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RESEARCH ARTICLE

SYNTHESIS CHARACTERIZATION AND BIOLOGICAL EVALUATION OF SCHIFF BASES CONTAINING COUMARIN MOIETY

***Kharode, B.G. and More, M. S.**

[1] Department of Chemistry, R.A. Art's, Shri M.K. Commerce and S.R. Rathi Scienece Mahavidhyalaya Washim. (M.S.India)

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****Corresponding Author:***
Kharode, B.G.

ABSTRACT

The Present workdivided in five part in first the coumarin was synthesise from resorcinol and ethyl acetoacetate in acidic medium. In second step coumarineacylated by reacting with chloroacetyl chloride, which on further react with hydrazine and finally the Schiff base of some aromaticaldehyde with amine containing coumarine moiety were synthesized. The physical measurement and structural elucidation by spectrum like FT-IRand 1H-NMR, used in this work.

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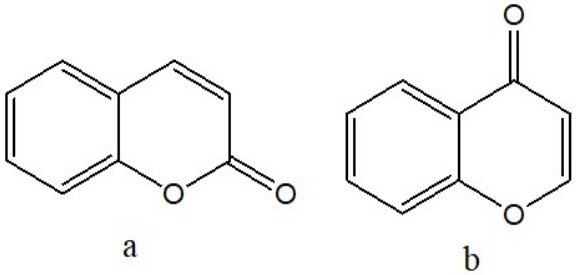
INTRODUCTION

The azomethine (-C=N-) containing compound typically known as Schiff bases have been synthesised by the condensation of primary amine with active carbonyls. Schiff base from a significant class of compounds in the medicinal and pharmaceutical chemistry with several biological application that include antibacterial and antitumor activity. They have been extensively studied as a class of ligand. Six remembered oxygen heterocyclic constitute a groupof compounds which occur widely in nature. These compounds contain basic unit pyran viz. 2-H-pyran (I) and 4-H-pyran (II). Pyrans with one more oxygen atom as carbonyl function are known as pyrones. Benzologues of α-pyrone fused at 5,6-positions are known as coumarins. Chemistry of coumarin. The fusions of pyrone ring with benzene nucleus gives rise to a class of heterocyclic compound known as benzopyrone and are recognized into two distinct types Benzo-α-pyrone 1, commonly called as coumarin and Benzo-γ-pyrone 2, commonly called as chromone (Figure 1). They are differing from each other only in the position of carbonyl group.



a b

EXPERIMENTAL

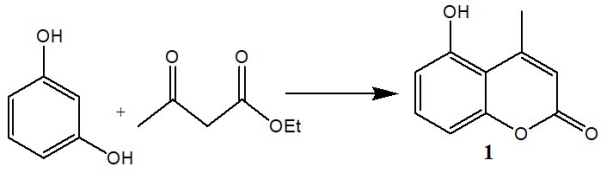
Solvents were employed as commercial anhydrous grade. The column chromatography was done over the silica gel (100-120 mesh). Melting points were determined in open capillary tube and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on Bruker advance II-400 MHz spectrometer. The molar conductivity measurements of complexes in (1 × 10−3 M) DMSO solution were measured at 25 ^{0}C with a Bibby conduct meter.

MATERIALS AND METHODS

All solvents were labouring as commercial anhydrous mark without further Refining. The column chromatography was carried out over silica gel (100120esh). Melting points determined by open capillary tube. ^{1}H NMR spectra were recorded on a Bruker400 MHz spectrometer in DCl3solvent TMS as internal standard. The crude product was recrystallizing from 80 percentage ethanol.

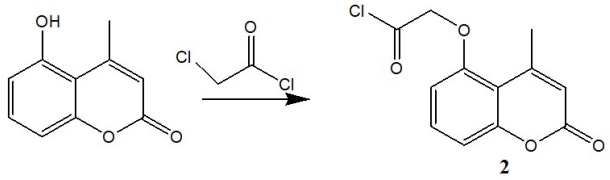
Present Work: In the present work, the aromatic substituted Schiff bases were synthesized by condensing substituted amine containing coumarine moiety.

Step I: General Procedure for the synthesis of Schiff base: The resorcinol (0.01 mole) and ethyl acetoacetate (0.01 mole) in round bottom flask containing 15ml ethanol and 3 ml of conc. Sulphuric acid was reflux for 1.5 hour a solid were obtain which is further cool and recrystallize from ethanol.



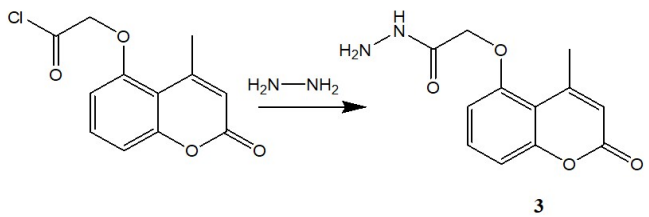
Scheme I

Step II: General Procedure for the synthesis of Schiff base: The mixture of Coumarine (0.01 mole) and Chloroacethyl chloride (0.01 mole) in round bottom flask containing 10 ml potassium carbonate was stirred at room temperature for 1.5 hour. After completion of reaction (by TLC) the mixture was poured on ice cold water and dried at room temperature.



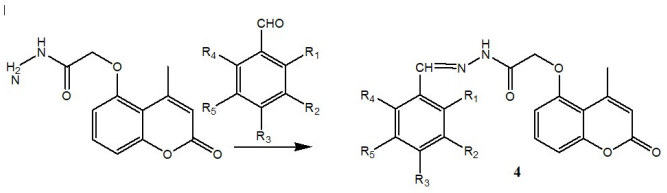
Scheme II

Step III: General Procedure for the synthesis of Schiff base: The compound 2 was heated with hydrazine hydrate in ethanol on a water bath for 1 hour to obtain 2-[4-Methyl-2-oxo-2H-Croman-7-yl)oxy]acetohydrazide compound 3.



Scheme III

Step IV: General Procedure for the synthesis of Schiff base: A mixture of alcohol (20 ml) and aromatic aldehyde (0.02 mol) was taken into a 100 mlround bottom flask. The mixture was stirred until a homogeneous solution was obtained;comarine Contain primary amine group(0.02 mol) was added with stirring. (As the reaction is exothermic it should becarried out by placing flask in a freezing mixture). Reaction mass is stirred for another 45 min. the Schiff base was precipitated out. The reaction mixture was cool with stirring. The isolatedcrude product is purified by the washing in acetone.



Scheme IV

Compound also purify by silica gel column chromatography eluent ethyl acetate hexane reaction was. Monitored by TLC & spot were visualized in iodine.

1)**G_7**

FT-IR760 cm^{-1}for aromatic C-C stretching,1110 cm^{-1}for C-O stretching , 1580 cm^{-1}forC=Nstretching ,1680 cm^{-1}for C=O stretching 1400 for C=C stretching, 1090 cm-1 for C-N stretching, 3210 for N-H stretching.

NMR

^{1}H NMR (400 MHz, $CDCl_3$):δ 1.7 (s, 3H),δ4.5 (s, 2H,),δ 6.33-7.98 (m, 9H),δ 8.33 (s, 1H NH), δ 8.0(s, 1H).

2)**G_{56}**

FT-IR,670 cm^{-1} 1for C-I stretching, 750 cm^{-1}for aromatic C-C stretching, 1150 cm^{-1}for C-O stretching ,1070 cm-1 for C-N stretching, 1560cm^{-1} for Ar C=C stretching, 1620 cm^{-1} for C=O stretching,2960cm^{-1}Ar C-H stretching 3190 for N-H stretching.

^{1}H NMR (400 MHz, $CDCl_3$):δ 2.1 (s, 3H),δ 5.5 (s, 2H,),δ 7.00-7.98 (m, 9H), δ 8.53 (s, 1H NH), δ 8.12(s, 1H).

3) **G_{63}**

FT-IR: 550 cm^{-1} for C-Cl stretching, 790 cm^{-1}for aromatic C-C stretching,1060 cm-1 for C-O stretching, 1190 cm^{-1}for C-N stretching , 1440cm^{-1} for Ar C=C stretching, 1670 cm^{-1} for C=O stretching,28100cm^{-1}Ar C-H stretching, 3120 for N-H stretching.

^{1}H NMR (400 MHz, $CDCl_3$):δ2.6 (S, 3H),δ 5.0 (S, 2H,),δ 6.20-7.98 (m, 9H), δ 8.55 (s, 1H NH), δ 8.20(s, 1H).

Antibacterial properties of the synthesized Schiff base metal complex [Zone of inhibition (mm)]: The in vitro antimicrobial activity of the investigated compounds was tested against the bacteria such as E. coli, S. aureus, by the serial dilution method. The minimum inhibitory concentration (MIC) values of the compounds against the growth of microorganisms are summarized in Table 2.

RESULT AND DISCUSSION

All the six Schiff base Congaing coumarine moiety i.e. compounds G_7,G_{56},G_{63} &G_{65} were successfully synthesized in excellent yield and their structures are elucidated using elemental analysis, FTIR, &^{1}HNMR spectroscopy. All the Synthesised Compound will screed for their biological activity.

Table 1. Synthesis of G_7, G_{56}, G_{63}, &$G_{65\ in}$ terms of Yield and melting point

S.N.	Compound	R_1	R_2	R_3	R_4	R5	M.P.(^{0}C)	% Yield
1	G7	H	H	H	H	H	276	80.58
2	G56	H	H	H	H	I	280	82.05
3	G63	H	H	Cl	H	H	296	81.20
4	G65	H	H	H	H	OCH_3	258	70.25

Table 2. Antibacterial properties of the synthesized Schiff base metal complex

Compound	E. coli	S. aureus
G7	4.2	3.2
G56	2.5	4.1
G63	3.2	2.8
G65	2.9	3.5

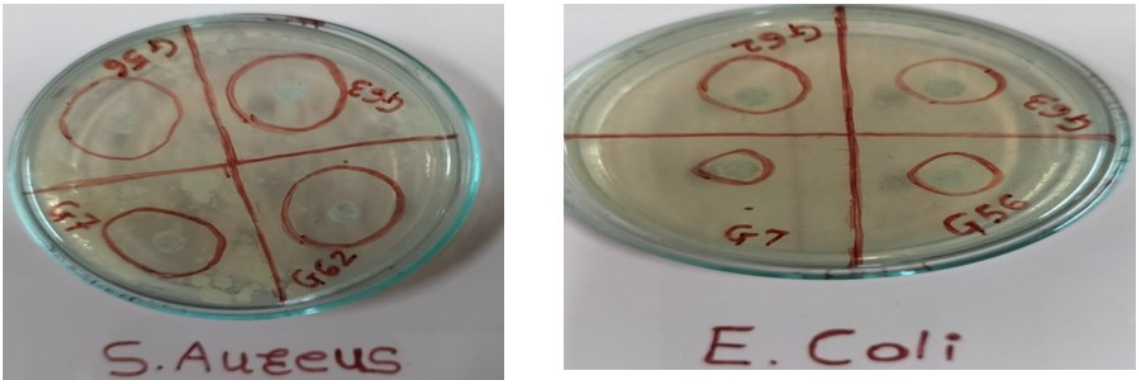


Fig : Zone of inhibition of comp. G_7, G_{56}, G_{63}, and G_{65} against S. Aureus and E. Coli.

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