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Synthesis And Biological Screening Of Newly Design Chalcones Without Solvent

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Abstract:
The purposes of green chemistry play an important role in development of synthetic strategy. The main criterion of green synthetic chemistry is to reduce the use of hazardous chemicals and make simple reaction procedure while design new synthetic molecules. Many solvent-free synthetic routes such as Grignard reactions such as Aldol condensations, Dieckmann condensations, Friedel-Craft, witting reactions and other had been synthesized by researchers. Most of these reactions are carried out at room temperature in absence of solvent. Present study also deals with synthesis of newly chalcones by grinding the reactant by using only a mortar that is non-conventional technique without solvent. The newly synthesized chalcones without solvent was obtained in good yield and its formation is confirmed by physical and spectral characteristics. Present study confirms that it was one of the green approaches for the synthesis of chalcones by using without solvent. Further compounds shows better zone of inhibition tested againts. S.aureus and E.coli.

Introduction:

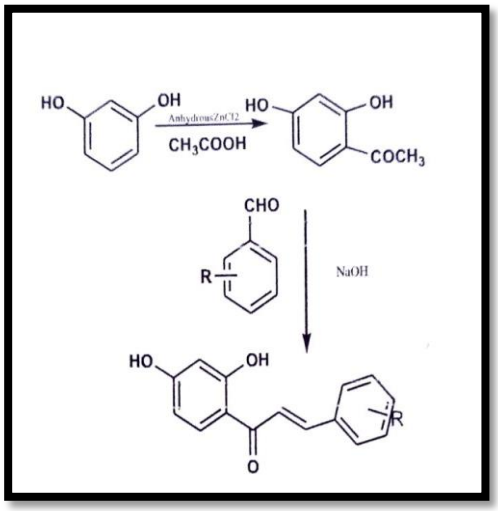
Green chemistry is the need of the day and hence it was planned to synthesize chalcone in an ecofriendly way without using solvents and technology has shifted more toward environmental and sustainable resources and progress. Green chemistry is a concept for sustainability which is a set of principal that minimize or get ride of the utilization or generation of hazardous option in the design, manufacturing and application for chemical product[1]. One of the significant feature of the ultrasound approach compare with traditional methods are in a higher yield, milder condition, lesser reaction time, improve reaction rates, formation of pure compound, easier manipulation and a role in a waste minimization and energy protection[2].

Chalcones are well known intermediates for synthesizing various heterocyclic compounds[3] .The chalcones has been synthesized by using the Claisen–Schmidt condensation under both acidic(HCl–EtOH) and basic catalysis (K_2CO_3) or KOH, $Ba(OH)_2$, $LiOH.2H_2O$ by using NaOH[4]. The several reagents and conditions such as basic alumina, zinc chloride, Lewis acid such as dry HCl gas, BF_3, $AlCl_3$,strong alkali with phase transfer catalysts had been used for the synthesis of chalcones[4]. Chalcones are unsaturated ketone containing the reactive keloethylenic group CO-CH=CH.

Many researchers Synthesized chalcones Duha Adnan etal Synthesized 2-hydroxychalcones by 2-hydroxyacetophenones and aryl aldehyde[5], Daniel R. Pallero Synthesized 1,3-bis(4-methylphenyl)-2-propen-1-oneby4-

Chlorobenzaldehyde and 4'-bromoacetophenone[6]. Nataliej O'Neil et.al, synthesize 4′-bromo-4-chlorochalcone by 4-chlorobenzaldehyde and 4-bromoactophenone[7]. Hery Suwito etal synthesized hydroxyl chalcones by hydroxy-acetophenone and benzaldehydes and study bioactivity[8]. Dhankhar Synthesized (2E)-1,3-diphenylprop-2-en-1-one by acetophenone and benzaldehyde and study biological activities[9]. Chalcones represent an important class of natural compound with variety of biological activity such as anti-microbial[9], anti-inflammatory[8], tyrosinekinase[9], inhibitors properties antiplatelet[3], anti-Ulcerative[4], anti-leisnmanal[5].

Actual Plan of Work:



R: 4-Br, 4-Cl, 4-N(CH$_3$)$_2$

Experimental Work:

Synthesis of 1-(2,4-dihydroxyphenyl)ethanon/(2,4dihydroxyacetophenone)(C1)

2,4dihydroxyacetophenone was synthesized by reported method. Synthesis was carried out by dissolving freshly fused and powdered zinc chloride (0.24mole) in 32 ml of glacial acetic acid by heating in sand bath. Dry resorcinol (0.2mole) was added with constant stirring at 140^{o}C.The solution was heated until it just begins to boil and kept for 20 minute at 15^{o}C.Dilute HCL (1:1) was added to the mixture and the solution was cooled to 5^{o}C.the separated product was filtrated and washed with dilute HCl. The product was recrystallised from hot water.

Synthesisof(E)-1-(2,4-dihydroxyphenyl)-3-(4-(dimethylamino)phenyl)prop-2-en-1-one. (C2)

P-dimethyl amino Benzaldehyde (0.01 mole), and 2,4dihydroxyacetophenone (0.01 mole) and 0.5g of NaOH was grind at room temperature for 30 minutes in a mortar and pestle. Ice-cold water (60ml) was added to the reaction mixture and neutralize with dilute HCL and solid precipitate was filtered and washed with water. The product was recrystallized from ethanol.

synthesis of (E)-3-(4-chlorophenyl)-1-(2,4-dihydroxyphenyl)prop-2-en-1-one. (C3)

P-chlorobenzaldehyde (0.01 mole) and 2,4dihydroxyacetophenone (0.01 mole) and 0.5g of NaOH was grind at room temperature for 30 minutes in a mortar and pestle. Ice-cold water (60ml) was added to the reaction mixture and neutralize with

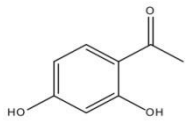
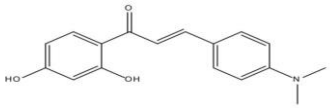
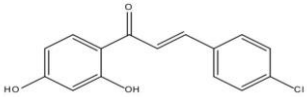
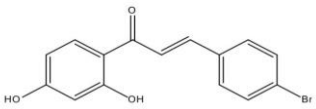
dilute HCL and solid precipitatewas filtered and washed with water.The product was recrystallisedfrom ethanol.

synthesis of (E)-3-(4-bromophenyl)-1-(2,4-dihydroxyphenyl)prop-2-en-1-one. (C4)

P-BromoBenzaldehyde (0.01 mole) and 2,4dihydroxyacetophenone (0.01 mole) and 0.5g of NaOH was grind at room temperaturefor 30 minutes in a mortar and pestle. Ice-cold water (60ml) was added to The reaction mixture and neutralize with dilute HCL and solid precipitate was filtered and washed with water.The product was recrystallised from ethanol.

Interpretation of Result:

Table 1: Physical Parameters

SR.NO	STRUCTURE	MOLECULAR FORMULA	MELTING POINT	SOLUBILITY
C1		$C_8H_8O_3$	145°C	DMSO
C2.		$C_{17}H_{17}NO_3$	65°C	DMSO
C3.		$C_{15}H_{11}ClO_3$	100°C	DMSO
C4.		$C_{15}H_{11}BrO_3$	105°C	DMSO

Spectral Analysis Of Compound C2:

IR

C2: $C_{17}H_{17}NO_3$,M.P. 65°C soluble in DMSO,

IR(OH Str) ,2859.71 cm^{-1},(C=O),1600 Cm^{-1} , (>C=C<) , 1567.50 Cm^{-1} , (Ar-CH) , 3000 Cm^{-1}, (C-O), 1029.65 Cm^{-1} .

NMR

C2:- $C_{17}H_{17}NO_3$

^{1}H NMR (DMSO-d6) : δ(ppm)6.255(s, 1H –CH proton on aromatic ring), 12.614 (s ,broad ,1H –OH attached to aromatic ring), 6.4 (d ,2H ,-CH of aromatic ring), 10.649(s,broad1H,-OH attached of aromatic ring), 7.769 (d,2H,-CH=CH-) ,8.043 (d ,2H ,-CH of aromatic),2.5 (s ,6H -CH_3 attached to aromatic ring -$N(CH_3)_2$),8.306 (d ,2H –CH).

Biological Screening of Antimicrobial Activity (Agar Well Diffusion Method):

Bacterial strains like staphylococcus aureus and Escherichia coli, were used to find out the anti-microbial activity of chalcones by using agar well diffusion technique. A hole having diameter 6-7mm is punched with sterile cork borrer over the

agar surface. Further, 20 of Licorice solution (01mg/ml) was introduced into the well. The control and experimental plates were incubated at 37°C for 24 hrs.

Observation:

Anti-microbial activity was tested in vitro against staphylococcus aureus and Escherichia coli culture.

1)ForS.aureus:

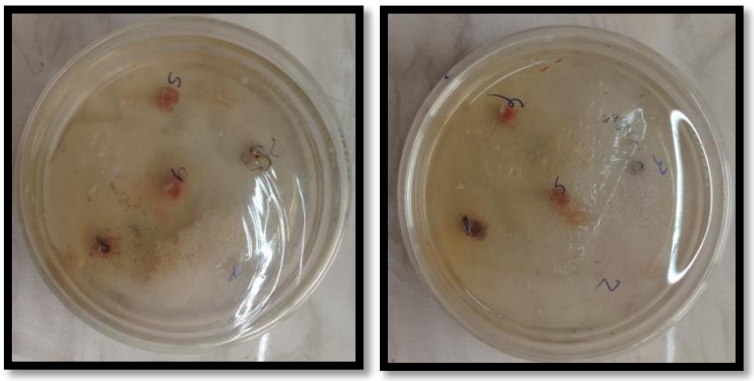
Compound	Concentration (mg/ml)	Bacterial culture	Zone of inhibition
C1	01mg/ml	S.aureus	12mm
C2	01mg/ml	S.aureus	14mm
C3	01mg/ml	S.aureus	16mm

2.For E.coli

Compound	Concentration (mg/ml)	Bacterial culture	Zone of inhibition
C1	01mg/ml	E.coli	13mm
C2	01mg/ml	E.coli	17mm
C3	01mg/ml	E.coli	12mm

Table2: showing one of inhibition for s.aureas and E.coli showing zone of inhibition

Biological Screening:



Result and Discussion:

The present study consult with the three newly design chalcones derived from substituted benzaldehydes with 2,4-dihydroxyacetophenon which involved the use of NaOH as an alkaline condition and solvent free atmosphere was carried out. Synthesized compound C2 (chalcones) having α ,β unsaturated carbonyl group Spectral characteristic usually appear as a prominent bands of IR at 1718.31cm^{-1}(C=Ostr) and (C=C str) at 1591.13 cm^{-1}.band at 2959.71 cm^{-1}due to (OH- str) and IR band for C2 is (C=O str) at 1600 cm^{-1} and (C=Cstr) at 1567.50 cm-1, band at 2859.71 cm^{-1} due to (OH-str), other region of IR absorption bands for C2 is due to substituents present on the rings. NMR study C2 exhibited singlet at δ 2.5ppm due to six protons of CH_3 groups attached to nitrogen. 2H showing broad singlet at δ 12.614 and δ 10.649 due to -OH groups which is attached to aromatic ring .2H showing doublet at δ 7.769 due to -CH=CH- Confirms the

formation of Chalcones. Further remaining 7H showing signal at δ 6.255, δ 6.4, δ 8.043 and δ 8.306 due to proton attached to aromatic ring. All signals due to proton of aromatic ring and aliphatic appeared at excepted region and hence confirm the formation of new chalcones having the molecular formulae **$C_{17}H_{17}NO_3$(C2)**. Antimicrobial activity was tested in vitro against staphylococcus aureus and Escherichia coli culture, for each concentration 01mg/ml respectively. Zone of inhibition as shown in table no 2. Compound **C2 and C3** shows better activity at concentration 10mg/ml for both the culture and proved that can be used for medicinal testing .

Conclusion:

The newly synthesized chalcones without solvent was obtained in good yield and its formation is confirmed by physical and spectral characteristics. Present study confirms that it was one of the green approaches for the synthesis of chalcones by using without solvent. Further compounds shows better zone of inhibition tested againts. S.aureus and E.coli.

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