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Natural Catalyzed Synthesis of Chalcone by Using Grindstone Chemistry under Solvent Free Conditions

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ABSTRACT:

A facile and efficient synthesis of Dibenzalacetone (3a-3i) has been achieved by the condensation of Aromatic aldehyde and aliphatic ketone in presence of different natural catalysts. This has been carried out by using Grindstone Chemistry through intramolecular aldol condensation for half an hour. All synthesized compounds were characterized on the basis of UV, IR, NMR, GCMS spectral and elemental analysis. The yields were excellent and requires short reaction time, easy work up, energy saver and without solvent are the notable advantages of this method.

Keywords: Chalcone, Natural catalysts, Grindstone chemistry, Benzaldehyde, Acetone.

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1. INTRODUCTION

The chalcones and their derivatives are significant versatile intermediates in synthesis of heterocyclic compounds [1] owing to their possession of a ketoethylenic group, -CO–CH═CH–. Recent literature survey proved that, the presence of enone functionality in chalcone moiety is found to be responsible for various pharmaceutical and medicinal applications. These applications encompass a wide range of therapeutic activities such as anticancer [2], anti-

inflammatory [3-4], anti-leishmanial [5] , analgesic[6] , antioxidant [7-8], antiviral [9], antifungal[10], antimalarial[11] , antibacterial[12] , vasorelaxant [13] activities etc. In the quest of harnessing the potential of chalcones for medicinal purposes, researchers face the challenge of developing environmentally benign and energy-efficient synthetic routes. In the present study, among the various methods for synthesizing chalcone derivatives, grindstone technique was used which presenting a simplified and eco-friendly route for the selective synthesis under solvent free condition [14]. The research not only strives to expand the synthetic toolbox for heterocyclic compounds but also emphasizes the importance of sustainable practices in chemical research. The exploration of these natural catalysts aligns with the global imperative to adopt sustainable and green chemistry practices in chemical synthesis. Therefore, we focused our attention towards applying natural catalysts derived from readily available natural sources such as *Coriander, Pineapple, Orange, Grapes, Cabbage, Cauliflower, Kiwi, Carrot, Cucumber and Ginger* by using grindstone chemistry.

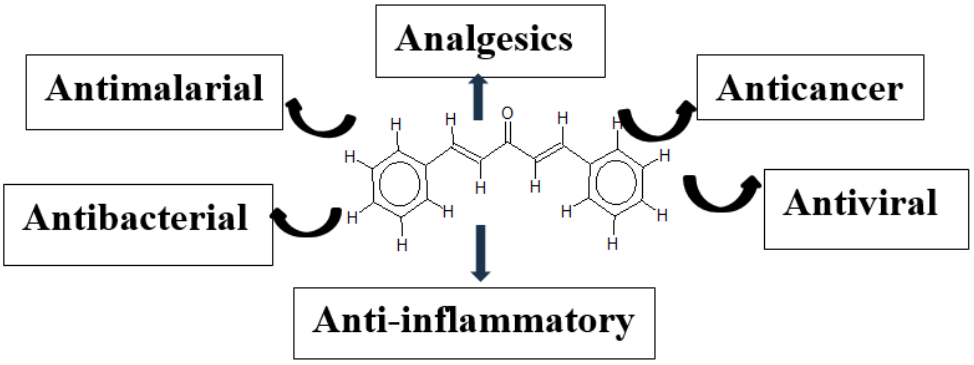


Fig. 1: Therapeutic activities of Dibenzalacetone

2. MATERIALS AND METHODS

All chemicals used in the study were of synthetic grade and obtained from S. D. Fine Chem. Ltd. Mumbai, India. The natural vegetables and fruits were purchased from the local market in Kolhapur city. The melting points were recorded on electro-thermal apparatus and are uncorrected. The purity of the compounds was checked by TLC on precoated SiO_2 gel (HF254, 200 mesh) aluminum plates (E Merk) using n-Hexane and Ethyl acetate (80:20) and visualized in iodine chamber. The products were characterized by UV,^{1}H NMR, IR and Mass spectral techniques. UV spectrum was recorded in ethanol on Beckmann DK-1 spectrophotometer. IR spectrum was recorded on a Perkin Elmer model-983 in the form of KBr pallet. ^{1}HNMR spectrum recorded on Varian Mercury 300 MHz instrument using $CDCl_3$, DMSO-d_6 as solvent (chemical shift in δ ppm), using TMS as internal standard. Mass spectrum was recorded on an EI-Shimadzu GC-MS spectrometer. Elemental analysis was performed on a Heracus CHN analyzer and was within the ±0.5% of the theoretical values. The synthesis of chalcone has been carried out by using grinding technique under solvent free conditions as a green technique [14] but we have modified this method by using various natural catalysts which are easily available instead of NaOH, LiOH or other hazardous chemicals to minimize the environmental pollution.

1.3 General Procedure:

1.3.1 Synthesis of natural source extract.

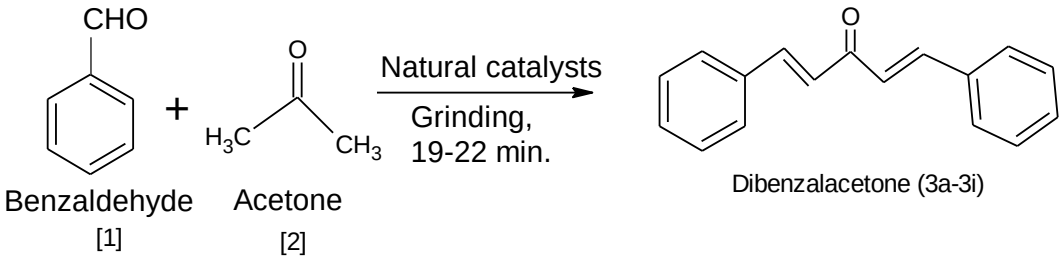
Fresh and easily available natural plants and fruits were selected and purchased from the local market. Then cut the selected plants and fruits separately by using knife at the time of reaction. Then each piece was pressed manually using domestic presser to extract juice at ambient

temperature. Then juice was filtered through cotton/muslin cloth and through filter paper to remove solid material to get clear juice which was used as a natural catalyst for the reaction.

1.3.2 Synthesis of Chalcone by using Natural catalysts.

An equimolar mixture of acetone (3.0 g; 0.05mol) and benzaldehyde (5.5 g; 0.05mol) was taken in pestal mortar and added 5 ml of natural source extract by grindstone chemistry for 19-22 min. The progress of the reaction was monitored) after every 5 min by using TLC (n-hexane: ethylacetate, 80:20). The reaction mixture was cooled and then the obtained solid was recrystallized from ethanol. (Scheme-I)

Scheme-



Natural catalysts: a. Coriandrum ; b. V.Aloe; c. S. Cucummis; d. C. Daucus ; e. D.Actinidia; f. I. Rosa ; g. O. Zingiber ; h. B.Brassica; i. C. Brassica

Table-I : Physical Data of Dibenzalacetone (3a-3i):

Sr.No.	**Natural Catalysts**	**PH**	**M.P. ^{o}C**	**Yield %**	**Time Required (Min).**
3a	Sativum Coriandrum	5.11	110	73.37	21.20
3b	Aloe Vera	7.2	108	83.20	18.50
3c	Cucumis Sativus	6.3	108	77.50	20.45
3d	Daucus Carota	6.8	112	81.71	22.00
3e	Actinidia Deliciosa	8.9	112	87.26	21.30
3f	Rosa Indica	6.5	111	78.33	19.10
3g	Zingiber Officinale	5.9	109	75.06	20.25
3h	Brassica Oleracea Var.Botrytis	6.5	110	79.55	21.50
3i	Brassica Oleracea Var. Capitata	7.4	112	84.68	19.30

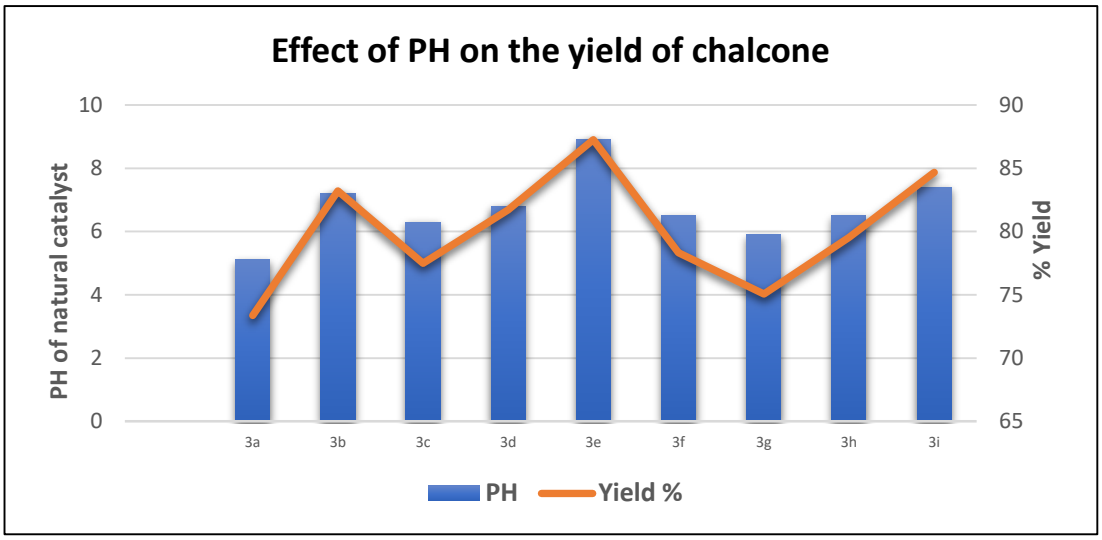


Fig.2: Effect of PH on the yield of chalcone

1.3.3 Spectral data of Dibenzalacetone

Physical constant (mp): 108-112ºC

UV : λmax, 320 nm

IR(KBr): ν, 3060-3028 (Ar-**H**),2918(=C**H),** 1651**(>C=O),** 1593,1495 **(>C=C<)** cm^{-1}

NMR : δ, 7.33-7.54(m,10H,Ar-**H**), 7.82(d,2H,=C**H**CO), 7.03(d,2H,=C**H**) ppm.

MS : m/z = 234 (M+1).

Elemental analysis; found (calculated): C,87.58 (87.17); H, 6.01(5.98); O, 6.87 (6.83).

3. RESULTS AND DISCUSSION

Chalcones are among the leading bioactive flavonoids with a therapeutic potential implicated to an array of bioactivities investigated by a series of preclinical and clinical studies. Many researchers have been done the synthesis of Chalcone by using different methods such as Conventional and non-conventional methods which requires larger time, less atom economy and in presence of hazardous

catalysts. so, we have developed efficient, operationally simple, environmental benign method under solvent free condition by simple reaction of Benzaldehyde and Acetone in presence of natural catalysts (**Scheme-I**) that are easily available from local market in Kolhapur city by using grindstone chemistry requires shorter reaction time and high yields up to 73 to 88% (**Table-I**) which is very fertile and easiest to all researchers. The structures of synthesized compound were assigned on the basis of UV, IR, ^{1}H-NMR and GCMS spectral data and we have examined peaks at their proper sites.

In the present investigation, we wish to report synthesis of chalcones by using variety of natural catalysts such as *actinidia deliciosa*, *Coriandrum sativam, Aloe vera, Cucumis sativas, Daucus carrotasub, Rosa, Zingiber officinails, Brassica oleracea, var-capitata.* Further, we have observed ,the effect of PH of each catalyst on the yield of the product. In this study, we observed that, the presence of high pH values of related catalysts (PH range from 7.4-8.9), the reaction offers excellent yields (**TableNo. I, Entry No. 3b,3e,3i**) indicates that when alkalinity of catalyst increases , the yield of chalcone is also increases **(Fig.2).** But when reaction proceeds in the pH range below 7.2 reaction offers less yields. In addition to this, when we have added 1 by 1 ml to the reaction mixture, it has been found that, if we have added 5 mi of extracted juice, it enhances the yield in short reaction time as compare to addition of 1 to 4 and 6 to 8 ml of extracted juice. So, we have preferred the addition of 5 ml of each extracted juice as a catalyst **(Table No. II, Fig.3).**

Table No. II: Effect of catalyst on the yield of the product

Catalyst	Extract added (ml)	Yield obtained (%)	Time required (Min.)
Sativum Coriandrum	1	70.44	23.05
	2	70.57	22.65
	3	71.33	22.30
	4	71.68	22.45
	5	**73.37**	**21.20**
	6	72.45	21.45
	7	72.13	21.50
	8	72.10	21.50

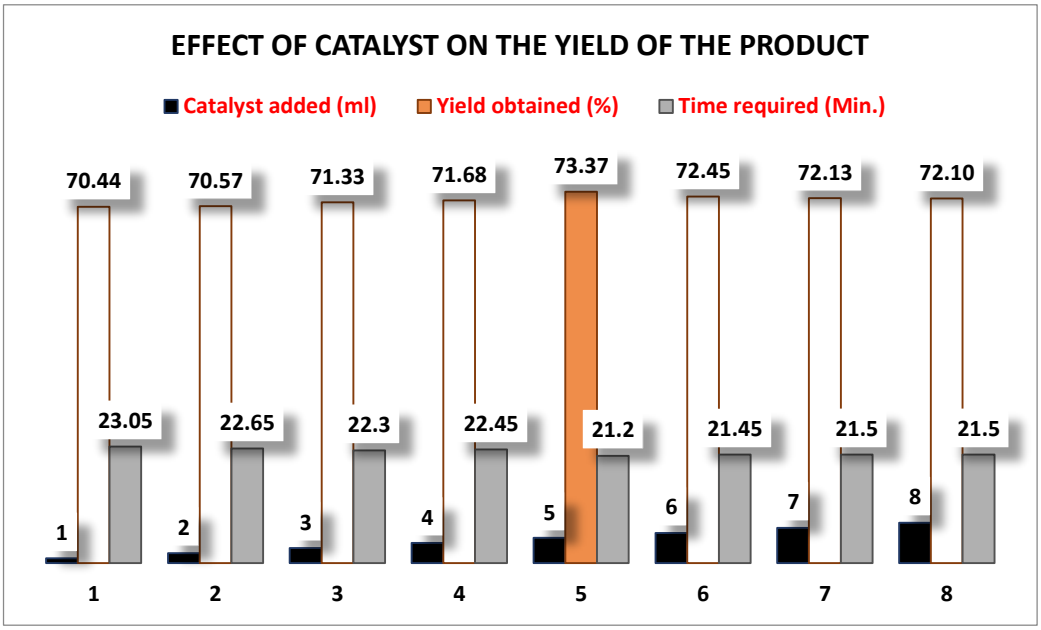
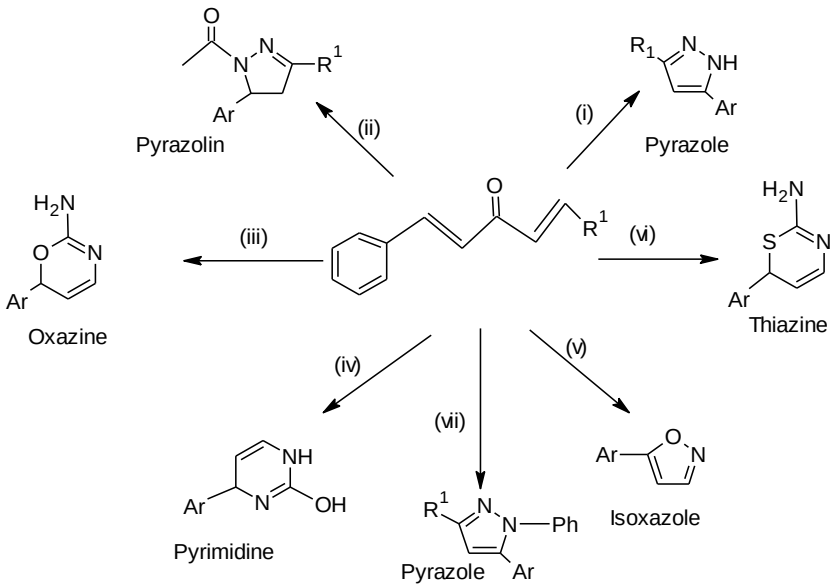


Fig. No.3: Effect of catalyst on the yield of the product

The Main object in synthesis of chalcone is that it can be further used in synthesis of heterocyclic compounds i.e. it acts as an intermediate in organic synthesis [14]**as per Scheme-II.**



Scheme-II

(i) Hydrazine/EtOH (ii) $NH_2.NH_2$ /GAA (iii) $O=C(NH_2)_2$,EtOH/NaOH

(iv) $S=C(NH_2)_2$,EtOH/NaOH (v) $NH_2OH.HCl$,EtOH (vi) $O=C(NH_2)_2$

(Vii) $Ph.NH.NH_2$/GAA

In addition to its simplicity, the results obtained are superior yields by using the natural products (Table I) in the Knoevenagel condensation. A stoichiometric amount of catalyst was sufficient to obtain good yield.

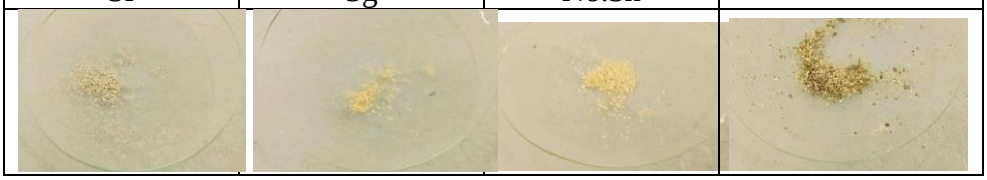
Compound No. 3a	Compound No. 3b	Compound No. 3c	Compound No.3d	Compound No.3e
				
Compound No. 3f	Compound No. 3g	Compound No.3h	Compound No.3i	
				

Fig. 3. The nature and color of corresponding compounds (3a-3i)

4. CONCLUSION

We have developed facile, efficient and operationally simple method in the synthesis of chalcone by using different easily available natural catalysts under solvent free condition using grinding technique. It was observed that, the alkaline PH catalysts such as Brassica *oleracea* and *actinidia deliciosa* gave an excellent yield than other catalysts. The notable advantages of present method are no organic solvent required (except for the product recrystallisation), waste minimization, simple operation, clean reaction profile, easy work-up, energy sever, shorter reaction time (18-22min.), high yields (75- 88%) and eco-friendly as compared to other conventional method.

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5. REFERENCES

1. Yogeesha N. Naik, Santosh L.Gaonkar,Mariya Sabu; Chalcones:Versite intermediates in heterocyclic synthesis ,Journal of Heterocyclic chemistry,2023,60,1301-1325.
2. Konieczny MT, Konieczny W, Sabisz M, Skladanowski A, Wakiec R, Augusttynowiczkopec E, Zwolska Z (2007). Synthesis of isomeric, oxathiolone fused chalcones and comparison, their activity towards various microorganisms and human cancer cell lines. Chemical and Pharmaceutical Bulletin 55: 817-820
3. Venkatesh, G., Vennila, P., & Balasubramaniyan, S. (2024). Solvent effects, chemical reactivity, docking and antimicrobial activity of silver and gold nanocages glimepiride: Experimental and theoretical calculations. *Chemical Physics Impact*, *8*, 100498.
4. Irfan, N., Balasubramaniyan, S., Ali, D. M., & Puratchikody, A. (2023). Bioisosteric replacements of tyrosine kinases inhibitors to make potent and safe chemotherapy against malignant cells. Journal of Biomolecular Structure and Dynamics, 41(19), 9437-9447.
5. Nagendran, S., Balasubramaniyan, S., & Irfan, N. (2023). Virtually screened novel sulfathiazole derivatives as a potential drug candidate for methicillin-resistant Staphylococcus aureus and multidrug-resistant tuberculosis. Journal of Biomolecular Structure and Dynamics, 41(11), 5086-5095.
6. Navabshan, I., Sakthivel, B., Pandiyan, R., Antoniraj, M. G., Dharmaraj, S., Ashokkumar, V., ... & Show, P. L. (2021). Computational lock and key and dynamic trajectory analysis of natural biophors against COVID-19 spike protein to identify effective lead molecules. Molecular biotechnology, 63(10), 898-908.
7. Balasubramaniyan, S., Irfan, N., Umamaheswari, A., & Puratchikody, A. (2018). Design and virtual screening of novel fluoroquinolone analogs as effective mutant DNA GyrA inhibitors against urinary tract infection-causing fluoroquinolone resistant Escherichia coli. RSC advances, 8(42), 23629-23647.
8. Emelda N. Okolo, David I. Ugwu, Benjamin E. Ezema, Joseph C. Ndefo, Florence U. Eze, Chidimma G. Ezema, James A. Ezugwu & Oguejiofo T. Ujam New chalcone derivatives as potential antimicrobial and antioxidant agent ,2021,11, 21781.
9. Díaz-tielas C, Grañam E, Reigosaa MJ, Sánchez M, Biological activities and Novel applications of chalcones.Planta daninha, 34 (03),2016, 1-7.
10. Go ML, Wu X, Liu, XL (2005). Chalcones: an update on cytotoxic and chemoprotective properties. Curr Med Chem 12: 483.
11. Shweta Sinha, Bikash Medhi, B. D. Radotra, Daniela I. Batovska, Nadezhda Markova,Ashish Bhalla, and Rakesh Sehgal Antimalarial and immunomodulatory potential of chalcone derivatives in experimental model of malaria, BMC Complement Med Ther. 2022; 22: 330. doi: 10.1186/s12906-022-03777-w.
12. Dragana D. Božić, Marina Milenković, Branka Ivković and Ivana Cirković, Antibacterial activity of three newly-synthesized chalcones & synergism with antibiotics against clinical isolates of methicillin-resistant Staphylococcus aureus, Indian J Med Res. 2014 ; 140(1): 130–137.
13. Prashar H,Chawla A,Sharma AK, Kharb R, Chalcone as a versatile moiety for diverse pharmacological activities, International journal of Pharmaceutical science and research, 3(7),2012, 1913-1927.
14. Piste PB, Synthesis of Chalcones by grindstone chemistry as an intermediate in Organic Synthesis, International Journal of Current Science, 13,2014,62-66.