"The Synthesis of Chalcone by Grinding Method''

Submitted in partial fulfillment of the requirements for the degree of Bachelor of Pharmacy

by

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Academic Year: 2019-2020

CERTIFICATE

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This is to certify that the project entitled, **"The synthesis of chalcone by grinding method"** is a bonafidework of **1. Shaikh Asif Ali Murutuja Ali Shajaha Begum (15PH40) 2. Chaudhary Mohd. Irshad Abdul Wadood Aktarunnisa (16PH13) 3. Siddique Nasreen MD.Usman Palimunnisa (15PH53) 4. Sayyed Tarannum Zehra Kamar Abbas Mariyam Begum (16PH44)** submitted for the appreciation of the degree of Bachelor of Pharmacy in Department of Pharmaceutical chemistry.

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ABSTRACT

The synthesis of different chalcones using substituted aldehydes and ketones with catalyst KOH, by grinding method.

Keywords: Chalcones, Aldehydes, Ketones, KOH, Grinding

Approval for Bachelor of Pharmacy

This project entitled, **"The synthesis of chalcone by grinding method"** by **1. Shaikh Asif Ali Murutuja Ali Shajaha Begum (15PH40) 2. Chaudhary Mohd. Irshad Abdul Wadood Aktarunnisa (16PH13) 3. Siddique Nasreen MD.Usman Palimunnisa (15PH53) 4. Sayyed Tarannum Zehra Kamar Abbas Mariyam Begum (16PH44)** is approved for the degree of Bachelor of Pharmacy in Department of Pharmaceutical Chemistry.

Examiners

1.....

2.....

Supervisors

1.....

2.....

Declaration

I declare that this written submission represents my ideas in my own words and where others ideas or words have been included; I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will be cause for disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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1.Shaikh Asif Ali Murutuja Ali Shajaha Begum (15PH40)

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Index

Keywords and glossary

1.	γ-oxo-α	γ-oxo α-amino acids
2.	**^{0}C**	degree centigrade
3.	NAOH	Sodium hydroxide
4.	TDP	Thermal depolarization
5.	Na_2CO_3	Sodium carbonate
6.	PEG400	Polyethlene glycol 400
7.	$ZrCL_4$	Zirconium chloride
8.	KOH	Potassium chloride
9.	$KF\text{-}AL_2O_3$	Potassium fluoride and aluminium oxide
10.	EtOH	Ethyl alcohol
11.	TLC	Thin layer chromatography
12.	CH_3COCH_3	Dimethyl ketone
13.	$C_6H_5COCH_3$	Aromatic ketone
14.	IR	Infrared radiation.

INTRODUCTION

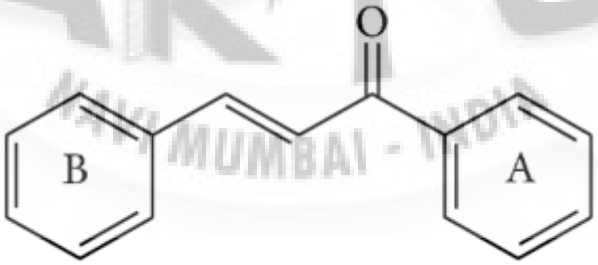
The chemistry of chalcones has generated intensive scientific studies throughout the world. Especially interest has been focused on the synthesis and biodynamic activities of chalcones. The name "Chalcones" was given by Kostanecki and Tambor. [1] Chalcone is an aromatic ketone and an enone that forms the central core for a variety of important biological compounds, which are known collectively as chalcones or chalconoids. Alternative names for chalcone include benzylidene acetophenone, phenyl styryl ketone, benzalacetophenone, *β*-phenylacrylophenone, *γ*-oxo-*α*, *γ*-diphenyl-*α*-propylene, and *α*-phenyl-*β*-benzoylethylene. Structure of chlcones contains, two aromatic rings are linked by an aliphatic three carbon chain. Chalcone bears a very good synthon so that variety of novel heterocycles with good pharmaceutical profile can be designed. Chalcones are unsaturated ketone containing the reactive ketoethylenic group –CO-CH=CH-. Chalcones (trans-1, 3-diaryl-2- propen-1-ones) are α, β-unsaturated ketones consisting of two aromatic rings (ring A and B) having different substituents. This rings are interconnected by a highly electrophonic 3 carbon α, β-unsaturated carbonyl system so it is assume that chalcones have linear or nearly planar structure. [2-5]

Chalcones can be prepared by an aldol condensation between benzaldehyde and acetophenone in the presence of sodium hydroxide as a catalyst.[6]

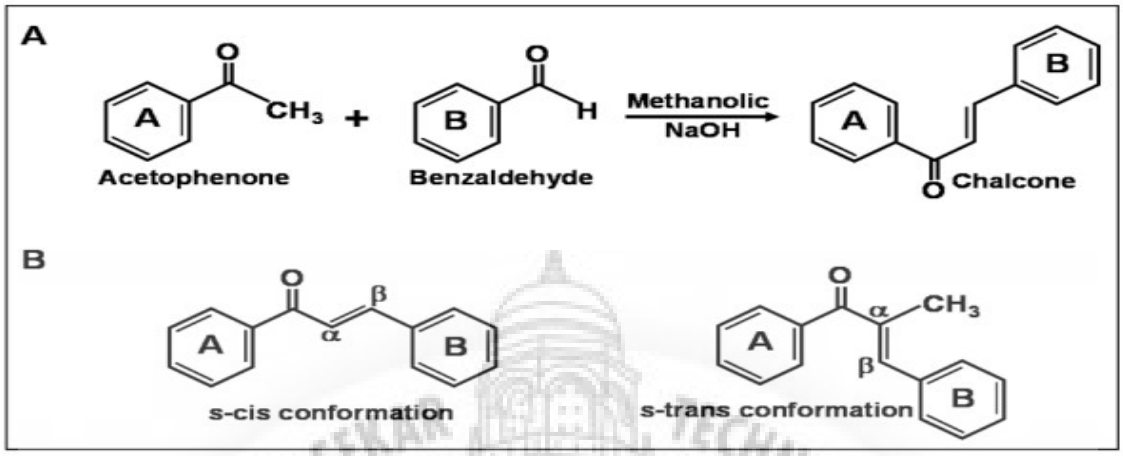
This reaction can be carried out without any solvent as a solid-state reaction. The reaction between substituted benzaldehydes and acetophenones can be used as an example of green chemistry in undergraduate education. [7] In a study investigating green syntheses, chalcones were synthesized from the same starting materials in high-temperature water (200 to 350 °C).

Substituted chalcones were also synthesised by piperidine-mediated condensation to avoid side reactions such as multiple condensations, polymerizations, and rearrangement.

The general structure of chalcone



Synthesis of chalcone



Uses of the chalcones: Nowadays, several chalcones are used for treatment of viral disorders, cardiovascular diseases, parasitic infections, pain, gastritis, and stomach **cancer**, as well as like food additives and cosmetic formulation ingredients. The various use of chalcones are

1. Anti HIV
2. Anti microbial
3. Anti fungal
4. Anti viral
5. Anti oxidant
6. Anti malarial
7. Anti inflammatory
8. Anti cancer
9. Anti convulsant.[3]

Importance of chalcones are:-

(1) They have close relationship with flavones, aurones, tetralones and aziridines.

(2) Chalcones and their derivatives find application as artificial sweeteners, scintillator, polymerization catalyst, fluorescent whitening agent, organic brightening agent, stabilizer againhalcones are flexible mst heat, visible light, ultraviolet light and aging

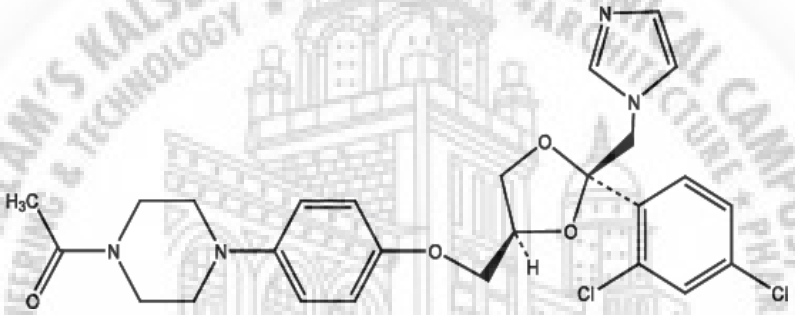
(3) 3, 2', 4', 6'-tetrahydroxy-4-propoxy-dihydrochalcone-4-β'-neohesperdoside has been used as synthetic sweetener and is 2200 times sweeter than glucose.

(4) They contain a keto-ethylenic group and are therefore reactive towards several reagents e.g. (a) phenyl hydrazine, (b) 2-amino thiophenol etc.

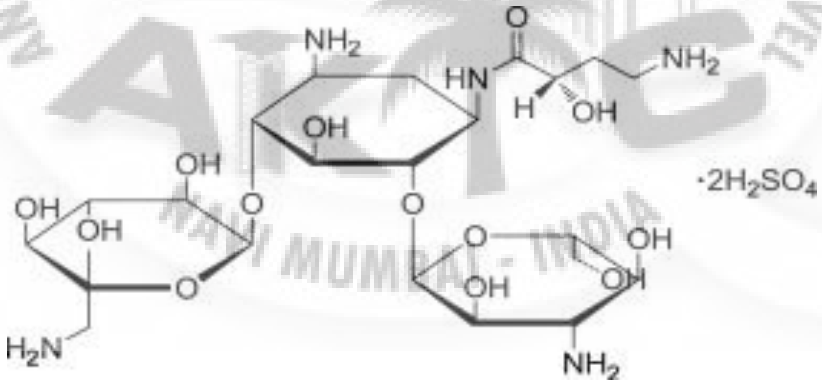
Advantages of chalcones are:-

1. Chalcones are used as natural resources.
2. Chalcones are found in many fruits (apple, tomato) and vegetables (potato).
3. Chalcones are flexible molecules, capable of existence in various conformation.
4. Chalcones can be used as an initial compound for synthesis of other compound.
5. Many compounds are known to possess antibacterial, antifungal activity such as azole antifungals and anti biotics etc.
6. Some example of such compound are ketoconazle, amikacin, etc.

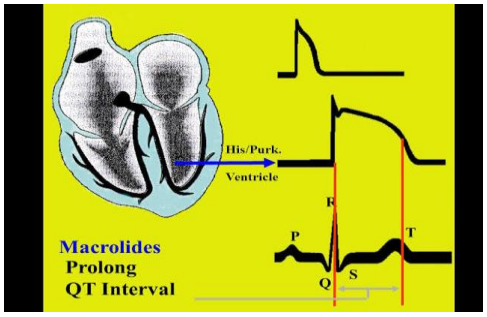
Ketoconazol



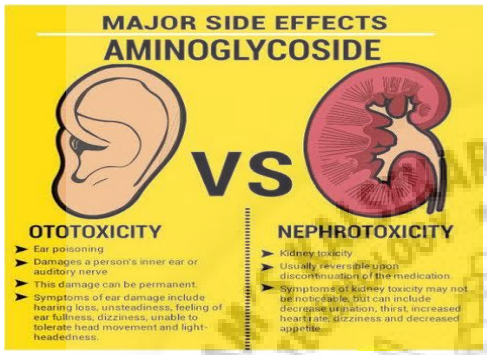
Amikacin



This compound although useful have serious side effect such as:



Macrolides can **prolong** the QT and QTc interval and cause cardiac arrhythmias, including TdP, ventricular tachycardia, and ventricular fibrillation, via their effect on the potassium channel.

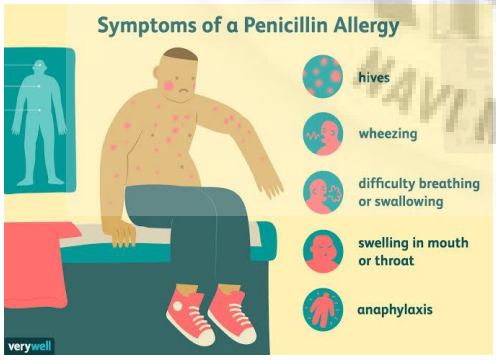


it's having side effect of:

- Loss of hearing
- Ringinging or buzzing in the ears
- Increased thirst
- Skin rashes or itchiness
- Unusual drowsiness
- weakness

side effect of tertracycline.

- Nausea
- Vomiting
- Swollen tongue
- Sores and etc

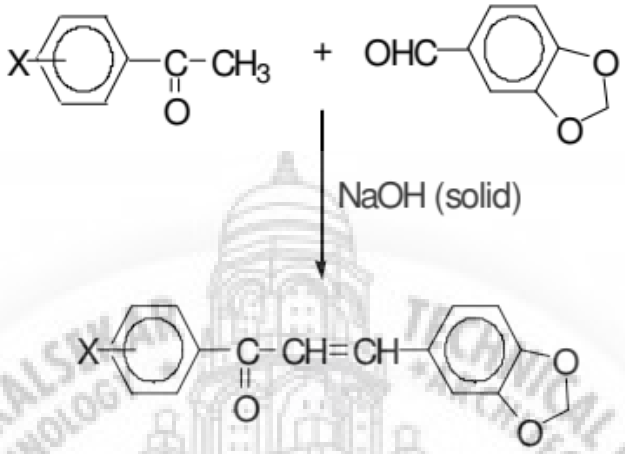


Penicillin side effect.

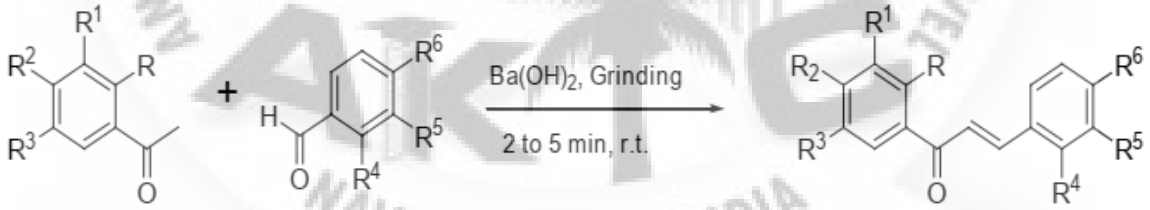
- Hives
- Wheezing.

REVIEW OF LITERATURE

1. Rajendra K. Saini et. al.[11] reported solvent free synthesis of six chalcones was carried out by grinding the piperanal and the acetophenone (unsubstituted, 4-methyl, 4-methoxy, 4-bromo, 4-nitro, 3-chloro) in the presence of solid sodium hydroxide with a mortar and pestle in year 2005.



2. S.kumar et. al.[12] reported simple, rapid, efficient and environmentally benign procedure for the synthesis of chalcones has been achieved by grinding aryl aldehydes and acetophenones with anhydrous barium hydroxide (C-200) in the absence of any solvent. The use of organic solvent for extraction of compound is also avoided. This present method is highly useful for the synthesis of 2'-hydroxy chalcones, required intermediates for the synthesis of flavanoids in year 2008



3. S. Zangade et. al.[13] reported an efficient and operationally simple reaction is shown between substituted 2-acetyl-1-naphthol/2-acetyl-1-naphthol and different substituted benzaldehydes in presence of base afford chalcones in quantitative yield using grindstone technique in year 2011.

4. Syed Nasir Abbas Bukhari et.al.[14] reported lot of methods and schemes have been reported for the synthesis of these compounds. Amongst all, Aldol condensation and Claisen-Schmidt condensation still grasp high up position. Other distinguished techniques include Suzuki reaction, Witting reaction, Friedel-Crafts acylation with cinnamoyl chloride, Photo-Fries rearrangement of phenyl cinnamates etc. These inventive techniques utilize various catalysts and reagents including SOCl2 natural phosphate, lithium nitrate, amino grafted zeolites, zinc oxide, water, Na2CO3, PEG400, silicasulfuric acid, ZrCl4 and ionic liquid etc. technique in year 2012.

5. Pravina Piste [15]reported A facile synthesis of some novel 4-(Sub Phenyl)-1-phenyl 2-propen-1-ones (I-IX) has been achieved by the condensation of Aromatic and aliphatic ketone with various substituted aromatic aldehydes through intramolecular aldol condensation using solid NaOH by using grinding technique in year 2014.

6. Nora M. Rateb et.al.[16] reported An improved Claisen–Schmidt condensation reaction of methyl ketones and aromatic aldehydes can be achieved by grinding at room temperature in the absence of solvents in year 2009.

7. Ji-tai li et.al.[17] reported Claisen–Schmidt condensation of acetophenone with aromatic aldehydes catalyzed by pulverized KOH and KF–Al2O3 results chalcones in 52–97% and 83–98% yields respectively in alcoholic solvent under ultrasound irradiation in year 2002.

8. Zsuzsanna rozmer et.al. [18] reported the various naturally occurring chalcone compounds which have been isolated from different plants in year 2014.

9. Isa karaman et.al. [19] reported a series of chalcone derivatives, 3 –60 , were prepared by Claisen –Schmidt condensation of appropriate acetophenones and 2-furyl methyl ketones with appropriate aromatic aldehydes, furfural, and thiophene-2-carbaldehyde in an aqueous solution of NaOH and EtOH at room temperature in year 2010.

10. Solankee et.al. [20] reported 2,4–Bis-ethylamino-6-[4'-{3"-(substitutedphenyl/2'"-furanyl)-2"-propenone-1"-yl} phenyl amino]-s-triazine 5a-d have been prepared by treating ketone 4 with different substituted aromatic and heterocyclic aldehydes in the presence of alkali in the year 2009.

11. Parvesh singh et.al. [21] reported Chalcones represent key structural motif in the plethora of biologically active molecules including synthetic and natural products in the year 2014.

12. Oruganthi sai kaushik et.al. [22] reported one of the most important classes of organic compounds present in nature or synthesized in the laboratory in heterocyclic systems. In the treatment of commonly occurring diseases and biological activities these compounds possess an array in the year 2016.

13. Goncalo p. rosa et.al. [23] reported Chalcones are naturally occurring α,β-unsaturated carbonyl compounds owning a wide range of pharmaceutical and industrial applications in the year 2017.

14. Anjanee solankee et.al. [24] reported Chalcones (E1-E5) have been prepared by the reaction between ketone (D) and different substituted aromatic aldehydes in the year 2009.

15. Markandewar et.al. [25] reported A series of some novel 2'Hydroxy Chalcone using inert Silica Gel (Mesh 230-400) by Microwave irradiation is reported. These newly synthesized compounds were screened for their antioxidant potential using DPPH screening method in the year 2013.

16. Christina noring hammond et.al. [26] reported This three-week guided-inquiry project involves an aldol-dehydration synthesis of a chalcone chosen from a set of nine, followed by a catalytic transfer hydrogenation reaction using ammonium formate and Pd/C in the year 2009.

AIM AND OBJECTIVES

From the literature survey it was found that large number of chalcones having pharmacological importantance was synthesised. It was also observed that some chalcones have major contribution in antibacterial agents.

1. To synthesize chalcone with simple technique without solvent
2. To characterize the synthesized chalcone using modern spectral technique.
3. To perform the pharmacological screening of synthesized compounds.

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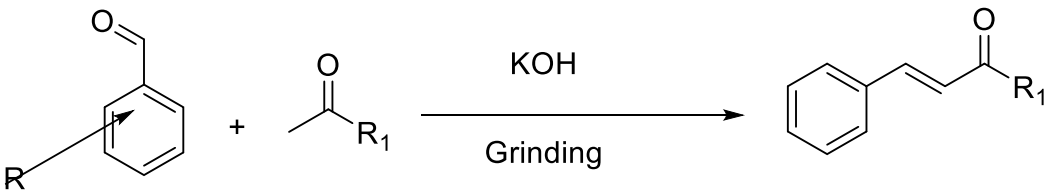
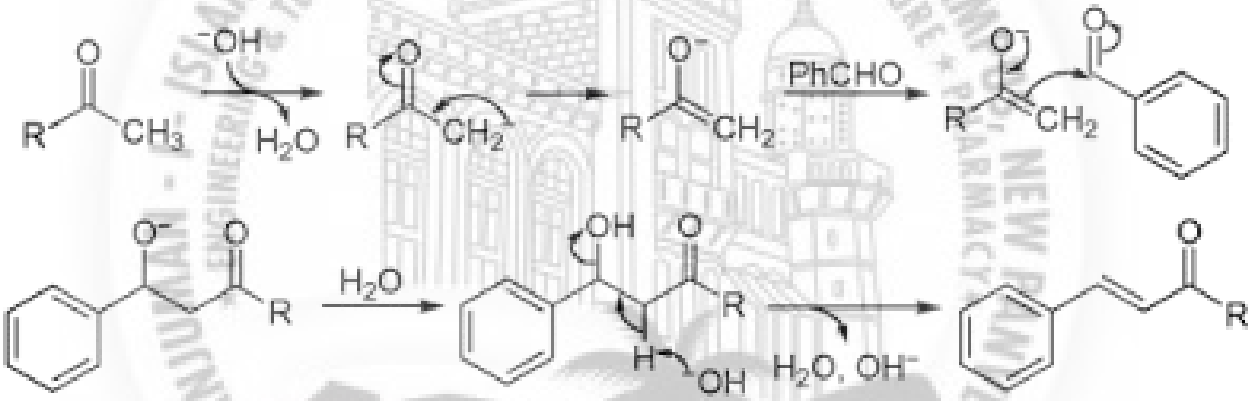
EXPERIMENTAL WORK

Route and scheme of Synthesis



Aldehyde Ketone Chalcone

General mechanism of reaction



Experimental

Different aldehydes (0.01 mole), substituted ketone (0.01 mole) and 0.56 g solid KOH is taken in a small mortar. Grinded this mixture thoroughly for 5-10 min. The mixture become solid and kept it overnight. Then washed with cold water and filtered to obtain the crude product. Recrystallize product by using the solvent as an ethanol, dried and weight. Procedure for TLC mobile phase: ethyle acetate, hexane, ether, choloroform. (Different ratio). On TLC there are 3 components for travel, which is first and second is the reactant and the third one is the product. And checked under the U.V.for confirmation of the reaction.

Mobile phase:

1. 10% Ethyle acetate + 90% Hexane, ratio (3 : 7) for 10 ml.
2. Ether + chloroform, ratio (6 : 4) for 10 ml.

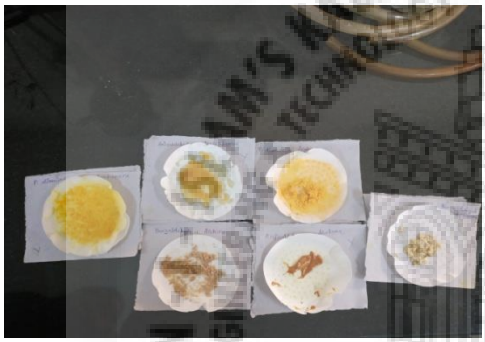
Table No. 1 The different types of R and R1

COMPOUNDS	REACTANT	R	R1
Compound 1	Para-dimethylaminobenzaldehyde + acetone	-N(CH_3)2	CH_3COCH_3
Compound 2	Benzaldehyde + acetone	-	CH_3COCH_3
Compound 3	Benzaldehyde + Acetophenone	-	$C_6H_5COCH_3$
Compound 4	Anisaldehyde + Acetophenone	-OCH3	$C_6H_5COCH_3$
Compound 5	Anisaldehyde + Acetone	-OCH3	CH_3COCH_3
Compound 6	Para-dimethylaminobenzaldehyde + acetophenone	-N(CH_3)2	$C_6H_5COCH_3$

Experimental procedure photos.

Grinding and cover with foil paper and Kept it for over night.

Washed with cold water and filter by Vacume

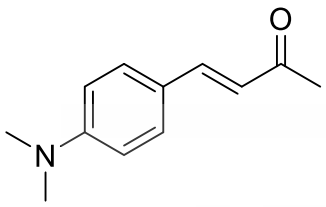
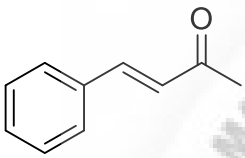
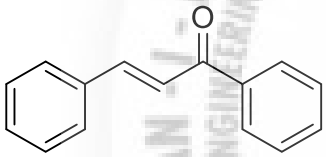
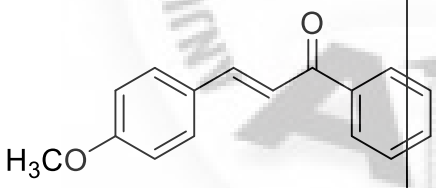
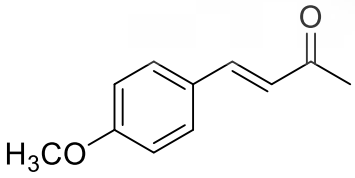


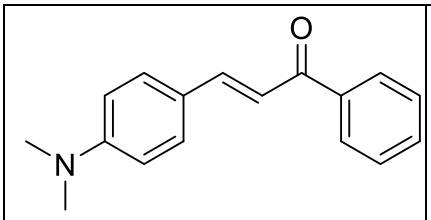
Obtained the crude product.

Recrystallize the product by using the Solvent as an ethanol. Dried and weight.

RESULTS

Table No. 2 Experimental

Compound No.	Molecular formula & Mol. wt.	Reactant	MP	% yield
Compound 1 	$C_{12}H_{15}NO$ 189.258	Paradimethylaminobenzaldehyde + Acetone	92^0C	till lockdown
Compound 2 	$C_{10}H_{10}O$ 146.11	Benzaldehyde + Acetone		78%
Compound 3 	$C_{15}H_{12}O$ 208.26	Benzaldehyde + Acetophenone		till lockdown
Compound 4 	$C_{16}H_{14}O_2$ 238.28	Anisaldehyde + Acetophenone	180^0C	till lockdown
Compound 5 	$C_{11}H_{12}O_2$ 176.21	Anisaldehyde + Acetone	142^0C	till lockdown
Compound 6	$C_{17}H_{17}NO$ 251.32	Paradimethylaminobenzaldehyde + Acetophenone	90^0C	till lockdown

Analytical

	Item	Value
2	Sample name	PHC4453
3	Sample ID	Solid
4	Option	
5	Intensity Mode	%Transmittance
6	Apodization	Happ-Genzel
9	No. of Scans	16
10	Resolution	4 cm-1

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area	Comment
1	698.23	71.09	14.57	711.73	680.87	706.835	250.750	
2	761.88	83.29	12.00	802.39	727.16	695.617	361.421	
3	817.82	92.46	5.33	825.53	806.25	83.189	44.322	
4	837.11	92.21	5.83	858.32	825.53	103.519	53.222	
5	883.40	98.00	2.80	896.90	864.11	17.505	40.086	
6	952.84	82.77	10.18	964.41	927.76	322.221	114.185	
7	1070.49	90.87	2.39	1083.99	1055.06	224.827	32.177	
8	1105.21	88.73	2.97	1118.71	1083.99	339.440	53.695	
9	1130.29	88.64	3.54	1145.72	1118.71	252.855	44.610	
10	1163.08	87.88	6.01	1184.29	1145.72	329.016	96.998	
11	1311.59	93.74	2.26	1325.10	1301.95	118.380	26.653	
12	1338.60	90.22	6.02	1352.10	1325.10	166.366	64.618	
13	1384.89	85.94	3.89	1390.68	1352.10	368.429	77.089	
14	1448.54	93.49	3.21	1454.33	1440.83	60.778	18.810	
15	1494.83	95.10	5.97	1504.48	1483.26	43.032	65.082	
16	1552.70	89.40	3.49	1566.20	1546.91	158.389	33.767	
17	1595.13	86.02	7.57	1612.49	1581.63	304.575	98.920	
18	1653.00	90.21	1.90	1658.78	1649.14	82.063	7.591	

19	2972.31	88.36	9.33	3003.17	2953.02	295.420	188.836	
20	3396.64	90.33	0.39	3402.43	3392.79	91.040	2.151	

Discussion and conclusion

Chalcones were synthesized by treating aldehyde with a ketone using base KOH. Initially, benzaldehyde was treated with acetone which gives good yield. Further, substituted aldehyde were treated with ketone to obtain various chalcones.

Changing the base/catalyst from NaOH to KOH showed a major increase in yield of the product. Performing TLC of compound with ether and chloroform showed better separation than with ethyl acetate and hexane.

Characterisation of the compound through IR showed the confirmation of product.

Also, the compounds were synthesized by grinding method which contributed towards a low cost for our project as well as provides an advantage over the use of expensive instruments.

FUTURE SCOPE

In future, it can be used for precursor of many compounds which give therapeutics effect. By products are not hazardous to the environment so green chemistry is maintained.

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APPENDIX

1. The basic **aldehyde** and **ketone** has been taken and its **catalyst base**.

2. we change the catalyst, so basically that catalyst was **NaOH** and we changed into **KOH**.

3. We tried a substituted aldehyde and substituted ketone with the different catalyst that is KOH.

4. The substituted aldehyde such as:

   - **Anisaldehyde**
   - **Paradimethylaminobenzaldehyde**
   - **Benzaldehyde**
   - **4-hydroxybenzaldehyde**

5. The substituted ketones such as:

   - **Acetone**
   - **Acetophenone**

6. Basically our project was based on chalcones, i.e aldehyde + ketone+catalyst base and we tried to increase the yield by the simple grinding method.

7. We got the positive result as we followed the TLC, melting point and IR.

8. The uses of the chalcones are antibacterial, antiviral and important role in the NSAID.

Synthesis of chalcones by Grinding method

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ABSTRACT

The synthesis of different chalcones using substituted aldehydes and ketones with catalyst KOH, by grinding method.

Keywords: Chalcones, Aldehydes, Ketones, KOH, Grinding