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Synthesis and characterization of some 2-hydroxy-4, 5-dimethyl substituted chalcones

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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 2-Chorobenzaldehyde, 3-Bromo- benzaldehyde, 4-Fluorobenzaldehyde and 4-Cyanobenzaldehyde in alkaline medium at room temperature which yields as corresponding chalcones. The structure of synthesized chalcones was elucidated by elemental analysis and spectroscopic techniques like IR, 1H-NMR, UV and Mass.

KEYWORDS: Chalcones, 2-hydroxy-4, 5-dimethyl acetophenone and substituted aldehydes.

Introduction:

Chalcones are the products of aromatic aldehydes with simple or substituted acetophenones obtained1 in the alkaline medium. Chalcones are aromatic compounds in which two aromatic rings are linked by three carbon α,β-unsaturated carbonyl system. The IUPAC name of chalcone is 1,3-diphenyl-1,2-propene-1-ene, in this it possess conjugate double bond and a delocalized π electron on both the benzene ring. Chalcones are very reactive compounds due to presence of ketoethylenic group and therefore undergoes variety of chemical reactions and used2 for the synthesis of several heterocyclic compounds. The chalcones show wide range of pharmaceutical activities such as antimicrobial3, anticancer4,5, antiinflamatory6, antimalerial7, antioxidant, antitumor8, antileishmanial9, analgesic10, antiulcerative11, immune modulatory12, antihyperglycemic13 and antiviral14.

Materials:

The chemicals 2-Hydroxy-4,5-dimethyl acetophenone, 2-Chlorobenzaldehyde, 3-Bromo- benzaldehyde, 4-Fluorobenzaldehyde, 4-Cyanobenzaldehyde, hydrochloric acid, sodium hydroxide, ethyl alcohol, ethyl acetate, n-hexane, methanol, chloroform, DMSO etc used in this work were of AR Grade.

Experimental:

Preparation of 2'-Hydroxy-4'5'-dimethyl acetophenone:

The 3, 4-Dimethyl phenyl acetate was cooled in ice and to it added anhydrous aluminum chloride lot wise. Then reaction mixture was heated to 160-180°C for 4-5 hrs. Then the reaction mixture cooled to room temperature. The AlCl3 decomposed by adding ice cold water and concentrated HCl to it. The solid product obtained15 was filtered and washed with demineralised water till the pH of filtrate is neutral and then dried it safely. The purity was checked by TLC in solvent ethyl acetate:n-hexane(80:20). Its melting point is 69-73°C and yield-63%.

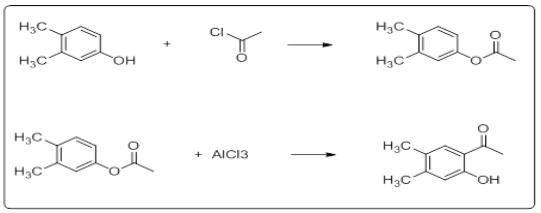

Figure-1: Reaction Scheme and mechanism for 2-hydroxy-4, 5-dimethyl acetophenone

Preparation of Substituted Chalcone:

The substituted chalcones prepared by stirring the equimolar concentration mixture of 2-Hydroxy-4, 5-dimethyl acetophenone (0.01 mol) and substituted aromatic benzaldehyde (0.01 mol) in 20 ml ethanol for 3 hrs in presence of 50% NaOH. The mixture stirred till completion of reaction (progress of reaction checked by TLC). The crude mixture poured into ice water then acidified the product with 10% hydrochloric acid. The yellow coloured compound formed was filtered, washed with water and dried. The compounds recrystallised from absolute ethanol.

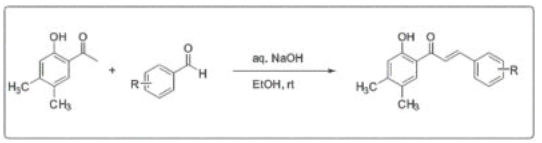

Figure-2: Reaction Scheme and mechanism for chalcones (R= 2-Cl, 3-Br, 4-F and 4-CN)

Results:

1. 3-(2-Chlorophenyl)-1-(2-hydroxy-4, 5,-dimethyl phenyl)-prop-2-en-1-one: Molecular Formula: $C_{17}H_{15}O_2Cl$; Formula Weight: 286.75; MS m/z(%) 287.05[M^+]; Melting point:150°C; yield:68%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR(KBr) in cm^{-1}: 3050(-OH), 1641(C=O), 1570-1460(C=C), 1180(C-O); ^{1}H-NMR(400MHz) in $CDCl_3$: δ 12.60 (s, 1H, OH, D_2O Exchangeable), 2.196 (s, 3H, CH_3), 2.176 (s, 3H, CH_3), 6.6-7.5(m, 6H, Ar-H), 7.6-8.5(m, 2H, =CH); UV (Bands λmax in nm) in methanol: 204, 310.

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

Molecular Formula: $C_{17}H_{15}O_2Br$; Formula Weight: 331.20; MS m/z(%) 330.75[M^+]; Melting point:125°C; yield: 75%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR(KBr) in cm^{-1}: 3100(-OH), 1647(C=O), 1570-1450(C=C), 1180(C-O); ^{1}H-NMR(400MHz in $CDCl_3$): δ 12.52(s, 1H, OH, D_2O Exchangeable), 2.233(s, 3H, CH_3), 2.500(s, 3H, CH_3), 6.6-7.5(m, 6H, Ar-H), 7.6-8.4(m, 2H, =CH); UV(Bands λmax in nm) in methanol: 204, 310.

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

Molecular Formula:$C_{17}H_{15}O_2F$; Formula Weight: 270.30; MS m/z(%) 270.96[M^+]; Melting point:130°C; yield: 60%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO,

ethyl acetate; IR(KBr) in cm^{-1}: 3100(OH),1653(C=O),1560-1460(C=C),1190(C-O); ^{1}H-NMR(400MHz in $CDCl_3$): δ 12.66 (s, 1H, OH, D_2O Exchangeable), 2.259(s, 3H, CH_3), 2.285(s, 3H, CH_3), 6.6-7.5(m, 6H, Ar-H),7.6-8.5(m, 2H, =CH); UV(Bands λmax in nm) in methanol: 202, 315.

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

Molecular Formula: $C_{17}H_{15}O_2F$; Formula Weight: 270.30; MS m/z(%) 270.96[M^+]; Melting point:130°C; yield: 60%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR(KBr) in cm^{-1}: 3100(OH), 1653(C=O), 1560-1460(C=C), 1190(C-O); ^{1}H-NMR(400MHz in $CDCl_3$): δ 12.66 (s, 1H, OH, D_2O Exchangeable), 2.259(s, 3H, CH_3), 2.285(s, 3H, CH_3), 6.6-7.5(m, 6H, Ar-H), 7.6-8.5(m, 2H, =CH); UV(Bands λmax in nm) in methanol: 202, 315.

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

Molecular Formula: $C_{18}H_{15}O_2N$; Formula Weight: 277.32; MS m/z(%) 278.19[M^+]; Melting point:190°C; yield: 65%; Colour: Yellow powder; Solubility: methanol, ethanol, chloroform, DMSO, ethyl acetate; IR(KBr) in cm^{-1}: 3100(OH), 1641(C=O), 1580-1450(C=C), 1188(C-O); ^{1}H-NMR(400MHz in $CDCl_3$): δ 12.87(s, 1H, OH, D_2O Exchangeable), 2.172(s, 3H, CH_3), 2.295(s, 3H, CH_3), 6.6-7.5(m, 6H, Ar-H), 7.6-8.5(m, 2H, =CH); UV(Bands λmax in nm) in methanol: 202, 314.

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