

**SYNTHESIS AND CHARACTERISATION OF 1,5-DIPHENYL 2,4 PENTADIENONE****C. Anuba¹ and T.F. Abbs Fen Reji²**¹Department of Chemistry and Research Centre, Scott Christian College, Nagercoil.²Department of Chemistry and Research Centre, Nesamony Memorial Christian College, Marthandam.**ABSTRACT**

A heterocyclic chalcone derivative 1,5-diphenyl 2,4 pentadienone were carefully prepared using the substrate acetophenone and cinnamaldehyde in the presence of three different catalyst. Ethanol was used as solvent. The synthesized chalcone can be expected to have biological property due to the presence of two aromatic ring bound by an α,β -unsaturated carbonyl group. Three different catalysts were used to carry out the reaction and the yield of the products can be compared. Also the heterocyclic compound 1,5-diphenyl 2,4 pentadienone can be characterised using UV-Vis spectrum and fluorescence emission spectrum.

KEYWORDS: Heterocyclic chalcone, UV-Vis spectrum, Fluorescence Emission Spectrum, Cinnamaldehyde, Acetophenone

INTRODUCTION

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. Benzylideneacetophenone is the parent member of the chalcone series. The alternative name given to chalcone are phenyl styryl ketone, benzalacetophenone, β -phenylacrylophenone, γ -oxo- α,γ -diphenyl- α -propylene and α -phenyl- β -benzoylethylene.

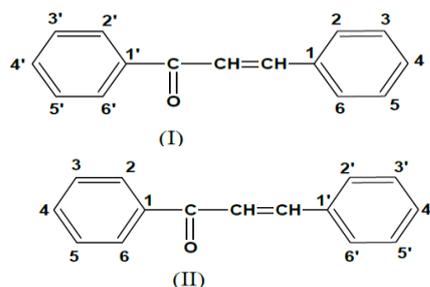
Chalcones are α,β -unsaturated ketone containing the reactive ketoethylenic group – CO-CH=CH-. These are coloured compounds because of the presence of the chromophore - CO-CH=CH-, which depends in the presence of other auxochromes.

The chalcones (1, 3-diaryl-2-propenones) and their derivatives are important intermediates in organic synthesis. They serve as starting material for the synthesis of variety of heterocyclic compounds which are of

physiological importance. Due to the presence of enone functionality in chalcone moiety confers biological activity upon it, like anti-inflammatory, antifungal, antioxidant, antimalarial, antituberculosis, analgesic, anti HIV and antitumor activities.

Chalcones are natural oxygen-containing aromatic compounds that belong to the group of flavonoids with an open γ -pyrone cycle connecting two benzene fragments. In nature they occur mainly in plants and are also present in food products of plant origin such as propolis, wine, beer, fruit juices and tea. The low content of chalcones in natural raw material and their proved wide-range physiological activities are the reason for the increased interest towards the preparation of new synthetic chalcones with desired properties for application in medicine. Chalcones have two absorption maxima at 280 nm and 340 nm.

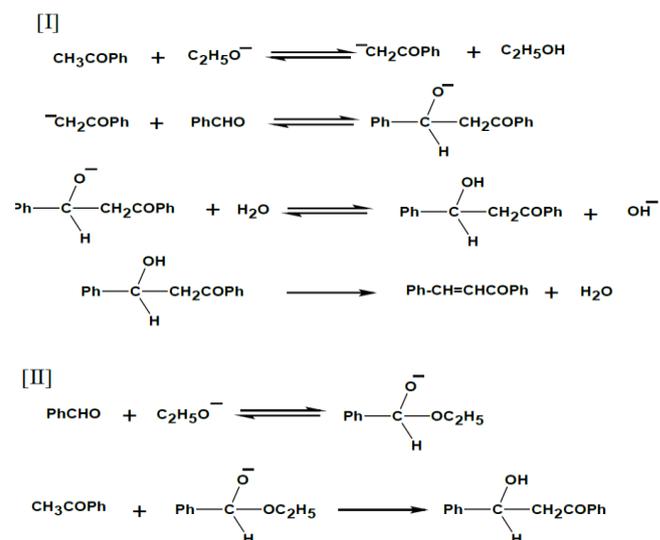
Different methods of nomenclatures for chalcone were suggested at different times. The following pattern has been adopted by "Chemical Abstracts" published by American chemical society.



Mechanism of chalcone formation:

Kinetic studies have been reported for the base-catalyzed formation of chalcone and

its derivatives. Two alternative mechanisms have been advanced for the reaction of benzaldehyde with acetophenone in the presence of a basic catalyst.



Importance of chalcones:

(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, and organic brightening agent, stabilizer against 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.

Biological importance:

The presence of unsaturated carbonyl system of chalcone makes it biologically active. They have shown antibacterial activity against *S. aureus*, *E. coli*, *C. albicans*, *T. utilis*, *S. sake*, *W. anomala* and some other organisms.

Devaux, Nuhrich and Dargelossynthesized some nitrofuryl chalcones and tested for their antibacterial activity. Among all those derivatives the most efficient was (XVII), which inhibited *Staphylococcus landon* at concentration 1µg/ml.

1,3-Diphenyl propenones (chalcones) are well known to exhibit a broad spectrum of biological activities. These are main precursors in the biosynthesis of flavonoids that are abundant in edible plants. They have been reported to possess various pharmacological activities like anticancer, antimalaria, antiplasmodial, anti-inflammatory, anti-tubercular, cytotoxic, anti-depressant, anti-bacterial, anti-HIV, anti-fouling, trypanocidal, leishmanicidal, gastroprotective, modulation of nitric oxide production and so on. Additionally, some of chalcone derivatives have been found to inhibit several important enzymes in cellular systems, such as xanthine oxidase and protein tyrosine kinase.

Chalcones are α,β -unsaturated ketones consisting of two aromatic rings (ring A and B) having diverse array of substituents. Rings are interconnected by a highly electrophilic three carbon α,β -unsaturated carbonyl system that assumes linear or nearly planar structure. They contain the ketoethylenic group ($-\text{CO}-\text{CH}=\text{CH}-$).

EXPERIMENTAL METHODS

Procedure for the preparation of chalcone using NaOH catalyst:

A mixture of acetophenone (3g) and cinnamaldehyde (3.053g) in ethanol (40 ml) and 40 % NaOH solution was stirred for (24 hrs.) at room temperature. The reaction mixture was acidified by 10% HCl solution. The product formed was filtered and recrystallized from ethanol to give 1,5-DIPHENYL 2,4 PENTADIENONE.

Procedure for the preparation of chalcone using KOH catalyst:

A mixture of acetophenone (3g) and cinnamaldehyde (3.053g) in ethanol (40 ml) and 40 % KOH solution was stirred for (24 hrs.) at room temperature. The reaction mixture was acidified by 10% HCl solution. The product formed was filtered and recrystallized from ethanol to give 1,5-DIPHENYL 2,4 PENTADIENONE.

Procedure for the preparation of chalcone using Na_2CO_3 catalyst:

A mixture of acetophenone or derivatives (3g) and cinnamaldehyde (3.053g) in ethanol (40 ml) and 40 % Na_2CO_3 solution was stirred for (24 hrs.) at room temperature. The reaction mixture was acidified by 10% HCl solution. The product formed was filtered and recrystallized from ethanol to give 1,5-DIPHENYL 2,4 PENTADIENONE.

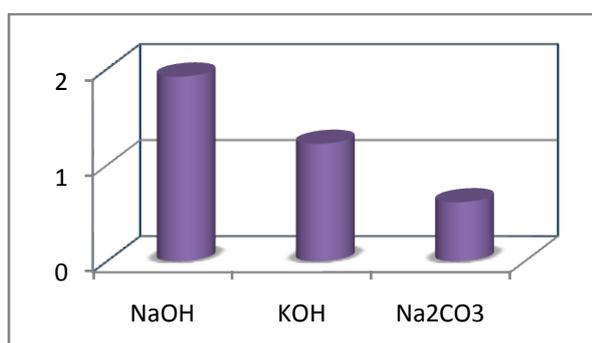
RESULT AND DISCUSSION

Accordingly one mole of acetophenone and one mole of cinnamaldehyde are mixed well using ethanol as solvent in the presence of three different catalysts. The mixture is allowed to stir over night. The product of the reaction

was monitored by TLC. Three different catalysts are used to carry out the reaction and the yield of the products can be compared.

Tabular column shows the yield of the product by varying the catalyst:

Reactant	Solvent	Catalyst	Yield
Acetophenone & cinnamaldehyde	ethanol	NaOH	1.921
Acetophenone & cinnamaldehyde	ethanol	KOH	1.221
Acetophenone & cinamaldehyde	ethanol	Na ₂ CO ₃	0.613



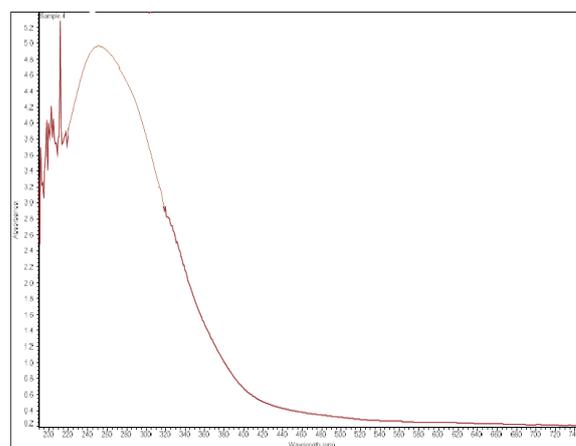
Tabular column shows the λ_{\max} of the product by varying the catalyst:

Solvent (0.01N)	Catalyst	λ_{\max}
NaOH	NaOH	260
	KOH	265
	Na ₂ CO ₃	260
KOH	NaOH	250
	KOH	260
	Na ₂ CO ₃	260
NH ₃	NaOH	255
	KOH	260
	Na ₂ CO ₃	255

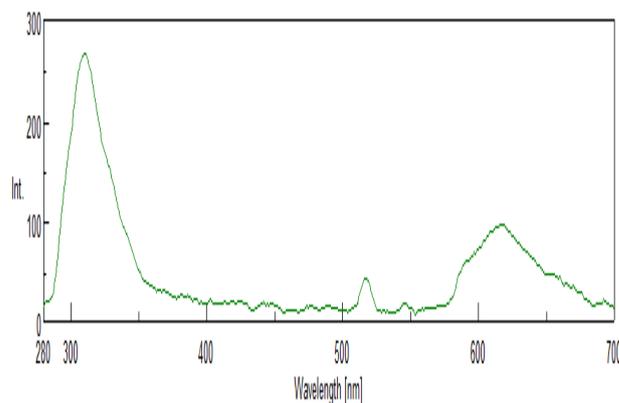
The strength of the three bases decreases in the order NaOH > KOH > Na₂CO₃. In this reaction, strong base catalyst increases the yield and weak base catalyst decrease the

yield. Sodium hydroxide is the strong base used here hence its yield is high and sodium carbonate is the weak base used hence its yield is low.

UV-visible spectrum of 1,5-diphenyl 2,4-pentadienone using NaOH as catalyst(0.01N NaOH)



Fluorescence Emission Spectrum of 1,5-diphenyl 2,4-pentadienone



The excitation and emission fluorescence spectrum of 1,5-diphenyl 2,4-pentadienone shows the fluorescent properties which clearly seen in the appearance of fluorescence emission spectrum. The emission spectrum was observed in various range of nm. The highest fluorescence emission maxima (λ_{\max}) obtained at 675nm for NaOH catalyst and

the lowest fluorescence emission maxima (λ_{\max}) obtained at 306nm for Na₂CO₃ catalyst.

CONCLUSION

The importance of chalcones and a brief survey on the methods of their preparation, mechanism and their reactions were analysed. A rapid, high yield, simple, practical, economic, readily available system, and convenient procedure for the synthesis of chalcones have been developed. The experimental details associated with the reaction describes it is the easiest method to synthesis chalcones. The yield of the reaction of acetophenone with aldehyde in the presence of different bases can be compared. The products formed has been characterized by spectral studies.

REFERENCES

1. Straub, T. S. (1995) *Tetrahedron Lett* .36, 663.
2. Sandler, S., Karo, W. (1972) In *Organic Functional Group Preparations*. 3, 372.
3. Bergman, E. D., Ginsibm, L., Pappo, R. (1959) *Org. React* .10, 179.
4. Ducki, S., Forrest, R., Hadfield, J. A., Kendall, A., Lawrence, N. J., McGown, A.T., Rennison, D. (1998) *Bioorg. Med. Chem*. 8, 1051.
5. Hart, H., Rajkumar, P. (1995) *Tetrahedron*. 51, 1313.
6. Viond, T. K., Rajkumar. P., Hart, H. (1995) *Tetrahedron*. 51, 2267.
7. Kei, G., Gaku, Y. (2001) *Tetrahedron Lett* .42, 4875.
8. Grewal, R. S., Hart, H., Viond, T. K. (1992) *J. Org. Chem* .57, 2721.
9. Rajkumar, P., Kannan, A. (1993) *Tetrahedron Lett*. 34, 4407.
10. Viond, T.K., Hart, H. (1990) *J. Org .Chem*. 55, 881.
11. Rajkumar, P., Srisailas, M. (1997) *Tetrahedron Lett*. 38, 5323.
12. Rajkumar, P., Srisailas, M. (2001) *Tetrahedron* .57 , 9749.
13. Dong-mee Song, Kyoung-hoon Jung, Ji-hye Moon and Dong-myung Shin, (2002). *Photochemistry of chalcone and the application of chalcone-derivatives in photo-alignment layer of liquid crystal display* *Optical Materials*, 21, 667–671.
14. *International Journal of ChemTech Research* CODEN (USA): IJCRGG ISSN: 0974-4290 Vol.2, No.2, pp 1080-1089, 2010
15. Maria J.Gozalez Moa, Marcos Mandado et al. (2007). *QTAIM electron density study of natural chalcones*. *Chemical physics letters*, 446.