ORIGINAL RESEARCH PAPER

Medical Science

SYNTHESES AND CHARACTERIZATION OF SOME SUBSTITUTED CHALCONES

KEY WORDS: Chalcones, 2-hydroxy-4,5-dimethylacetophenone, aromatic aldehydes.

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ABSTRACT

The present work deals with some novel substituted chalcones prepared from 2-hydroxy-4,5-dimethyl acetophenone and aromatic aldehydes like benzaldehyde, 4-hydroxy benzaldehyde, 4-nitro benzaldehyde and 3-chloro benzaldehyde in alkaline medium at room temperature which yields corresponding chalcones. The structure of synthesized chalcones were elucidated by spectroscopic techniques like IR, Mass, Physical Constant, 1H-NMR, UV and Elemental Analysis.

Introduction:

Chalcones-one of the major classes of natural products with widespread distribution in vegetables, fruits, tea and spices have been great interest for their interesting pharmacological activities[1]. Chemically, they consist of open chain flavonoids in which the two aromatic ring are joined by a three carbon α, β unsaturated carbonyl system[2]. Synthetic chalcones are commonly synthesized with the reaction of acetophenone and benzaldehyde via a Claisen-Schmidt condensation reaction. This reaction is catalysed by bases and acids under homogeneous conditions[3].The IUPAC name of chalcones is 1,3-diphenyl-2-propene-1-one, in this it possess conjugate double bond and a delocalized π electron on both the benzene ring[4]. Chalcones are very reactive compounds due to presence of ketoethylenic group and therefore undergoes a variety of chemical reactions and used[5] for the synthesis of several heterocyclic compounds. The chalcones shows a wide range of biological activities such as antiviral[6], antibacterical[7], antiinflammatory[8], antifungal[9], anticancer[10], analgesic[11], antiulcer[12], antimalarial[13], antihelmintic[14] and antihyperglycemic[15].

Materials and methods:

Materials:

The chemicals 2-hydroxy-4,5-dimethyl acetophenone, benzaldehyde, 4-hydroxy benzaldehyde, 4-nitrobenzaldehyde, 3-chlorobenzaldehyde, hydrochloric acid, sodium hydroxide, ethyl alcohol, ethyl acetate, n-hexane, methanol, chloroform, DMSO etc. used during the work were of AR Grade. Doubled distilled water was used throughout the work.

Experimental:

Synthesis of 2-Hydroxy-4,5-dimethyl acetophenone:

A mixture of anhydrous aluminium chloride and 3,4-dimethyl phenyl acetate was heated for 3 hours and then cooled at room temperature (Fig.1). The $AlCl_3$ decomposed by adding mixture of conc. HCl and ice cold water. The solid product was then filtered, washed with double distilled water and dried. The purity was checked by TLC in solvent pet ether : ethyl acetate (80:20). Its melting point is 62°C-67°C.

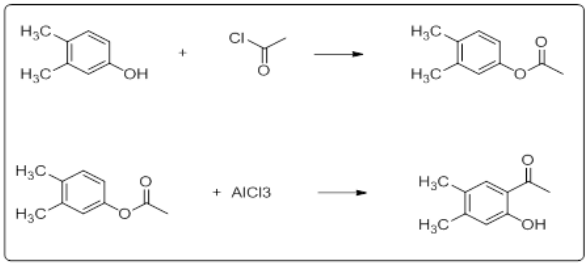

Fig. 1: Synthesis of 2-Hydroxy-4,5-dimethyl acetophenone

Synthesis of substituted 2-Hydroxy-4,5-dimethyl chalcone:

General procedure

An appropriate equal molar quantity of 2-hydroxy-4,5-dimethylacetophenone (0.01mol), various substituted benzaldehydes (0.01 mol), 0.5g of sodium hydroxide and 20 ml of ethanol were warmed in a 50 ml conical flask and shaken occasionally. The mixture stirred till completion of the reaction (progress of reaction checked by TLC). The mixture acidified with 10% hydrochloric acid then filtered by using suction pump, washed with cold water and crystallized by ethanol. The yield of the yellow coloured product was more than 60% (Fig.2).

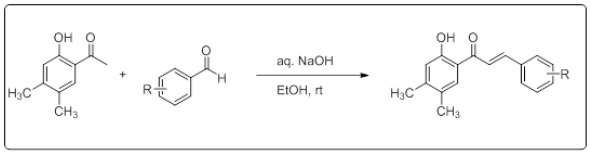

Fig. 2: Schematic synthesis of substituted 2-hydroxy-4,5-dimethyl chalcone (R=H, 4-OH, 4-$NO_2$and 3-chloro)

Result and Discussions:

1. 3-phenyl-1-(2-hydroxy-4, 5,-dimethyl phenyl)-prop-2-en-1-one: (1a)

To a stirred mixture of 2-hydroxy-4,5-dimethyl (0.01mol) and benzaldehyde in ethanol (20ml), sodium hydroxide (0.5g) was added and treated as in general procedure to give (1a).

Molecular Formula: $C_{17}H_{16}O_2$; Formula Weight: 252; MS m/z(%) 252 [M^+]; Melting point: 150°C; Yield: 58%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR (KBr) in cm^{-1}: 2921(-OH), 1640(C=O), 1503(C=C), 1221(C-O), 2858 (Ar C-H); ^{1}H-NMR (400MHz) in $CDCl_3$: δ 4.97 (s, 1H, OH, D_2O Exchangeable), 2.13 (s, 3H, CH_3), 2.30 (s, 2H, CH_2), 7.40(s, 5H, Ar-H), 6.597(s, 2H, =CH); UV (Bands λ_{max} in nm) in methanol: 420.

2. 3-(4-Nitrophenyl)-1-(2-hydroxy-4, 5,-dimethyl phenyl)-prop-2-en-1-one: (1b)

To a stirred mixture of 2-hydroxy-4,5-dimethyl(0.01mol) and p-nitro benzaldehyde in ethanol(20ml), sodium hydroxide (0.5g) was added and treated as in general procedure to give (1b)

Molecular Formula: $C_{17}H_{15}O_4N$; Formula Weight: 297; MS m/z(%) 296 [M^+]; Melting point: 181°C; yield: 60%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR (KBr) in cm^{-1}:3390(-OH), 1615(C=O), 1513(C=C), 1190(C-O)2921 (Ar C-H) ; ^{1}H-NMR (400MHz in $CDCl_3$): δ 4.92(s, 1H, OH, D_2O Exchangeable), 2.16(s, 3H, CH_3), 2.32(s, 2H, CH_2), 7.33-7.76(d, 4H, Ar-H), 6.56(s, 2H, =CH); UV (Bands λ_{max} in nm) in methanol: 410.

3. 3-(4-Hydroxyphenyl)-1-(2-hydroxy-4, 5,-dimethyl phenyl)-prop-2-en-1-one: (1c)

To a stirred mixture of 2-hydroxy-4,5-dimethyl (0.01mol) and p-hydroxyl benzaldehyde in ethanol (20ml), sodium hydroxide (0.5g) was added and treated as in general procedure to give (1c).

Molecular Formula: $C_{17}H_{16}O_3$; Formula Weight: 268; MS m/z(%) 268[M^+]; Melting point:105°C; Yield: 51%; Colour: Brown

powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR (KBr) in cm^{-1}: 3242(OH), 1639(C=O), 1588(C=C), 1221(C-O),2987 (Ar C-H) ; ^{1}H-NMR (400MHz in $CDCl_3$): δ 4.96 (s, 1H, OH, D_2O Exchangeable), 2.22(s, 3H, CH_3), 2.59(s, 2H, CH_2), 7.57-7.60(d, 4H, Ar-H), 6.58-6.70(s, 2H, =CH); UV (Bands λ_{max} in nm) in methanol: 380.

4. 3-(3-Chlorophenyl)-1-(2-hydroxy-4, 5,-dimethyl phenyl)-prop-2-en-1-one: (1d)

To a stirred mixture of 2-hydroxy-4,5-dimethyl (0.01mol) and m-chloro benzaldehyde in ethanol (20ml), sodium hydroxide (0.5g) was added and treated as in general procedure to give (1d).

Molecular Formula: $C_{17}H_{15}O_2Cl$; Formula Weight: 286.5; MS m/z(%) 287[M^+]; Melting point:110°C; Yield: 56%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR (KBr) in cm^{-1}: 3527(OH), 1635(C=O), 1567(C=C), 1185(C-O) 2920 (Ar C-H); ^{1}H-NMR (400MHz in $CDCl_3$): δ 4.91(s, 1H, OH, D_2O Exchangeable), 1.90 (s, 3H, CH_3), 2.29(s, 2H, CH_2), 7.32-7.54(m, 4H, Ar-H), 6.712(s, 2H, =CH); UV (Bands λ_{max} in nm) in methanol: 420.

Conclusions:

The synthesized Chalcone compounds were characterized by T.L.C., Mass, IR, NMR Spectroscopy and Elemental Analysis. The results obtained from this study confirmed that the product has formed. Further, viewing these properties many different complexes can be synthesized and subjected to pharmacological studies. These Chalcone derivative complexes may have catalytic property as well as a variety of biological activities.

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