm,C-O-C:1124 nm,tri substituted benzene :1620-1550nm, Adjacent H's:880-790nm

CONCLUSION:

The synthesized compounds were characterized by TLC, melting point, IR spectroscopy, elemental analysis and NMR spectroscopy. The result obtained from this study confirmed that the product has formed. Henceforth viewing these characteristic properties more compounds can be synthesized and subjected to evaluation of pharmacological activity. These chalcone derivatives may have variety of biological activities viz' anti-inflamatory analgesic antitubercular, leishmanicidal, anticancer activity, etc and may be a pave way for the synthesis and characterization of some new chalcone derivatives**.**

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