# Efficient and green synthesis of chalcones catalyzed by natural surfactant

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**Abstract:**

Chalcones are chemical entities belonging to flavonoid class of compounds. They occur naturally in many plants and have been used as medicines in traditional therapies. Significant research interest has been devoted to find new and efficient methods to synthesize chalcones. Aldehyde is one the substrate in most synthetic protocols leading to chalcones. Diversely substituted aldehydes under Claisen-Schmidt conditions may lead to undesired products. Hence acid-catalyzed synthesis of chalcones offers a promising alternative. In the present work, aqueous extract of dry soap nut fruits is used as catalyst to obtain chalcones from condensation of aromatic aldehydes with acetophenones. This extract is acidic in nature and possesses surfactant properties due to saponins present in it. Acidic nature coupled with surfactant property facilitates the reaction and yields desired products efficiently.

## 1. Introduction:

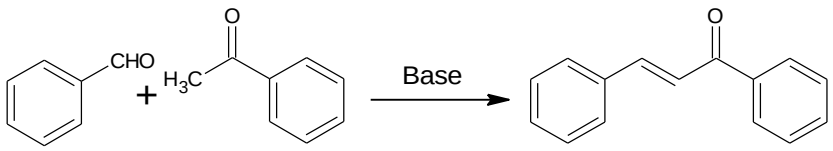
Chalcones are a genre of chemical substances that occur in a variety of plants species belonging to different families [1]. There are numerous reports pertaining to pharmacological properties exhibited by chalcones [2]. Some of the biological activities reported to be shown by chalcones are anti-bacterial [3], antimalarial [4], antifungal [5], anti-cancer [6], etc. Chalcones are also a very good synthon to obtain diverse and novel heterocyclic compounds with good pharmacological activity [7]. In terms of chemistry, they contain two aromatic rings joined together by a three carbon carbonyl system, with carbon-carbon double bond at 2,3-position with respect to carbonyl carbon. This conjugated carbonyl fragment of chalcones renders them highly reactive.

Chalcone synthesis is an immensely challenging task for the chemists and specialists to create new product and processes that are beneficial to environment. Pioneered by Claisen and Schmidt [8], synthesis of chalcones is achieved by various pathways which differ in reaction conditions. However aryl methyl ketone and aromatic aldehyde remained the most preferred substrates in chalcone synthesis.

Claisen-Schmidt condensation reaction between equimolar amounts of aryl methyl ketone and aromatic aldehyde is the preferred method of chalcone synthesis. This chemical process is carried out in alcoholic alkali as the catalyst [9]. Diverse alternative processes are reported but the most usual one is Claisen-Schmidt condensation of equimolar masses of a substituted acetophenones with substituted aldehydes **[Scheme: 1]** in aqueous alcoholic alkali [10]. In the Claisen-Schmidt reaction, the concentration of alkali used, usually ranges between 10 and 60 %.The reaction is carried out at about 50°C for 12-15 hours or at ambient temperature for one week. The reaction conditions employed may favour Cannizzaro

reaction and actually some amount of Cannizzaro product is also formed thereby decreasing the yield of the desired product.

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




**Scheme: 1**

Acid-catalyzed synthesis of chalcones is also reported in literature. S. Shylesh and others have reported application of sulfonic acid functionalized mesoporous silicas and organosilicas as catalysts to synthesize various chalcones [11]. Solid sulfonic acid from bamboo has also been applied to catalyze chalcone synthesize [12]. Chalcone synthesis via acid catalysis becomes significant in view of diversity of aldehydes to be used as substrates. Acid-catalyzed synthesis of chalcones may help in avoiding competing reactions thereby enhancing product purity.

## 2. **Experimental:**

Preparation of catalyst material:

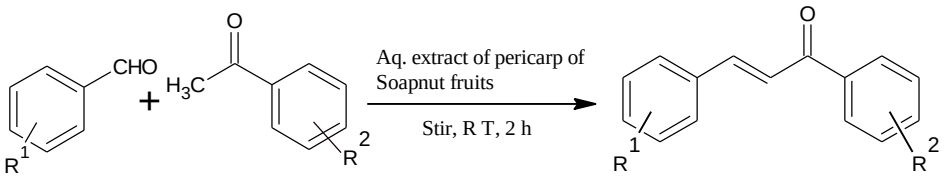
Dried fruits of *Sapindus trifoliatus* (soap nut) were purchased from local market. 100gm dried pericarp of fruits was soaked in 400ml water for 12 hours. The material was then macerated with the water in which it was soaked and filtered. The filtrate that is the aqueous extract was kept below $5^0$ c and used as catalyst for 14 days.

Typical procedure for the synthesis of chalcone:

Equimolar mixture of the starting materials was taken in a round bottom flask provided with magnetic needle. The catalyst material was added (10 mL) and was stirred magnetically for appropriate period. After completion of the reaction, the reaction mixture was poured in ice cold water and filtered at suction. The crude product was purified by recrystallization.

## 3. Results and Discussion:

Aqueous extract of pericarp of *Sapindus trifoliatus* (soap nut) fruits was employed by us as acidic catalyst with surfactant properties to catalyze synthesis of aldimines [13] and bis(indolyl)methanes [14].




**Scheme: 2**

Synthesis of chalcones was carried out in aqueous medium as it was efficient, easy to operate and environmental. Aqueous extract of pericarp of *Sapindus trifoliatus* (soap nut) fruits was used as a catalyst as well as reaction medium for the synthesis (Fig. 1). Reaction efficiently proceeds to give desired product

with excellent yield. Simple filtration allows us to separate the catalyst from the product. Hence this method provides us a facile one pot synthesis of the product.

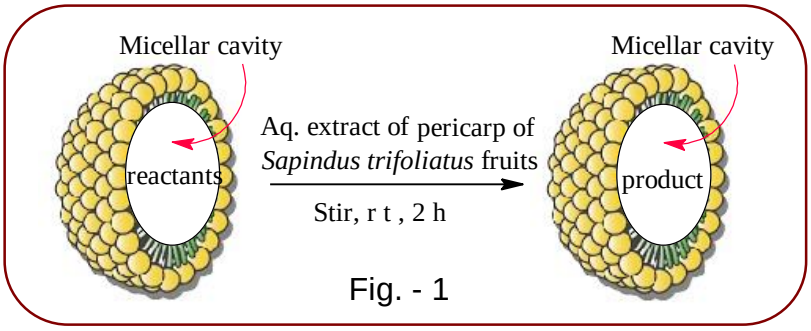



Fig. - 1

Synthesis of chalcone is a single step method. The structure of synthesized compound was confirmed with the help of IR and $^1$H NMR spectra. All the compounds exhibited characteristic IR peak that proved the presence of particular functional group and NMR spectroscopy helped to ascertain type and number of protons. A summary of synthesized chalcones is presented in Table-1.

Table-1: Summary of some synthesized chalcones

| Entry | $R^1$ | $R^2$ | Time | Yield (%) | Melting point (°C) |
|---|---|---|---|---|---|
| 1 | H | H | 3 h | 85 | 52-54 |
| 2 | H | Cl | 3 h | 86 | 123-125 |
| 3 | 2,4-OH | Cl | 4 h | 85 | 149-152 |
| 4 | 2,4-OH | 4-$OCH_3$ | 4 h | 82 | 178-182 |
| 5 | 4-$OCH_3$ | 4-OH | 4 h | 85 | 221-225 |
| 6 | 4-Cl | H | 3 h | 91 | 118-120 |
| 7 | 4-Cl | 2,4-OH | 4 h | 80 | 152-155 |
| 8 | 4-$OCH_3$ | H | | 82 | 72-75 |

**Spectral Data:**

1,3-Diphenylprop-2-en-1-one (Table 1, entry 1)

IR (KBr) νmax / $cm^{-1}$ 3061.73, 1662.20, 1604.43, 1574.35, 1447.59, 1336.59, 1214.26, 1013.90,$^1$H NMR (400 MHz, CDCl3) δ 8.03 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.71–7.64 (m, 2H), 7.60–7.47 (m, 4H), 7.45–7.38 (m, 3H)

3-(4-Chlorophenyl)-1-phenylprop-2-en-1-one (Table 1, entry 6)

IR (KBr) νmax / $cm^{-1}$ 3059.83, 1656.96, 1600.19, 1486.72, 1310.86, 1214.12, 1010.51, 981.74, 820.20, 773.14, 683.46; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.34 Hz, 2H), 7.40-7.51 (m, 5H,), 7.58 (d, *J* = 14.98 Hz, 1H), 7.74 (d, *J* = 15.86 Hz, 1H), 8.01 (d, *J* = 7.28 Hz, 2H)

3-(4-Methoxyphenyl)-1-phenylprop-2-en-1-one (Table 1, entry 8)

IR (KBr) νmax / $cm^{-1}$ 3089.58, 3016.86, 2955.13, 2902.07, 2842.33, 1656.32, 1593.93, 1509.34, 1261.24, 1209.29, 1015.39, 984.24, 824.33, 778.69, 688.02; $^1$H NMR (400 MHz, $CDCl_3$) δ 3.80 (s, 3H), 6.90 (d,

2H, J 8.69 Hz), 7.38 (d, 1H, J 15.64 Hz), 7.44-757 (m, 5H), 7.76 (d, 1H, J 15.63 Hz), 7.98 (d, 2H, J 7.29 Hz)

## 4. Conclusion:

The protocol developed for the chalcone synthesis uses green catalyst, the aqueous extract of pericarp of *Sapindus trifoliatus* (soap nut) fruits, easily accessible and of low cost. The need of organic solvent is minimum to nil because only in a few cases ethyl alcohol was added. The reaction times have been significantly reduced that at room temperature. There is no need to take special precautions while using the catalyst as it is absolutely innocuous to human and the biosphere.

## 5. References: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