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Chemical Synthesis of Chalcones by Claisen-Schmidt Condensation Reaction and Its Characterization

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Abstract*: *Many phytochemical compounds with chalcones as an important group, exhibits considered potential pharmacological activity. In recent years a variety of chalcones have been reported as anticancer chemopreventive compounds. Thus in this study we focused on synthesize of chalcone scaffold as a potential template for the design of various derivatives with potential pharmacological activities against cancer. In general, these chalcones are open chain flavonoids with two aromatic rings attached by a three carbon α .β unsaturated carbonyl system. In this present study, we employed Claisen-Schmidt condensation reaction to chemically synthesize the chalcones and characterized by FT-IT and XRD. In IR peak, C = O (Str) group, the C-H (aromatic) stretching, CH2 stretching and aromatic bending are observed. While, XRD analysis showed eleven different and important characteristic peaks with diffracted intensities from 0˚ to 30˚ at 2 theta angles.
Keywords*: *Chalcones, Claisen-Schmidt condensation reaction, FT-IR, XRD

I. INTRODUCTION

Cancer is one of the most dreadful diseases in the modern era, which is taking millions of life every year worldwide [1]. Day by day it becomes more dangerous as it can affect any age group and any ethnicity. In general, Cancer occurs by changing normal cells growth in an uncontrolled manner. This uncontrolled growth appears as lump known as tumour. If not treated, the tumor can cause problems in one or more of the following ways: Spreading into normal tissues nearby, causing pressure on other body structures, spreading to other parts of the body through the lymphatic system or bloodstream [2]. Now-a-days various types of cancers are reported every month that spread with various mechanisms. There are over 200 different types of cancers that occur in different types of body cells. The most frequently diagnosed cancers are bone, brain, breast, colon and skin cancers. There are many known causes of cancers like exposure to chemicals, drinking excess alcohol, excessive exposure to sunlight, and genetic differences [3].

Many research groups have reported the anticancer activities of chalcones and its derivatives. Also, they have been reported with various pharmacological activities such as antiviral, insecticidal and enzyme inhibitory properties [4]-[6]. Hence, these chalcones have become as one of the important constitute for the design of novel anticancer agents with promising therapeutic efficacy and also an crucial component for human cancer management. Chalcones are polyhydroxylated in the aryl ring and phenol group present in their basic structure shows radical quenching properties. Chalcones rich plant extracts or chalcone compounds are also raised great interest as drugs or food preservatives due to its different pharmacological properties [7].

many pharmaceutical compound syntheses, these chalcones are found as important intermediates. Though there is tremendous rogress in medicinal chemistry and many anti-bacterials, and anti-fungal compounds are available in market, the bacteria, fungi and viruses infections still remain as a major concern to public health sectors [8]. In line with this, the wide range of biological and chemical properties of chalcone and its derivatives have been mainly considered and the determination of their structural and chemical reactivity is majorly focused [9].

Chalcones or 1, 3 diphenyl-2-propene -1-one have been found to posses various biological activities like anti-bacterial, anti-fungal, anti-malarial, mutagenic, anti-cancer, anti-cytotoxic anti-leismenial anti-protozal, tyrosinase inhibitors, lipid peroxide inhibitors, immunosuppressive agent [10]. Many naturally occurring and chemically synthesized chalcones and its derivative have been extensively studied and reported as an important pharmaceutical molecule class for the design of novel potential compounds. Chalcone derivatives are widely screened for their anti-inflammatory activity, chemo preventive activity, cardiovascular disease, anticancer activity, cytotoxic activity, anti-prolifirative activity, antimalarial activity, antiviral activity and anti-HIV activity[11].

Thus in this research we propose to synthesize the scaffold of chalcones through Claisen-Schmidt condensation reaction and characterized by FT-IR and XRD.

II. MATERIALS AND METHODS

An All the chemicals and reagents used in the present study were of analytical grade and purchased from Sigma aldrich. The glassware were washed with double distilled water and dried in hot air oven

A. Synthesis of Chalcone

Chalcone scaffold was synthesized by using Claisen-Schmidt condensation reaction. In this base catalyzed condensation reaction, to a 250 ml flask with 10 ml rectified spirit, 0.01 mol of benzaldehyde and acetophenone was added and dissolved. Further, 10 ml NaOH (1g in 10ml water) was added drop wise and vigorously stirred for about 30 minutes on the magnetic stirrer until the solution becomes turbid and the temperature of the reaction mixture was maintained between 20-25˚ C by using cold water [12]-[13]. After 4-5 hours of stirring, 0.1-0.2N HCl was added to neutralize the reaction mixture and precipitation occurred. From this precipitate, the pure product was obtained by filtering and separating the solid precipitate from the reaction mixture and recrystallized to power form with rectified spirit

B. Characterization of Chalcones

1) *Fourier transforms Infrared Spectroscopy:* FT-IR spectral analysis was employed to identify the structures of synthesized compounds. The crystalline powered were analyzed by (Shimadzu) FT-IR Spectrometer. FT-IR spectrum was recorded over the range of (500-4000) cm-1 (with FTLA 2000 ABB) [14]. Fourier Transform Infra Red Spectroscopy (FTIR) spectra of the chalcone samples were recorded in KBr discs using FT/IR - 4100 JASKO model in the ratio of 1:100.
2) *X-ray Diffraction (XRD) :* XRD of chalcone samples (powder form) was measured at 2Θ range of 20–80 °, 8.04 keV energy and 1.54 A° wavelength by using XRD6000, (Shimadzu) of CuKa radiation with a scan rate of 20 /min and by using the Debye–Scherrer equation the crystallite sizes are determined [15]. In Debye–Scherrer equation, the mean crystallite size and the peak breadth are related.

$$D = k\lambda / \beta\cos\theta$$

Where,
D - crystallite domain size (average) upright to reflection planes ,
K - Constant
λ – Wavelength (X-ray)
β - Angular FWHM of the XRD peak at the diffraction angle
θ - Diffraction angle

III.RESULT AND DISCUSSION

Chalcones or benzylide and acetophenone are the important constituents of natural sources. It was first isolated from Chinese liquorice (Glycyrrhizae inflata) [16]. It has 1,3-diaryl-1-ones skeletal system, which was recognized as the main pharmacophore for chalcones. Chalcones and its derivatives are an important group of natural product and have been reported to possess varied biological and pharmalogical activity.

A. Synthesis Reaction

In Claisen-Schmidt condensation reaction, the α, β unsaturated ketones (called as chalcones) are formed by using the aliphatic or aromatic ketones to condense the aromatic aldehydes in the presence of aqueous alkali. In this study, the chalcone synthesis was carried out by using acetophenone and benzaldehyde in the presence of NaOH as a base. The scheme of the chalcone sysnthesis is given in figure.1.

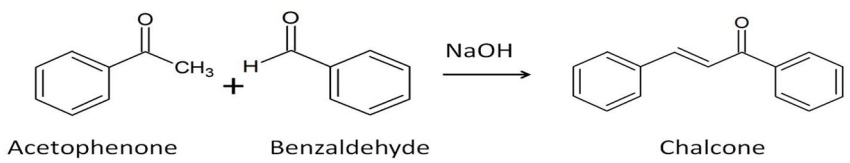

Fig. 1 The general scheme of chalcone synthesis by Claisen-Schmidt condensation reaction used in this study

As a first step in the reaction, a proton from α -carbon of acetophenone will be removed to by the base to form a resonance-stabilized enolate ion. In second step, the new carbon- carbon bond formation occurs as a result of nucleophilic enolates (α-carbon of acetophenone) attack over the electrophilic carbonyl carbon of benzaldehyde and form intermediate joined compound. Finally, protonation and deprotonation by hydroxide ion occurs to form an α, β -unsaturated ketone or chalcone as a light yellow solid (Figure.2).

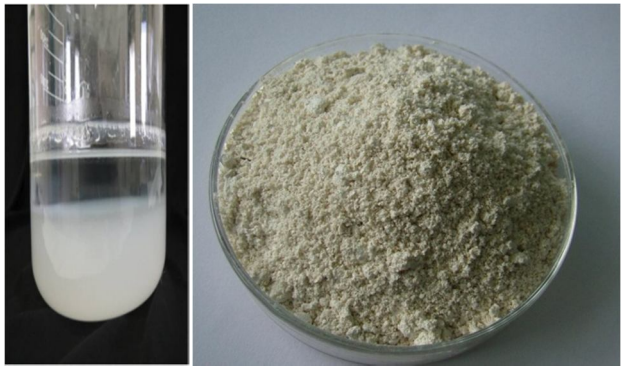

Figure.2. Precipitated and recrystallized chalcones

B. Fourier Transforms Infrared Spectroscopy

The vibrational and rotational mode information on molecules provided by FT-IR analysis is very important crucial for the identification and characterization of any substance. Generally, the organic compounds Infrared spectrum provides a unique fingerprint, which can be easily distinguished from the other compounds. Hence FTIR is considered an important technique and analysis measurement for identification and characterization of a compounds. The FTIR measurement was carried out to identify compound structure by determining their vibrations peaks identified at defined wave numbers.

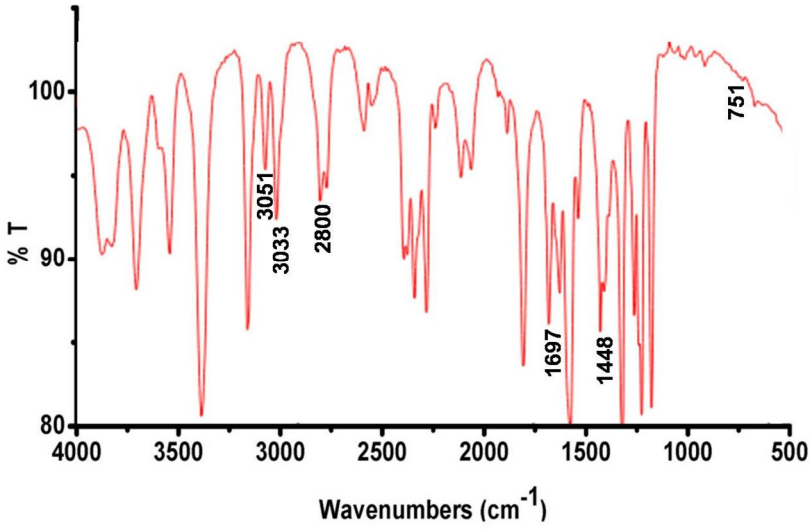

Figure.3 The FTIR spectrum of the chalcone

The IR spectrum of chalcone (Figure.3) manifests prominent absorption bands located at 3051, 3033, 2800, 1697, 1448 and 751cm−1. This result showed similarity with those reported by Mandge et al, 2007 and Jain et al, 2014 [15,16]. The IR peak at 1697 cm-1 suggests the presence of C = O (Str) group and the peak at 3051cm-1 indicates the presence of C-H (aromatic) streching. The IR peak at 2800 cm-1 indicates the presence of CH2 streching and peak at 751 cm-1 represents the presence of aromatic bending.

C. X-ray Diffraction

X-ray diffraction is used as analytical tool for the identification and quantitative determination of compound at various phases including crystalline forms, powder and solid samples. The diffraction pattern is the resulted when the repeat distance of a regular structure is about the same as the wavelength. The crystalline nature of the powder chalcone sample is confirmed by XRD analysis.

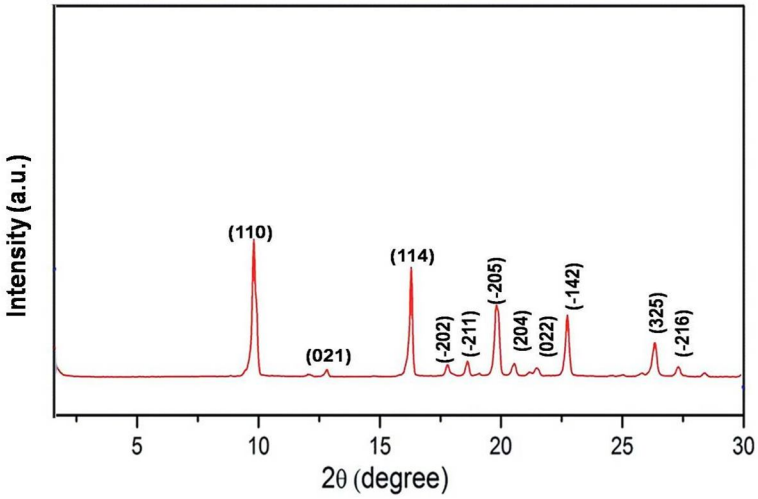

Figure.4: The XRD pattern of chalcone

The Braggs reflections indexed on the basis of the cubic structure of chalcone are determined. The diffracted intensities were recorded from 0˚ to 30˚ at 2 theta angles (Figure. 4). Eleven different and important characteristic peaks were observed at the 2 h of 10˚, 12.8˚, 16.2˚,17.9˚, 18.5˚, 19.9˚, 20.8˚, 21.6˚, 22.9˚, 26.2˚ and 27.5˚ that correspond to (110), (021), (114), (-202), (-211), (-205), (204), (022), (-142), (325) and (-216) planes, respectively. Thus the synthesized and characterized chalcone might be used as a scaffold to design and synthesize various chalcone derivatives and can be explored further for their various pharmacophore activities.

IV. CONCLUSIONS

Chalcones are considered to be an important class of anti-cancer agents with promising therapeutic efficacy and management of human cancers. In this study, Claisen-Schmidt condensation reaction was used to synthesize chalcones and characterized by FTIR abd XRD. The IR peak at 1697 cm-1 suggested the presence of C = O (Str) group and the peak at 3051cm-1 indicated the presence of C-H (aromatic) stretching. The IR peak at 2800 cm-1 indicated the presence of CH2 stretching and peak at 751 cm-1 represented the presence of aromatic bending. The X-ray diffracted intensities were recorded from 0˚ to 30˚ at 2 theta angles with eleven different and important characteristic peaks. Thus the chalcone synthesized can be explored further for their various pharmacophore activities.

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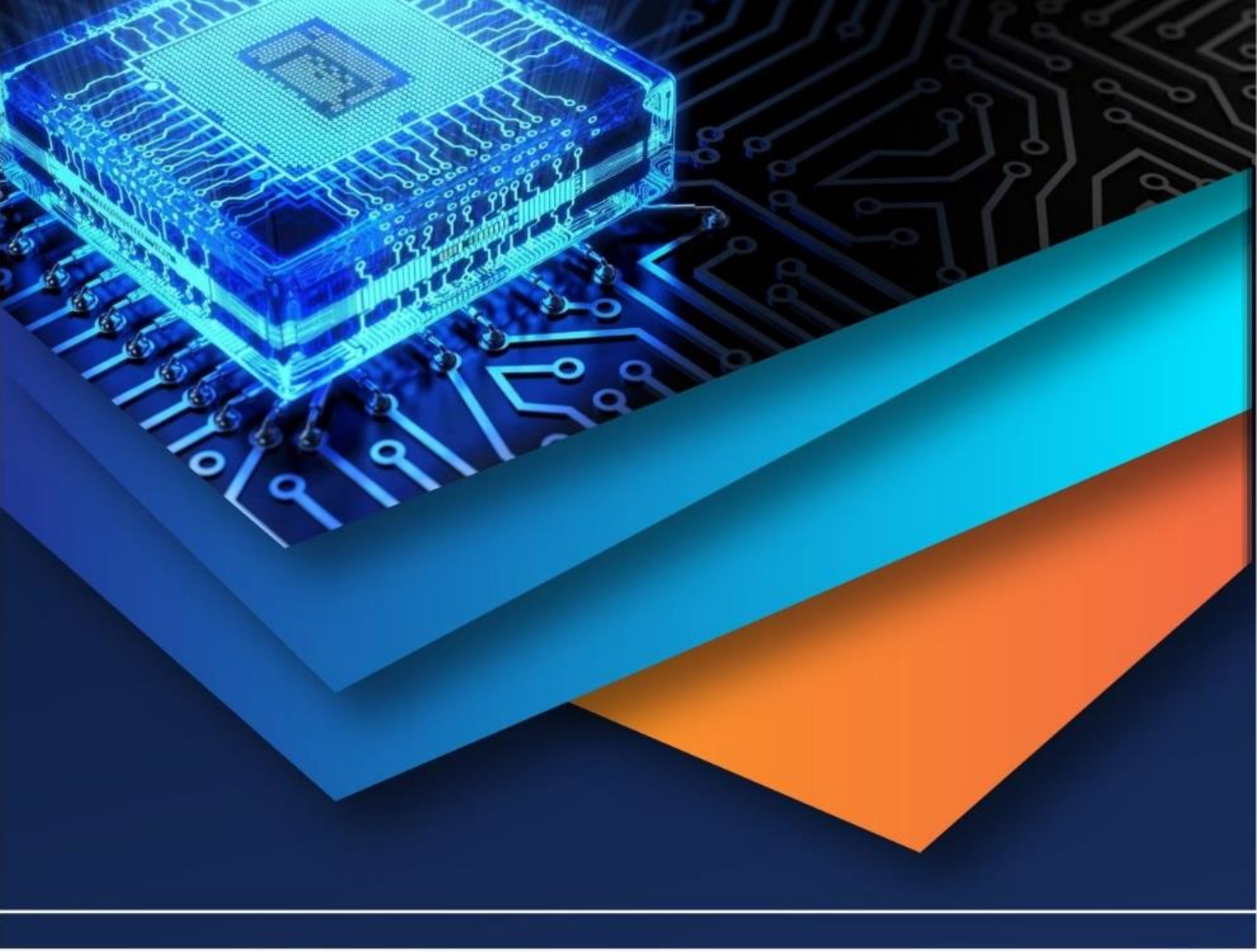

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