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THE CHEMISTRY OF QUINOLINYL CHALCONES

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Abstract: **In this article, the chemistry of Quinolinyl Chalcones has been laid bare and shows that Quinolinyl Chalcones is a derivative of Chalcones and one of the secondary metabolites of natural products. The structural elucidation by IR (Infrared), NMR (Nuclear Magnetic Resonance), MS (Mass Spectrometry) of Quinolinyl Chalcones are concisely studied and reveal the Canonical structures of various Quinolinyl compounds. This article went further to explain, the different methods (Grinding Conventional heating and Ultrasound-Assisted) of Quinolinyl Chalcones synthesis, with comparison made of Conventional heating and Ultrasound-Assisted methods. More so, the diverse applications of Quinolinyl Chalcones in the treatment of various diseases such as HIV, Inflammatory, Malaria, Bacteria and Cancer etc. are extensively dealt with, which is supported by the Structural-Activity Relationship (SAR) of the compound (ie the arrangement and distribution of functional groups).**

Keywords: **Quinolines, Chalcones, Quinolinyl Chalcones, Structural Elucidation, Synthesis, Structural-Activity Relationship, Pharmacological Applications.**

1. INTRODUCTION

In recent years, problems of multi-drug resistant on micro-organism have reached an alarming level in many countries around the world. A number of recent clinical reports have described the increasing occurrence of meticillin-resistant *S. aureus* and other antibiotic-resistant human pathogenic microorganisms in United State and European countries. Infections caused by those micro-organisms pose a serious challenge to the medical community and there is need for an effective therapy which will afford a novel antimicrobial agent (Kishor *et. al.* 2008).

1.1 Chalcones

Chalcones are a class of α, β- unsaturated carbonyl compounds that form the central core for a variety of naturally occurring biologically active compounds. They exhibit tremendous potential to act as pharmacological agents. Besides their various pharmacological activities, Chalcones have been explored for different optical applications including second harmonic generation materials in non-linear optics, fluorescent probe for sensing different molecules. Chalcones (trans-1,3-diaryl-2- propen-1-ones) (Figure 1.0) is a biosynthetic product of the shikimate path way, belonging to flavonoids family and are precursors of open chain flavonoids and iso-flavonoids which are abundant in edible plants. Chalcones are also key precursor in the synthesis of many biologically important heterocyclic such compounds as benzothiienzepime, pyrazolines, 1,4-diketones, and flavones. Thus, the synthesis of Chalcones has generated vast interest organic as well as for medicinal chemists. The traditional methods for the synthesis of 1,3- diaryl- 2 – propenones involves the use of strong base such as NaOH, KOH, $Ba(OH)_2$, hydrotalcities, LiHMDS (Lithium bis trimethylsilyl) amide, calcined $NaNO_3$ natural phosphate (Rahman, 2011).

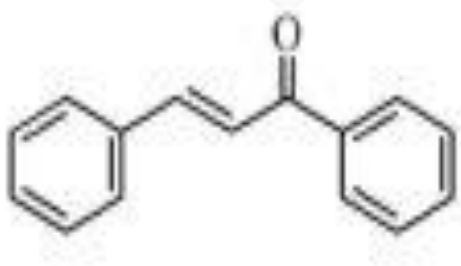


Figure 1.0: Structure of a Chalcones compound

1.2 QUINOLINES

Quinolines are important compounds because of their bioactive properties and medicinal uses such as antimalarial (Larsen *et al.*, 1996), anti-inflammatory (Chen *et al*., 2001) antiasthmatic (Roma *et al.*, 2000), antibacterial (Dube *et al.*, 1998) and tyrosine kinase inhibiting agents (Billker *et al*., 1998). Quinoline was discovered in 1842 by Gerhardt as a result of the drastic decomposition of quinine and of Cinchonine antedates. The structure of a Quinoline compound is as shown below, figure 1.1.

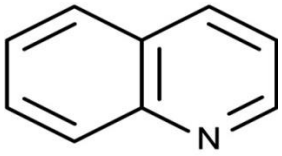
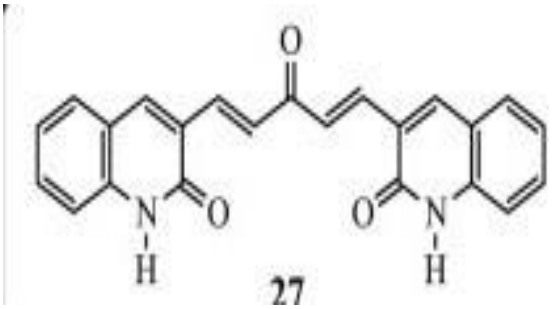


Figure 1.1: Structure of Quinoline compound

1.3 Quinolinyl Chalcones

Quinolinyl Chalcones is a derivative of Chalcones and one of the secondary metabolites of natural product. They are medicinally important. Many have been reported to possess antimalarial, antibacterial and antifungal properties (Katritzkey, 1984). Anticancer properties of some Quinolinyl Chalcones have also been reported in the literature (Rezig *et al* 2000; Ducki *et al*., 1996).

Quinolinyl Chalcones are of particular interest for various studies because of their vital role as precursor in the studies of biosynthesis of flavonoids which are abundantly available in plant kingdom. These bichromophoric molecules separated by a Keto-vinyl chain are very useful as substrate for the synthesis of biologically important heterocyclic compounds like cyclohexenone and pyrazoline derivatives. The structure of Quinolinyl Chalcone compound is as shown below, Figure 1.2.



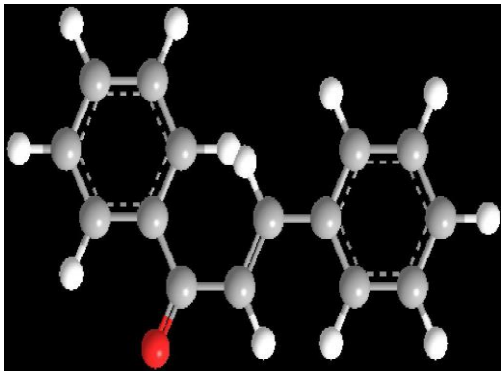
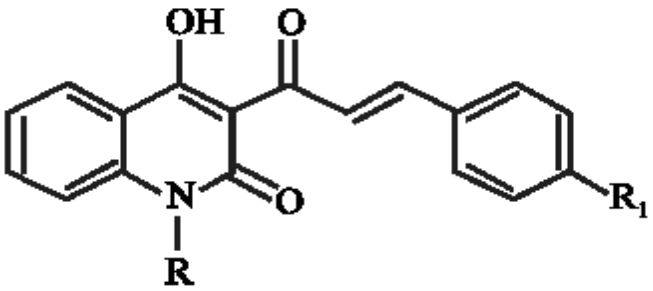


Figure 1.2: Quinolinyl Chalcones

2. THE STRUCTURAL ELUCIDATION OF QUINOLINYL CHALCONE COMPOUND



R= CH_3, C_2H_5, Ph

R1 = H, Cl, OCH_3

Figure 2.2: Quinolinyl Chalcone

The structure of the Quinolinyl Chalcone illustrated above was elucidated by some basic important spectroscopic data such as IR (Infrared), NMR (Nuclear Magnetic Resonance), MS (Mass Spectrometry) analysis. It was observed that in ^{1}HNMR spectra of the compound in figure 2.2, α, β unsaturated enone system protons appeared as two doublets around δ8.6ppm and δ7.8 ppm for its H_{β} and Hα respectively, with coupling constant between 15 – 16Hz (Muhammad *et. al*., 2007). The coupling constant predicts that Chalcone derivatives (Quinolinyl Chalcones) are trans- isomers. The other peaks appeared in the expected region and the number of protons is in accordance with the expected protons. Additional support elucidating the structure of figure 2.2 is obtained from ^{13}CNMR spectra. The appearance of peak around δ194ppm indicates that α, β unsaturated carbonyl carbon is present in the compound. M+1 peak observed in mass spectra is a promising peak in the compound (Muhammad *et. al*., 2007).

2.1 Canonical Structure of Quinolinyl Chalcones

One difference between the isomeric Quinolinyl Chalcones can be traced to their electronic character as shown in figures 2.3 and 2.4, the electron withdrawing effect of the carbonyl oxygen can be transmitted by conjugation to the azomethine nitrogen in 4- Quinolinyl Chalcones. Such an effect is not possible in the 3- Quinolinyl Chalcones as shown in figure 2.4 (Liu, 2003).

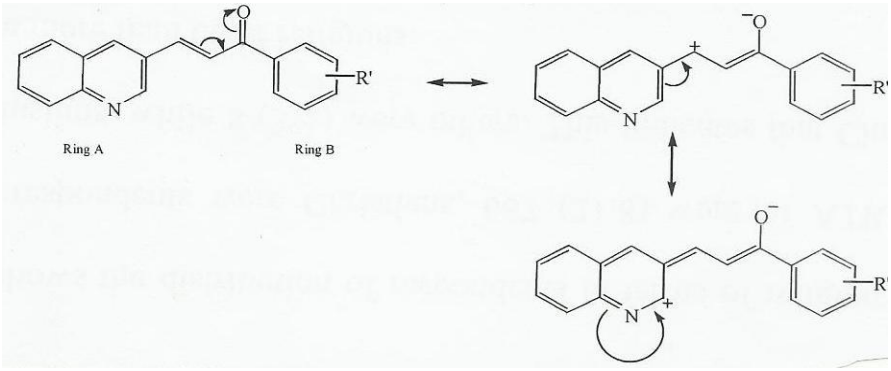


Figure 2.3: The canonical structures of 3- Quinolinyl Chalcones

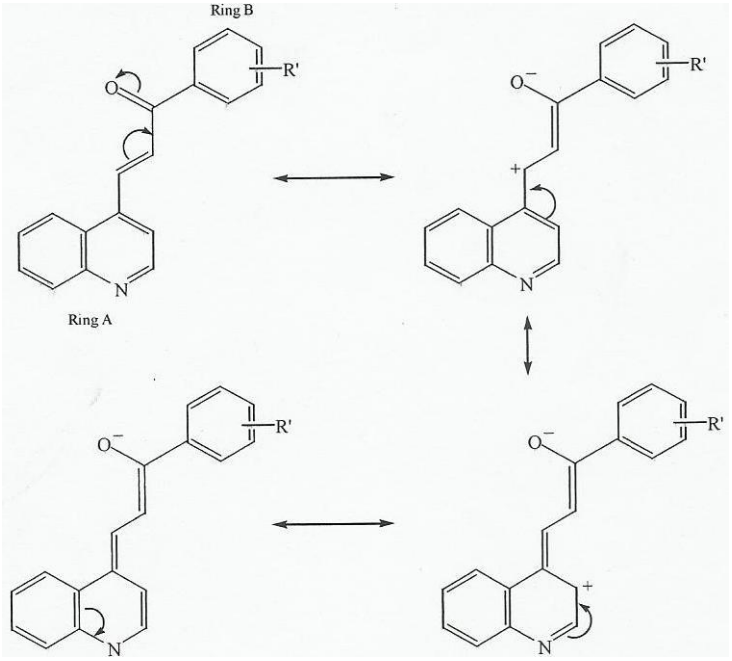


Figure 2.4: The canonical structures of 4- Quinolinyl Chalcones

The chemical shifts of the carbonyl carbon of trimethoxychalcones bearing 3-quinolinyl and 4-quinolinyl rings A suggested that differences do exist among the isomeric Quinolinyl Chalcones. The chemical shift differences (Δδ = δR - δH) of the carbonyl carbon in figures, 2.3 and 2.4 were found to be –1.329 ppm and 0.040 ppm respectively. Negative sign associated with figure 2.3 suggests that the 3-quinolinyl ring has a stronger electron withdrawing effect, which would

be the case if electron delocalization takes place. On the other hand, such an effect is absent in the 4-quinolinyl derivative and this might explain its small positive Δδ value. Although not clearly understood at this juncture, it is apparent that differences in antimalarial activities of the isomeric Quinolinyl Chalcones can be traced to their electronic properties (Liu, 2003).

2.2 Synthesis of Quinolinyl Chalcones

Generally, Quinolinyl Chalcones have been synthesized via Claisen-Schmidt condensation between acetophenones and benzaldehydes and a number of condensing agents such as Sodium ethoxide, zinc chloride, potassium phosphate, basic alumina, sodium methoxide in methanol and lewis acids such as $AlCl_3$, BF_3 and Mg-Al-OBu have been used. Recently, the use of NaOH/Ethanol, KOH/Ethanol, ultrasonic and microwave conditions has also been reported in the literature (Sharma, 2013).

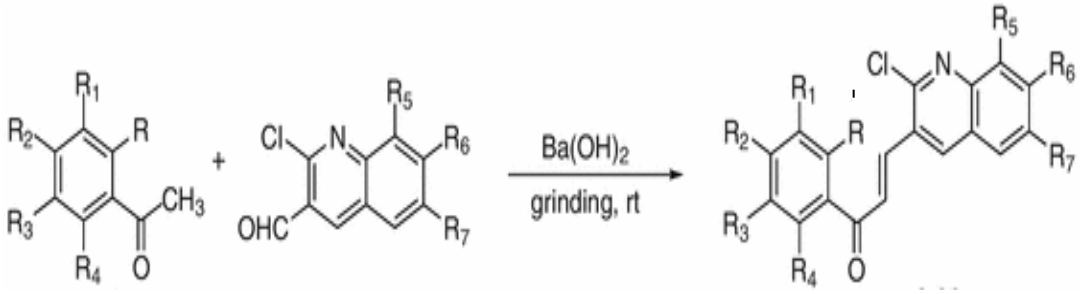
Some of the above-mentioned conditions possess shortcomings, such as the use of harsh and hazardous chemicals, mainly organic solvents, longer reaction times, elevated temperatures, poor yields and the formation of side products. Due to their volatile nature, these organic solvents affect human health and cause extreme damage to our environment. These shortcomings have led researchers to the development of a safe, environmentally benign and more efficient method for the synthesis of Quinolinyl Chalcones. However, the grinding technique has received much attention due to its operational simplicity, and it is considered to be an important tool to carry out reactions under solvent-free conditions with minimum cost and maximum yield as compared to conventional methods (Sharma, 2013)

2.2.1 Techniques for the Synthesis of Quinolinyl Chalcone

2.2.1.1 Grinding Method

Grinding technique is an efficient and eco-friendly synthesis of Quinolinyl Chalcones under solvent-free conditions at room temperature involving grinding of quinoline-3-carbaldehyde and acetophenones with anhydrous barium hydroxide (Sharma, 2013).

The equation of reaction for the synthesis of Quinolinyl Chalcones by grinding technique from the reaction between substituted acetophenones and quinoline-3-carbaldehyde using aqueous barium hydroxide or aqueous sodium hydroxide as catalyst is as shown in equation 2.1.



Equation 2.1: Synthesis of Quinolinyl Chalcone by grinding technique

2.2.1.2 Conventional Heating Method

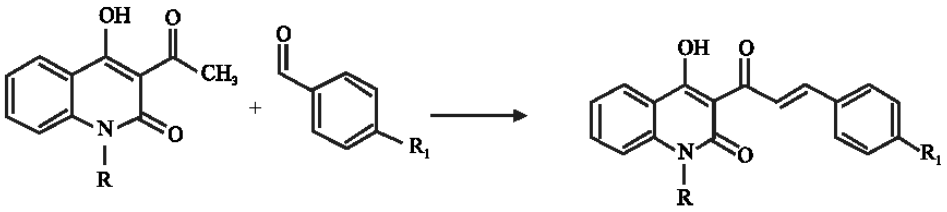
In a conventional heating method, a mixture of N-substituted-3-acetyl-4-hydroxyquinolin-2(1H)-one, aromatic aldehyde, drops of piperidine and 1-butanol is refluxed for about 6 hours. The precipitates are filtered under suction, dried and the crude product is purified by column-chromatography after which the percentage yield is determined (Muhammed *et. al*., 2007).

2.2.1.3 Ultrasound-Assisted Method

In this method, a mixture of N-substituted-3-acetyl-4-hydroxyquinolin-2(1H)-one, aromatic aldehyde, drops of piperidine and 1-butanol and neutral alumina is stirred well for 5 minutes and the solvent is removed under suction. The semi-dried material is heated and irradiated in ultrasound bath at 60^0C. Thin-layer chromatography (TLC) is used to monitor the completion of the reaction. The reaction mixture is extracted with chloroform and drying is carried out over anhydrous magnesium sulphate and the crude product is purified by column chromatography after which the percentage yield is determined (Muhammed *et. al*., 2007).

2.3 Comparison of Conventional Heating and Ultrasound Assisted Methods

The two methods are compared with reference to reaction time, temperature and percentage yields. By the use of ultrasound-assisted method, reaction time is reduced marvelously from 6 hours to 70-90 minutes and product yields increased approximately in two folds. Energetically, preparation of Quinolinyl Chalcone derivatives by using ultrasound-assisted method is more feasible than the conventional heating (Muhammed *et. al*., 2007). Equation of reaction for the conventional heating and ultrasound-assisted methods is as shown in equation 2.2.



$R = CH_3, C_2H_5, Ph$
$R_1 = H, Cl, OCH_3$

Equation 2.2: The conventional heating and ultrasound-assisted synthesis of Quinolinyl Chalcone.

Structures of some synthesized Quinolinyl Chalcones are illustrated below,

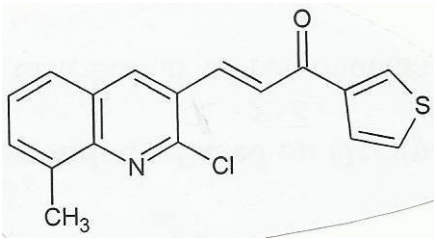


Figure 2.3: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl) -1-thien-3-ylprop-2-en- 1-one

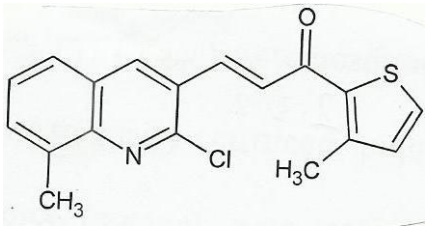


Figure 2.4: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl)-1-(3-methylthien-2- yl) prop-2-en-1-one

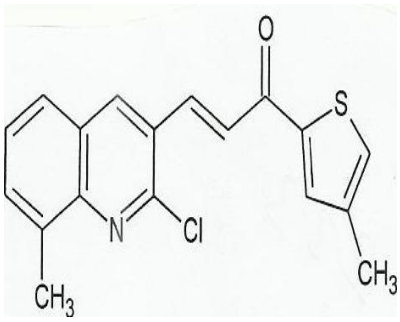


Figure 2.5: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl) -1-(4-methylthien-2- yl) prop-2-en-1-one

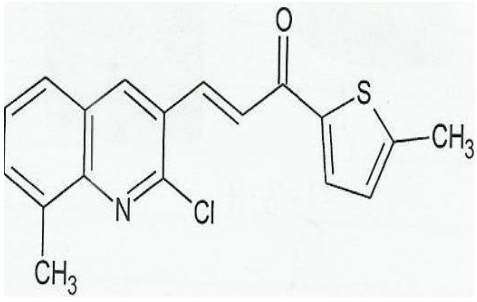


Figure 2.6: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl)-1-(5-methylthien-2- yl) prop-2-en-1-one

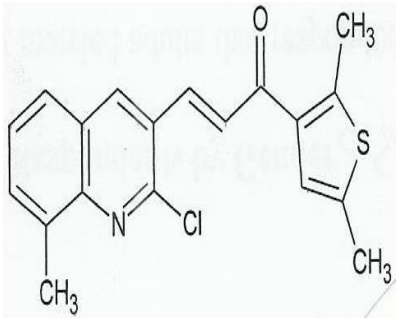


Figure 2.7: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl) -1-(2, 5-dimethylthien- 3-yl) prop-2-en-1-one

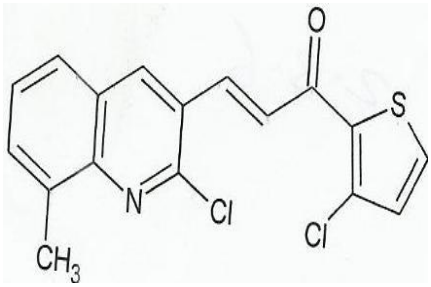


Figure 2.8: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl)-1-(3-chlorothien-2- yl) prop-2-en-1-one

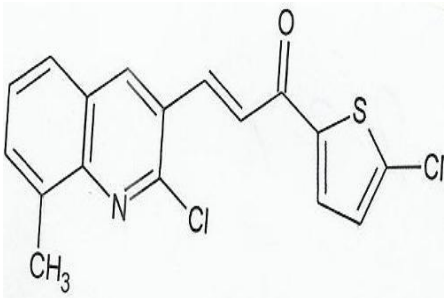


Figure 2.9: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl) -1-(5-chlorothien-2- yl) prop-2-en-1-one

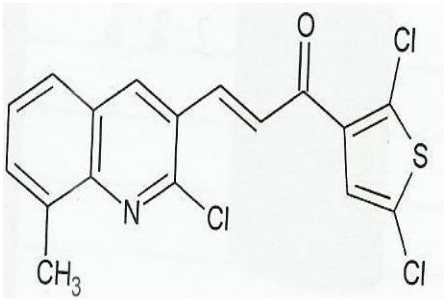


Figure 2.10: (2*E*)-3-(2-Chloro-8-methylquinolin-3-yl)-1-(2, 5-dichlorothien-3- yl)prop-2-en-1-one

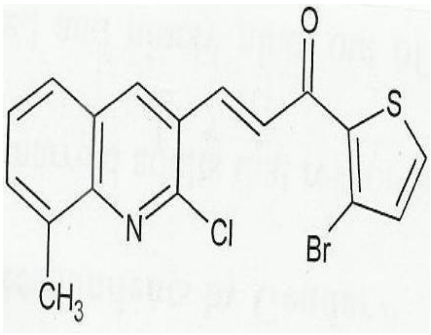


Figure 2.11: (2*E*)-1-(3-Bromothien-2-yl)-3-(2-chloro-8-methylquinolin-3- yl) prop-2-en-1-one

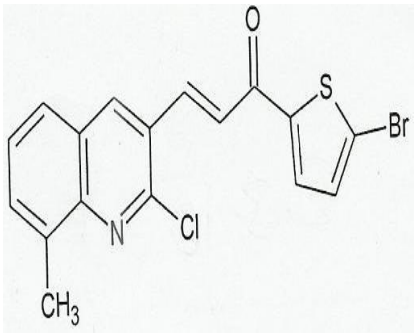


Figure 2.12: (2*E*)-1-(5-Bromothien-2-yl)-3-(2-chloro-8-methylquinolin-3-yl) prop-2-en-1-one

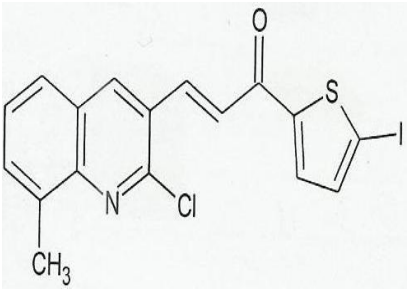


Figure 2.13: (2*E*)-3-(2-Chloro-8-methylquinolin -3-yl)-1-(5-iodothien-2- yl)prop-2-en-1-one

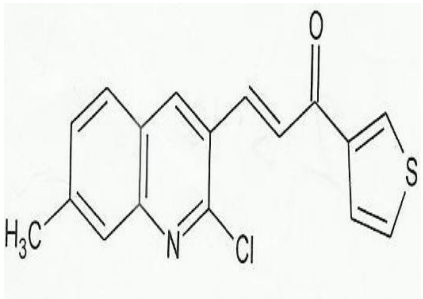


Figure 2.14: (2*E*)-3-(2-Chloro-7-methylquinolin-3-yl)-1-thien-3-ylprop-2- en-1-one

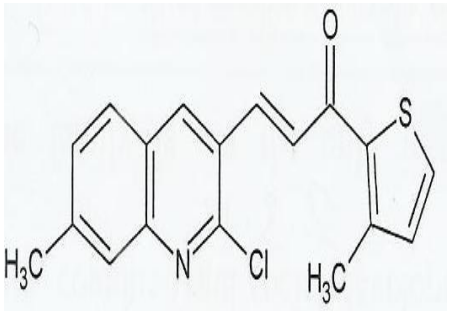


Figure 2.15: (2*E*)-3-(2-Chloro-7-methylquinolin -3-yl)-1-(3-methylthien-2- yl) prop-2-en-1-one

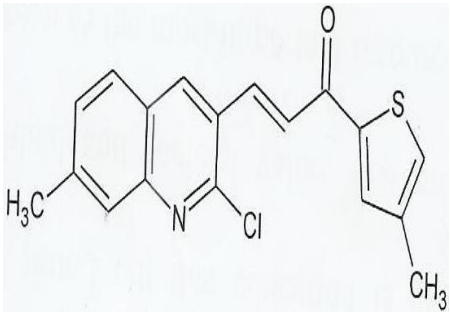


Figure 2.16: (2*E*)-3-(2-Chloro-7-methylquinolin-3-yl)-1-(4-methylthien-2-yl) prop-2-en-1-one

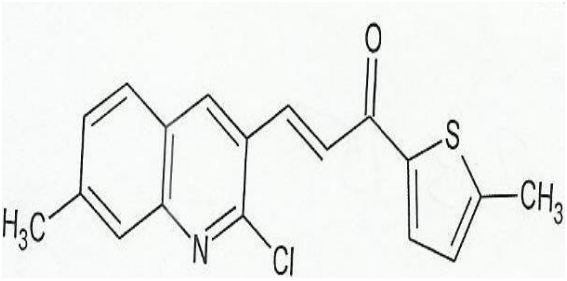


Figure 2.17: (2*E*)-3-(2-Chloro-7-methylquinolin-3-yl)-1-(5-methylthien-2- yl) prop-2-en-1-one

3. APPLICATIONS OF QUINOLINYL CHALCONES

3.1 Pharmacological Application

Quinolinyl Chalcones are groups of secondary metabolites which have attracted increasing attention owing to their numerous potential pharmacological applications. They have displayed a broad spectrum of pharmacological activities. Modifications in their structure have offered a high degree of diversity that has proven useful for the development of new medicinal agents having improved potency and lesser toxicity (Rahman, 2011).

3.1.1 Quinolinyl Chalcone as Anti-HIV compound

A series of Quinolinyl Chalcones have been synthesized by using Claisen-Schmidt condensation of 6-methoxy-2-chloroquinoline-3-carbaldehyde and 6, 7-dimethoxy-2-chloroquinoline-3-carboxaldehyde with substituted acetophenones for potential anti-HIV activity (Gupta *et. al*., 2010)

Acquired Immune Deficiency Syndrome (AIDS) caused by Human Immunodeficiency Virus (HIV) is a global health threat and the leading cause of deaths owing to infectious disease. The urgent need for anti-HIV/AIDS drugs is a global concern. In addition to obvious economical and commercial hurdles, HIV/AIDS patients are faced with multifarious difficulties associated with the currently approved HIV drugs. Adverse effects, the emergence of drug resistance and the narrow spectrum of activity have limited the therapeutic usefulness of the various reverse transcriptase and protease inhibitors that are currently available in the market. This has driven many scientists to look for new anti-retroviral with better efficacy, safety and affordability (Gupta *et. al*., 2010).

Series of literatures have revealed that most of the promising naturally derived anti-HIV compounds are flavonoids, coumarins, terpenoids, alkaloids, polyphenols, polysaccharides or proteins. In the light of these findings, the synthesis of some Quinolinyl Chalcones incorporating the 6-methoxy-2-chloroquinoline-3-carbaldehyde and 6, 7-dimethoxy-2-chloroquinoline-3-carboxaldehyde nucleus has been undertaken in order to assess their anti-HIV profile (Gupta *et. al*., 2010).

3.1.2 Quinolinyl Chalcone as Anti-Inflammatory compound

Vijay *et. al.*, (2010) synthesized Quinolinyl Chalcones and investigated their anti-inflammatory activity using carrageenan induced paw oedema method. Acute oral toxicity tests were performed for all the synthesized compounds as per organization of economic co-operation and development (OECD) guidelines. Statistical analysis (ANOVA) followed by Dunnett's test was performed for anti-inflammatory activity to ascertain the significance of the exhibited activity (Vijay *et. al.*, 2010).

The anti-inflammatory activity was performed by carrageenan-induced acute paw oedema method in rats. Albino rats of either sex, weighing between 300-250g were used in the experiment. Indomethacin (20mg/kg) was administered as standard. The test compounds were orally administered to the animals and after one hour of treatment, 0.1ml of 1% carrageenan suspension was injected subcutaneously into the subplantar tissue of the right hind paw and 0.1ml of saline was injected into the subplantar tissue of the left hind paw. The thickness of both paws of each rat, lower and upper surface was measured using Zeithin's constant load lever. The paw thickness was determined at 1,2,3,4 and 5 hours after induction of inflammation (Vijay *et. al.*, 2010)

3.1.3 Quinolinyl Chalcone as Antibacterial compound

A series of Quinolinyl Chalcones were prepared and tested by Chikhalia *et. al.*, 2008 for their *in vitro* antimicrobial activity against four strains of bacteria; *S. aureus, B. subtillis, E. coli, S. typhosa* (Gram positive and gram negative)

strains using a microdilution procedure. Five compounds of ten series showed high *in vitro* antibacterial activity. One compound showed excellent activity against *Staphylococcus aureus* and *Bacillus subtillis*. Another showed good activity against *Bacillus subtillis.* The third, the fourth and the fifth compounds showed excellent activity against *Bacillus subtillis*, *Escherichia coli* and *Salmonella typhosa*. The presence of more than one electron-withdrawing group on the aromatic ring generally increase the antibacterial activity compared to compounds with electron-donating groups (Chikhalia *et. al.*, 2008).

3.1.4 Quinolinyl Chalcone as Anticancer compound

Prasher *et. al.*, 2007 evaluated a series of new 6-quinolinyl and Quinolinyl N-oxide Chalcones which were efficiently prepared by the synthesis of all Chalcones tested by minimal inhibitory concentration (MIC) against three species of *Candida*, *Cryptococcus gattii* and *paracoccidioides brasiliensis*. The effect of these compounds was also tested on the survival and growth of the human cancer cell lines (melanoma), (breast), (renal) and leukemic cells and Jurkat. The Leukemic cells compounds illustrated in figure 3.1 showed the best activity. (Prashar *et. al.*, 2012)

3.1.5 Quinolinyl Chalcone as Antimalaria

The antimalarial activity of Quinolinyl Chalcones was first noted when a natural product isolated from Chinese liquorice roots, was reported to exhibit potential antimalarial activity. Later on, it was observed that good antimalarial effects were exerted by alkoxylated Chalcones with polar B rings, in particular, those substituted with electron withdrawing groups or those incorporating Quinoline rings. Since then, a number of Chalcones derivatives containing Quinoline and other heteroaryl moiety have been synthesized and evaluated for potential antimalarial activity. Charris et al., 2011 have reported a series of E-2-quinolinylbenzocy chalcanones and evaluated their activity to inhibit β-hematin formation and the hydrolysis of haemoglobin *in vitro* and their efficacy in rodent plasmodium berghei inhibition of β-hematin formation was minimal when a hydrogen or methoxy groups were present on the position (8) of the Quinoline and position (4) of the indanone ring. They also described the synthesis of Quinolinyl Chalcones having dimethoxy group (Begum *et. al.*, 2010).

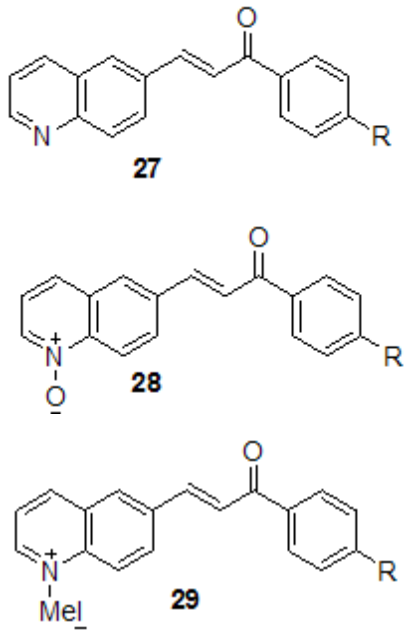


Figure 3.1: Structures of best active leukemic cell compounds

Also, a series of Quinolinyl and chloroqinolinyl Chalcones were synthesized by Vijay et. al., 2010 to study the effect of Quinoline moiety present in Chalcones on their anticancer activities (Urmila, 2012). The selected Chalcones were screened for their *in vitro* anticancer potential on cell lines which was based on the appearance of highly coloured blue formazan product by mitochondrial reduction of yellow tetrazolium dye and noted the results as % inhibition of cell growth. Quinolinyl Chalcones possessing high anticancer activity were found to exhibit appreciable reduction in paw edema (which is beneficial for cancer treatment) up to 81.78% at a concentration level of 20mg/kg as compared to the standard (82%, 10mg/kg) (Urmila, 2012).

3.2 Structure Activity Relationship (SAR) of Quinolinyl Chalcone

The structure activity relationship of Quinolinyl Chalcones has to do with the relationship between the chemical structure of Quinolinyl Chalcone and its biological or pharmacological activity. They are often joined together because they have structural characteristics in common including shape size, stereochemical arrangement and distribution of functional groups.

3.3 Conclusion/Summary

This article has showed that Quinolinyl Chalcones are versatile, having diverse applications most importantly their pharmacological activity; anti-HIV, anti-inflammatory, antibacterial, anticancer e.t.c. The structure activity relationship has supported the pharmacological potency of Quinolinyl Chalcones on the basis of their chemical structure which has to do with the arrangement and distribution of functional groups. The vital information reported in the literature has supported the utilization by researchers' different design and development of potent Quinolinyl Chalcones as drugs in the treatment of various diseases.

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