Chapter I
Synthesis of Novel Chalcones
Chapter I: Synthesis of Novel Chalcones

Synthesis of

- (E)-3-(4-(allyloxy)-3-methoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-ones
- (E)-1-(2-hydroxyphenyl)-3-(4-(phenylthio)phenyl)prop-2-en-1-ones
- (E)-1-(2-hydroxyphenyl)-3-(1-methyl-1H-pyrrol-3-yl)prop-2-en-1-ones
1.1. Introduction

Heterocycles make up an exceedingly important class of compounds. In fact more than half of all known organic compounds are heterocycles. Many natural drugs such as quinine, papaverine, emetine, theophylline, atropine, procaine, codeine, morphine and reserpine are heterocycles. Almost all the compounds we know as synthetic drugs such as diazepam, chlorpromazine, isoniazide, metronidazole, azidothymidine, barbiturates, antipyrine, captopril and methotrexate are also heterocycles. Some dyes (e.g. mauveine), luminophores, (e.g. acridine orange), pesticides (e.g. diazinon) and herbicides (e.g. paraquat) are also heterocyclic in nature.

All these natural and synthetic heterocyclic compounds can and do participate in chemical reactions in the human body. Furthermore, all biological processes are chemical in nature. Such fundamental manifestations of life as the provision of energy, transmission of nerve impulses, sight, metabolism and the transfer of hereditary information are all based on chemical reactions involving the participation of many heterocyclic compounds, such as Vitamins, Enzymes, Co-enzymes, ATP, DNA, RNA and Serotonin. So nature utilize heterocycles. The answer to this question is provided by the fact that heterocycles were able to get involved in an extraordinarily wide range of reaction types. Depending on the pH of the medium, they may behave as acids or bases, forming anions or cations. Some interact readily with electrophilic reagents, others with nucleophiles, yet others with both. Some are easily oxidized, but resist reduction, while others can be readily hydrogenated but are stable toward the action of oxidizing agents. Certain amphoteric heterocyclic systems simultaneously demonstrate all of the above-mentioned properties. The ability of many heterocycles to produce stable complexes with metal ions has great biochemical significance. The presence of different heteroatom’s makes tautomerism ubiquitous in the heterocyclic series. Such versatile reactivity is linked to the electronic distributions in heterocyclic molecules. Evidently, all the natural products and the synthetic drugs mentioned above are good examples of nature’s preference for heterocycles whose biological activity cannot be determined by one or a combination of two or three of the above mentioned properties.

It can be questioned that it is specifically appropriate to emphasize the role of heterocycles, since analogies to the roles of other classes of organic compounds are easily found. In fact,
dyes, luminophores, pesticides, herbicides and drugs do not necessarily have to be heterocyclic in structure. We know the reason for this; the introduction of a heteroatom into a cyclic compound imparts new properties. Heterocycles are chemically more flexible and better able to respond to the many demands of biochemical systems. Organic molecules containing five-membered, six-membered and seven-membered heterocycles are widely distributed in nature. They play an important role in various biochemical processes. A number of heterocyclic compounds containing nitrogen, sulphur and other hetero atoms, find applications in various fields ranging from dyes, drugs, pigments and so on. Structure-metabolism relationship (SMR) studies often reveal that incorporation of one or more of these heteroatom’s in an aromatic ring influences the chemical and biochemical reactivity of these compounds and therefore alter their metabolism. Thus, In modern days medicinal chemistry is based on heterocyclic molecules and thus owe to them due to their close association with numerous biological as well as pharmacological activities. In heterocyclic molecules these biological activities are based on following important aspects

1. The presence of heteroatoms like oxygen, nitrogen or sulphur in the molecule
2. The presence of heterocyclic moiety in the drug molecule
3. The position of nucleus and heteroatom in the molecule

Owing to the emergence of multi-drug resistant strains in recent decades, microbial diseases have become more complex to tackle as compared to the first half of the last century. To combat such microbial infections, various synthetic and semi-synthetic antimicrobial drugs have been discovered and used in the clinical practice. In spite of significant developments in the antimicrobial therapy, problems such as resistance development, spectrum of activity, potency and toxicity remain unresolved for most of the available antimicrobial drugs. Hence, the discovery and development of novel antimicrobial agents with optimized pharmacological profile together with increased activity towards resistant strains is highly desirable.

So the greatest concern with infectious diseases, especially malaria and tuberculosis, is the alarming rate at which resistance against clinically used drugs develops. This imparts a degree of urgency for the discovery of affordable and effective alternative drugs. The appeal of working with chalcones (1,3-diarylprop-2-en-1-ones) systems from their synthetic accessibility or utility, the various ways the core structure can be diversified and
their ability to confer drug-like properties to compound libraries modeled on them [1]. The consistent with its privileged status, a wide range of biological activities such as anti-inflammatory [2], antileishmanial [3], antibacterial [4], antifungal [5], antitumor [6], antimalarial [7,8] and anti-TB activity [9] has been reported for chalcone derivatives. Regarding the antimalarial activity, it has been proposed that chalcones exert their mode of action by the inhibition of cysteine proteases [10]. Failure to correlate the antiplasmodial activity of some chalcones with cysteine protease inhibition led to the conclusion that other targets and/or pathways may be compromised [8d]. In fact, recent reports suggest that chalcones also play a role in the inhibition of new permeability pathways induced by the parasite in the erythrocyte membrane [8g]. Chalcones are also precursors for flavonoids and isoflavonoids family. They are widely distributed in fruits, vegetables, spices, tea and soya based foodstuff. Chalcones have been subject of great interest for their interesting and variable pharmacological activities. [11] Chemically they consist of open-chain flavonoids in which the two aromatic rings are joined by a three carbon α,β-unsaturated carbonyl system. The radical quenching property of the phenolic groups in polyhydroxylated chalcones makes them drugs or food preservatives and rapidly gaining attention towards themselves [12]. All these are well reported in reviews [13, 14].

A number of chalcone derivatives have also been found to inhibit several enzymes in cellular systems, including xanthine oxidase [15], aldose reductase [16], epoxide hydrolase [17], protein tyrosine kinase [18, 19], and quinine reductase [20].

In pure synthetic organic chemistry chalcones are considered to be an important and versatile synthones. Chalcones are reported to behave as precursors for synthesis of different heterocyclic molecules in which chalcones serve as fundamental building block for their synthesis. The molecules derived from chalcones include, chromones pyrazolines, pyrimidines, pyrazoles, iso-oxazolines, benzodiazepines, benzothiazepines etc. (Fig. 1)
Fig 1. Chalcones as precursor/synthons for different heterocycles
1.2. Methods of Synthesis

i. Claisen-Schmidt condensation

The Claisen–Schmidt condensation is basic reaction for the synthesis of chalcones in which aliphatic or aromatic ketones condense with an aldehyde in presence of soluble alkaline hydroxides or NaOEt [21]. Other than the soluble bases various heterogeneous solid bases are also employed to obtain chalcones. These include alumina [21], Ba(OH)$_2$ [23], hydrotalcites [24], MgO, and calcined NaNO$_3$/ natural phosphate [25, 26]. The acid catalyzed methodologies include the use of AlCl$_3$ [27], BF$_3$ [28], dry HCl [29], TiCl$_4$ [30], Cp$_2$ZrH$_2$/ NiCl$_2$ [31], Zeolites [32] and RuCl$_3$ [33]. Very recently chalcone synthesis has been accomplished by the use LiOH.H$_2$O [34] catalyst and by employing Suzuki coupling reaction [35] (Scheme 1.01).

\[
\begin{array}{c}
\text{R}_1\text{R}_2\text{R}_3\text{R}_1\text{R}_2\text{R}_3\text{O} \\
\text{H} \\
\text{EtOH}
\end{array}
\xrightarrow{\text{Aq. KOH}}
\begin{array}{c}
\text{R}_1\text{R}_2\text{R}_3\text{R}_1\text{R}_2\text{R}_3\text{O} \\
\text{O}
\end{array}
\]

Scheme 1.01

ii. Friedel-Craft acylation method

In Friedel-Craft acylation the aromatic hydrocarbons were acylated using α,β-unsaturated acid chloride in the presence of the Lewis acid shown [36-38] (Scheme 1.02).

\[
\begin{array}{c}
\text{Anhyd. AlCl}_3 \\
\text{Heat}
\end{array}
\xrightarrow{\text{Cl}}
\begin{array}{c}
\text{O}
\end{array}
\]

Scheme 1.02

iii. Reaction of benzene selenyl halide on ketones

In this reaction aromatic ketones form α-phenyl seleno-carbonyl compound which were oxidized by NaIO$_4$ to yield corresponding α,β-unsaturated carbonyl compounds [39] (Scheme 1.03).
iv. Heck reaction for the synthesis of chalcones

Heck reaction which is a reaction of alkenes and alkyl halides catalyzed by Pd metal complexes is used for synthesis of naturally occurring chalcones in single step [40] (Scheme 1.04).

v. Suzuki Coupling Reaction for Chalcone synthesis

Boronic acid catalyzed Suzuki coupling reaction was used recently for the synthesis of chalcones [41]. Two alternate pathways are described, one path uses aryl boronic acid with cinnamoyl chloride while other uses vinylboronic acid and acetyl chloride (Scheme 1.05).
vi. Synthesis of Chalcones under Ultrasonic Irradiation

Gill et al. [42] have reported the synthesis of 1-(2-Hydroxy-phenyl)-3-piperidin-1-yl-propenones by Ultrasonic Irradiation (Scheme 1.06).

\[
\begin{align*}
\text{CHO} & \quad \text{R}_2 \\
\text{R}_3 & \quad \text{N} \\
\text{R}_1 & \quad \text{H}
\end{align*}
\]

Scheme 1.06

vii. Claisen-Schmidt condensation under Ultrasonic irradiation

Martin Aranada and co-workers [43] have synthesized the chalcones under ultrasound irradiation by Claisen-Schmidt condensation using two basic activated carbons (Na and Cs-Norit) (Scheme 1.07).

\[
\begin{align*}
\text{CHO} & \quad \text{R} \\
\text{Basic activated carbons} & \quad \text{Basic activated carbons} \\
\text{(Na and Cs-Norit)} & \quad \text{(Na and Cs-Norit)}
\end{align*}
\]

Scheme 1.07
1.3. Present Work

Considering the importance of chalcones derivatives as the active therapeutic agents and are biologically important scaffolds due to the properties associated with them. Here, we designed the synthesis of novel chalcones i.e. \((E)-3-(4-(\text{allyloxy})-3\text{-methoxyphenyl})-1-(5\text{-chloro}-2\text{-hydroxy}-4\text{-methylphenyl})\text{prop-2-en-1-ones}\) 4 (a-g), \((E)-1-(2\text{-hydroxyphenyl})-3-(1\text{-methyl-1H-pyrrol-2-yl})\text{prop-2-en-1-ones}\) 6 (a-g) and \((E)-1-(2\text{-hydroxyphenyl})-3-(4\text{-((phenylthio)phenyl})\text{prop-2-en-1-ones}\) 10 (a-g) from substituted \(\text{o-hydroxy acetophenone}\) 3 (a-g) and \(4\text{-((allyloxy)-3-methoxybenzaldehyde}\) (2), \(4\text{-((phenylthio)benzaldehyde}\) (9) and \(1\text{-methyl-1H-pyrrole-2-carbaldehyde}\) (5) in ethanol and potassium hydroxide (KOH) at room temperature is carried out in the present investigation.

![Scheme A](image1)

![Scheme B](image2)
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\[ \text{Reflux} \]

Scheme C
1.4. Experimental

1.4A. Preparation of 4-(allyloxy)-3-methoxybenzaldehyde (2)
To the solution (1.0 gm, 0.006 mmole) 4-hydroxy-3-methoxybenzaldehyde dissolved in 5 mL of DMF (0.99 gm, 0.0072 mmole) was added K$_2$CO$_3$. The resultant reaction mixture was irradiated under ultrasonication at 60 °C for 5-10 min. followed by addition of (0.798 gm, 0.0066 mmole) allyl bromide and the reaction was continued for 46 min. The reaction was monitored by TLC. After the completion of the reaction, the mixture was poured over ice-cold water and extracted with ethyl acetate and washed with sodium sulphate, a liquid compound of 4-(allyloxy)-3-methoxybenzaldehyde (2) was obtained in 92% yield. The obtained liquid was directly used for next reaction without any purification.

1.4A. Preparation of (E)-3-(4-(allyloxy)-3-methoxyphenyl)-1-(5-chloro-2-hydroxy-4-methylphenyl)prop-2-en-1-ones 4 (a-g)
Alcoholic KOH (0.18 gm, 0.0032 mmole) was added to a suspension of 1-(5-chloro-2-hydroxy-4-methylphenyl)ethanone (2d) (0.272 gm, 0.0016 mmole) and 4-(allyloxy)-3-methoxybenzaldehyde (1) (0.3 gm, 0.0016 mmole) in 10 ml ethanol. The mixture was stirred at room temperature for overnight. The reaction was monitored by TLC. After the completion of reaction, mixture was poured into crushed ice and acidified with HCl (2N) till pH = 4. The solid product separated out was filtered off and crystallized from ethanol to afford 4 (a-g). The physical data of the compounds 4 (a-g) were recorded in Table 1. Their structures have been confirmed by $^1$H NMR, Mass and IR spectra.

1.4B. Preparation of (E)-1-(2-hydroxyphenyl)-3-(1-methyl-1H-pyrrol-2-yl)prop-2-en-1-ones 6 (a-g)
(E)-1-(2-hydroxyphenyl)-3-(1-methyl-1H-pyrrol-2-yl)prop-2-en-1-ones are prepared by using above procedure i.e. 1.4A method. The physical data of the compounds 6 (a-g) were recorded in Table 1. Their structures have been confirmed by $^1$H NMR, Mass and IR spectra.
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1.4C. Preparation of 4-(phenylthio)benzaldehyde (9)
To the solution of Thiophenol (1.0 g, 0.006 mmol) in 10 mL of DMF, was added (0.99 gm, 0.0072 mmole) of K$_2$CO$_3$. The reaction mixture was refluxed for 1 hr at 120 °C, Followed by addition (0.798 gm, 0.0066 mmole) of 4-Flurobenzaldehyde and continued the reaction for 5-6 hr. The reaction was monitored by TLC. After the completion of reaction. It was cooled and poured over crushed ice, white solid obtained was filtered off and crystallized from water.

1.4C. Preparation of $(E)$-1-(2-hydroxyphenyl)-3-(4-(phenylthio)phenyl)prop-2-en-1-ones 10 (a-g)
Alcoholic KOH (0.23 gm, 0.0040 mmole) was added to a suspension of o-hydroxyacetophenone (0.25 gm, 0.00135 mmole) and 4-(phenylthio)benzaldehyde (0.28 gm, 0.0015 mmole) in 10 mL ethanol. The mixture was irradiated under ultrasonication for 4-5 hr. The reaction was monitored by TLC. After the completion of reaction, mixture was poured over crushed ice and acidified with (2N) HCl till pH=4. Yellow solid obtained was filtered off and crystallized from ethanol. The physical data of the compounds 10 (a-g) were recorded in Table 1. Their structures have been confirmed by $^1$H NMR, Mass and IR spectra.
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Table 1: Physical data of the compounds. 4 (a-g), 6 (a-g) and 10 (a-g)

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Elemental analysis

Elemental analysis was performed on Perkin-Elmer EAL-240 elemental analyzer.

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1.5. Spectral analysis

**\(^1H\) NMR:** \(^1H\)NMR spectra were recorded in CDCl\(_3\) on a Brucker instrument at 400 MHz using TMS as an internal standard.

**IR.:** IR spectra were recorded in KBr disc on Shimadzu FT-IR 8300 spectrophotometer.

**Mass:** Mass spectra were taken on a Macro mass spectrometer (Waters) by electrospray method (ES).

\[
\text{4d}
\]

\(^1H\) NMR: (4d) (CDCl\(_3\)) \delta ppm: 2.37 (s, 3H, CH\(_3\)), 3.91 (s, 3H, OCH\(_3\)), 4.67 (d, J = 1.4 Hz, 2H, OCH\(_2\)), 5.32 (dd, J = 1.3 Hz & J = 10.3 Hz, 1H, =CH), 5.41 (dd, J = 1.4 Hz & J = 17.3 Hz, 1H, =CH), 6.04 (m, J = 17.3 Hz & J = 10.3 Hz, 1H, O-CH\(_2\)-CH=CH\(_2\)), 6.91 (s, 1H, Ar-H), 7.18 (s, 1H, Ar-H), 7.41 (d, J = 15.32 Hz, H\(_b\)), 7.90 (d, J = 16.16 Hz, H\(_a\)), 6.10-7.86 (m, 3H, Ar-H), 12.58 (s, 1H, -OH).

**IR.:** (4d) (cm\(^{-1}\)): 3427 (OH); 1654 (C=O); 1510 (C=C); 1020 (Ar-Cl).

**ES-MS** (m/z): 359.2 (M+1) and 361.2 (M+3)
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\[
\begin{align*}
\text{6b} \\
\text{1H NMR: (6b) (CDCl}_3\text{)} & \delta \text{ ppm: 2.17 (s, 3H, CH}_3\text{), 2.28 (s, 3H, CH}_3\text{), 3.76 (s, 3H, N-CH}_3\text{), 6.24 (dd, J = 3 Hz, 1H, Ar-H), 6.84 (d, J = 1.88 Hz, 1H, Ar-H), 6.91 (d, J = 2.72 Hz, 1H, Ar-H), 7.17 (d, 1H, Ar-H), 7.37 (d, 1H, Ar-H), 7.41 (d, J = 15.00 Hz, H}_b\text{), 7.86 (d, J = 14.96 Hz, H}_a\text{), 13.24 (s, 1H, -OH).} \\
\text{IR.: (6b) (cm}^{-1}\text{): 3211 (OH); 1631 (C=O); 1527 (CN).} \\
\text{ES-MS (m/z): 256.12 (M+1)}
\end{align*}
\]

\[
\begin{align*}
\text{10a} \\
\text{1H NMR: (10a) (CDCl}_3\text{)} & \delta \text{ ppm: 6.93 (dd, 1H, Ar-H), 6.95 (m, 2H, Ar-H), 7.10 (dd, 2H, Ar-H), 7.14 (dd, 2H, Ar-H), 7.26 (m, 1H, Ar-H), 7.31-7.69 (m, 5H, Ar-H), 7.95 (d, J = 15.64, H}_b\text{), 8.03 (d, J = 14.76, H}_a\text{), 12.64 (s, 1H, -OH).} \\
\text{IR.: (10a) (cm}^{-1}\text{): 3417 (OH); 1645 (C=O); 1546 (C=C).} \\
\text{ES-MS (m/z): 333.2 (M+1)}
\end{align*}
\]
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$^1$H NMR (Comp. 4d)

![H NMR spectrum](image1)

Mass (Comp. 4d)

![Mass spectrum](image2)
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IR (Comp. 4d)

$^1$H NMR (Comp. 6b)
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Mass (Comp. 6b)

IR (Comp. 6b)
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\(^1\text{H} \text{NMR (Comp. 10a)}\)

[Image of NMR spectrum]

\(\text{Mass (Comp. 10a)}\)

[Image of Mass spectrum]
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IR (Comp. 10a)
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1.6. References


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