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Synthesis and Characterization of Schiff Base and Their Chalcone Derived from 3-Amino Acetophenone

A.R. Mehetre[1], S. R. Deshmukh[2] and V. N. Bhosale[3]
[1,3]*Shivaji Arts, Commerce and Science college Kannad. Dist-Aurangabad (MS)India.*
[2]*R.M.G.Arts and Commerce college Saoli, Dist Chandrapur (MS)India.*

Abstract: Chalcone consisting unsaturated carbonyl group and C=N bond were synthesized from Schiff base. Schiff base [S_1] was prepared by condensation of 3-Amino Acetophenone with 4-Nitrobenzalehyde. Chalcone[C_1] has been prepared from synthesized Schiff base[S_1] was mixed with 4-Nitrobenzalehyde in presence of base(NaOH). Synthesized compounds were characterized by IR and NMR.
Keywords: 3-Amino Acetophenone , 4-Nitrobenzalehyde , Synthesis, Schiff base, Chalcone.

I. INTRODUCTION

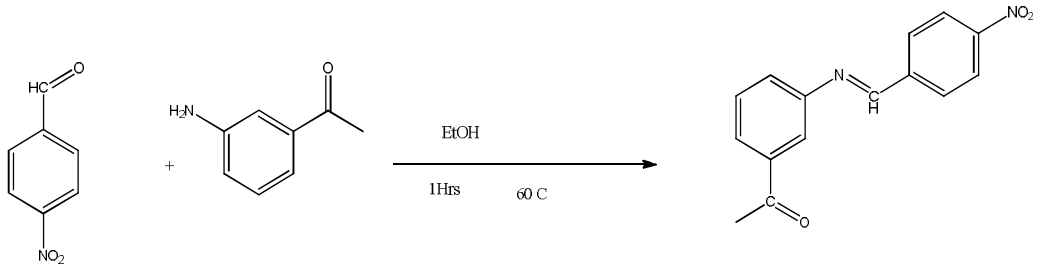
Schiff base compounds have been shown to be promising leads for the synthesis of efficient antimicrobial agents because these compounds exhibited broad range of biological activities. Schiff base compounds and their Chalcone, metal complexes were shows properties like antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral and antipyretic[1].
The molecular structure of the compounds of Schiff bases determines mode of interaction and inhibition effectiveness with bacteria and fungi. Recent advances in this field will require analyses of structure activity relationships of Schiff bases along with account of the mechanism of action of these compounds[2]. In particular, Schiff bases composed of nitrobenzaldehyde are very promising in the search of new functional materials.
Chalcones impart main role in synthesizing a range of therapeutic compounds for the treatment of various diseases. Chalcone compounds shown worth mentioning therapeutic efficacy. Chalcone based derivatives have gained attention, they own simple structures and diverse pharmacological actions. In Chalcones presence of a reactive α,β-unsaturated keto function is found to be responsible for their pharmacological activities. Variety of chalcones have been reviewed for their a wide variety of pharmacological effects including antimalarial, antileishmanial, analgesic, antiulcerative, antihyperglycemic, antioxidant antiplatelet, antiviral, antibacterial, antitubercular, antifungal, antiinvasive, enzyme inhibitory, cytotoxic and antitumor properties[3-4].

II. MATERIALS AND METHODS

All the chemicals purchased from S.D.Fine chemicals were used. 1H NMR spectra were recorded on a Bruker-AV 400 MHz spectrometer using chloroform-d as solvents and TMS as an internal standard. The infrared spectra (4000–400 cm^{-1}) were recorded by using KBr pellet on Shimadzu IR Affinity-1.

A. Experimental:

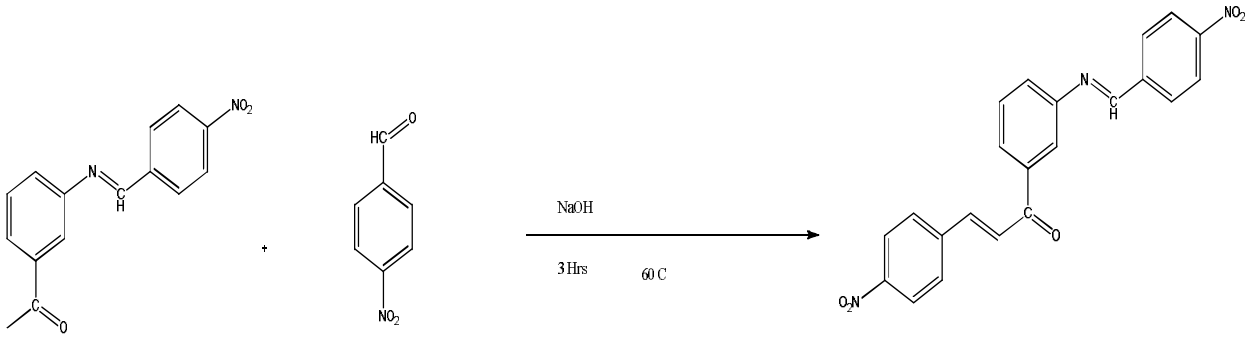
1) Synthesis of Schiff base [S_1]: Schiff base ((E)-1-(3-((4-nitrobenzylidene)amino)phenyl)ethanone) prepared from 3-Amino Acetophenone(0.001mole) dissolved in ethanol and mixed with 4-Nitrobenzalehyde(0.001mole) dissolved in ethanol. Mixed solution stirred for one hour at 60* c and allow solution to cool down yellow solid was obtained. Product was filtered and dried. Product was recrystallized from ethanol.74.55% reaction yield was obtained.
IR (KBr, cm–1): [C=O: 1665], [N=CH: 1626],[CH:3004].
1H-NMR (_, ppm): 7.19–7.56 (d&s3H&1H,Ar–H),8-8.12(m,4H,Ar-H),8.54 (s,1H, N=CH),2.10(s,3H-CH_3).
Reaction:



(E)-1-(3-((4-nitrobenzylidene) amino) phenyl) ethanone

2)Synthesis of Chalcone: Chalcone ((E)-1-(3-((E)-(4-nitrobenzylidene)amino)phenyl)-3-(4-nitrophenyl)prop-2-en-1-one) [C_1] has been prepared from synthesized Schiff base[S_1] was mixed with 4-Nitrobenzalehyde in presence of base(NaOH)in ethanol solvent. Mixed solution stirred continuously for three hour at 60* c. Reaction was carried out 1:1ratio at 0.001mole.
IR (KBr,cm^{-1}): [C=N: 1625],[C=O:1662],[C=C:1597],[CH:3072].
1H-NMR (_, ppm): 8.64(s,1H,N=CH),8.27-8.39(m,4H,Ar-H),8.10-8.14(d,2H,Ar-H),7.95-7.99(d,2H,Ar-H),7.84-7.91(m,4H,Ar-H) and 7.57-7.65(dd,2H,olefinic).

B. Reaction



(E)-1-(3-((E)-(4-nitrobenzylidene)amino)phenyl)-3-(4-nitrophenyl)prop-2-en-1-one

III. RESULT AND DISCUSSION

Table 1: Physical Parameters and IR Data [υ max ($cm^{-}1$)] of Schiff base [S_1] and Chalcone[C_1].

Sr.No.	Name	Molecular formula	Molecular weight	Melting point-^{0}C	Yield	Functional group	IR value Cm^{-1}
1	Schiff base[S1]	$C_{15}H_{12}N_2O_3$	268.27	120	74.55%	C=N C=O CH-stretching	1626 1665 3004
2	Chalcone[C1]	$C_{22}H_{15}N_3O_5$	401.37	190	72.22%	C=N C=O C=C CH-stretching	1625 1662 1597 3072

A. IR Spectra

In the IR spectra of the Schiff base shows band at 1626 cm-1 assigned to the azomethine group(HC=N), this confirms the condensation between the amino group of 3-amino acetophenone and the aldehyde group of 4-Nitrobenzaldehyde in formation of Schiff base. The intensity peak of absorption in the Schiff base around 1665cm-1 due carbonyl group of ketone and CH-stretching observed at 3004 cm-1. In the Chalcone shows band around 1625 cm-1 assigned to the azomethine group, a peak at 1662cm-1 due carbonyl group of ketone. A band observed at 1597 cm^{-1} assigned to carbon-carbon double bond which confirms Chalocone formation and CH-stretching observed at 3072 cm-1.

B. 1H-NMR

The structure of Schiff base and Chalcone was confirmed by1H NMR spectra. In the 1H NMR spectra of Schiff base showed singlet signal at 8.54 ppm may be assigned to the azomethine group. Signals observed at 7.19-7.56 and 8-8.12 ppm are due to protons of aromatic ring and a singlet signal observed at 2.10 ppm assigned to methyl group. In the 1H NMR spectra of Chalcone showed singlet signal at 8.64 ppm may be assigned to the azomethine group. Signals observed at 7.84-7.91, 7.95-7.99, 8.10-8.14 and 8.27-8.39 ppm are due to protons of aromatic ring and a signal observed at 7.57-7.65 ppm assigned to ethene(HC=CH) group which confirmed Chalcone formation.

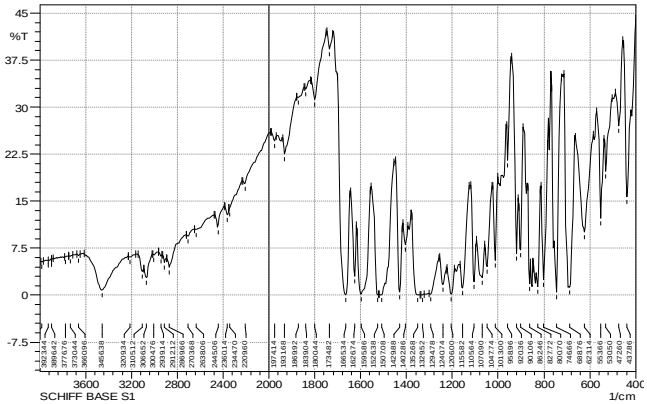


Fig. 1 IR spectra of Schiff base [S1]

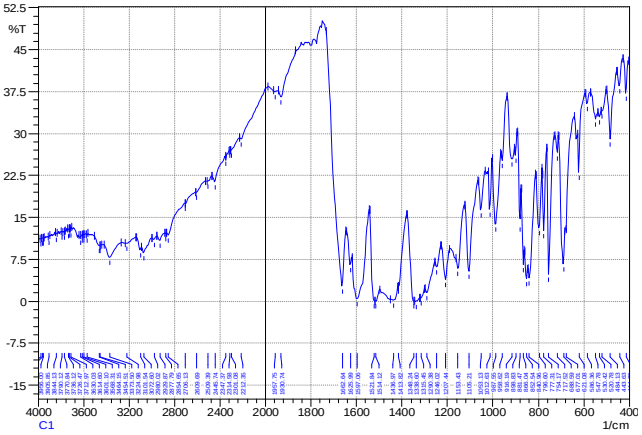


Fig. 2 IR spectra of Chalcone[C1].

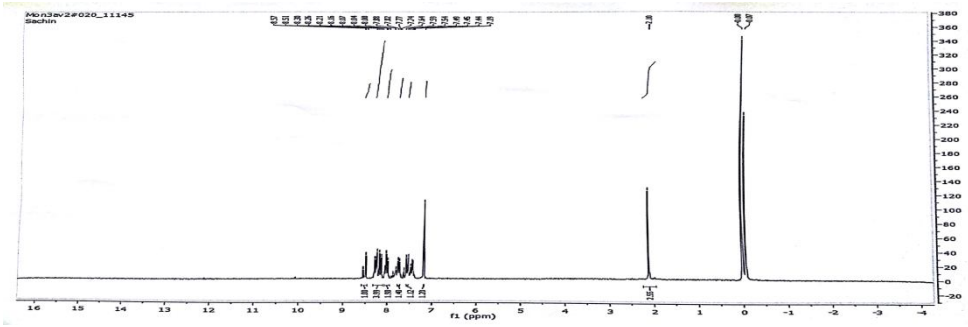


Fig.3 1H NMR spectra of Schiff base[S1]

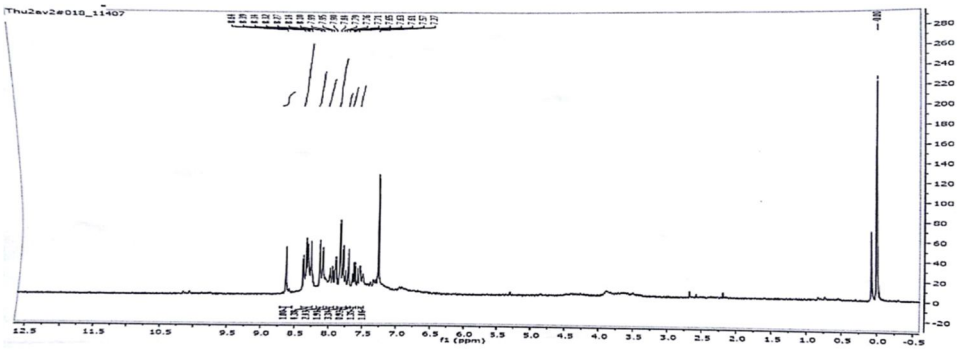


Fig.4 1H NMR spectra of Chalcone [C1]

IV. CONCLUSION

Synthesis of Chalcone from Schiff Base derived from 3-Amino Acetophenone were synthesized and characterized by IR and NMR. In the synthesis at first step new HC=N bond formed between amine group of 3-Amino Acetophenone and 4-Nitrobenzaldehyde which confirmed Schiff base formation. In second step new HC=CH bond formed betweenmethyl group of Schiff base and aldehyde group of 4-Nitrobenzaldehyde which confirmed Chalcone formation.

V. ACKNOWLEDGMENTS

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