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Synthesis of coumarin-Chalcone Derivatives

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Abstract: *Based on the observed biological activities of Coumarin and chalcones, we have synthesized Coumarin -chalcone hybrids with the aim of evaluating their anti-oxidant properties and Trypanocidal activity against Trypanosoma cruzi, parasite responsible for Chagas disease. All derivatives have been proved to be good anti-oxidants. These preliminary findings encourage us to future structural optimization of these kind of compounds. Various Coumarins were synthesized and converted to Coumarin-Chalcone hybrids. Coumarins were synthesized by treating Salicylaldehyde and Ethyl acetoacetate in presence of piperidine as base and ethanol as solvent. Then Coumarins were converted to Chalcones by reacting with 3-acetyl Coumarin with aldehydes to give Coumarin Chalcone hydrides.*

Keywords: *3-acetylcoumarin, 3-Cinnamoylcoumarins, 3-(4'-Methoxycinnamoyl) coumarin.*

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I. Introduction

Chemotheraphy is the treatment of disease by chemicals especially by killing micro-organisms or cancerous cells. Nowadays are known a wide range of different chemotherapeutic agents. Drug discovery plays an important role in today life. Lot of drugs have been synthesised for various biological activities such as anti-cancer, anti-diabetic, anti-microbial, anti-oxidant, anti-malarial, anti-fungal. Coumarin has been reported to serve as antibacterial, anti-oxidant, anti-inflammatory anticoagulant and anti-tumour agents. These pharmacological properties of Coumarin aroused our interest in synthesizing some Coumarin-Chalcone derivatives. Chalcone or benzylidene acetophenone are the important constituents of natural sources. Coumarin constitutes one of the major classes of naturally occurring compounds, and interest in its chemistry continues because of its usefulness as biologically active agents. It also represents the core structure of several molecules of pharmaceutical importance. Interest in synthesizing some coumarin derivatives with the aim of testing their microbiological activity.

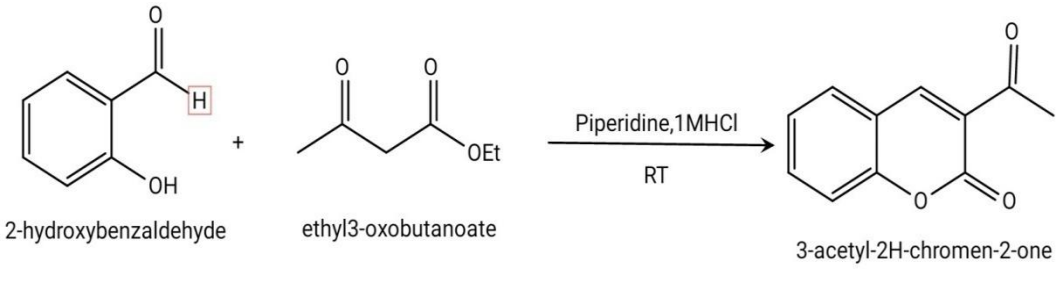
II. Synthesis

All reactions were carried out under dry and deoxygenated argon Atmosphere. Solvents were used as anhydrous by reflux of each solvent over an appropriate dryer agent and further distillate under argon atmosphere.

2.1) Synthesis of 3-acetylcoumarin:

A mixture of Salicylaldehyde (1eq.) ethyl-acetoacetate (1eq.). And a few drops of piperidine were mixed for 5 minutes at room temperature without any solvent. Reaction was neutralized with HCl (1M) and finally the product was isolated by filtration. The final compound was then recrystallized in EtOH.

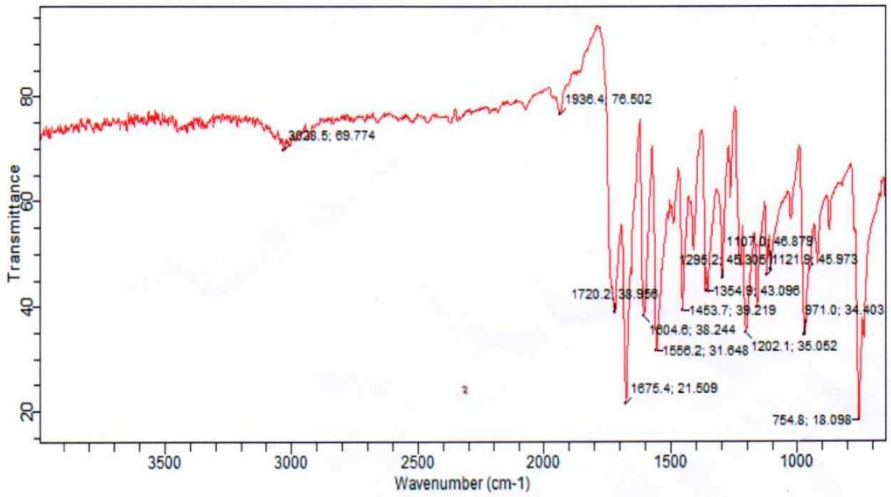
Reaction:



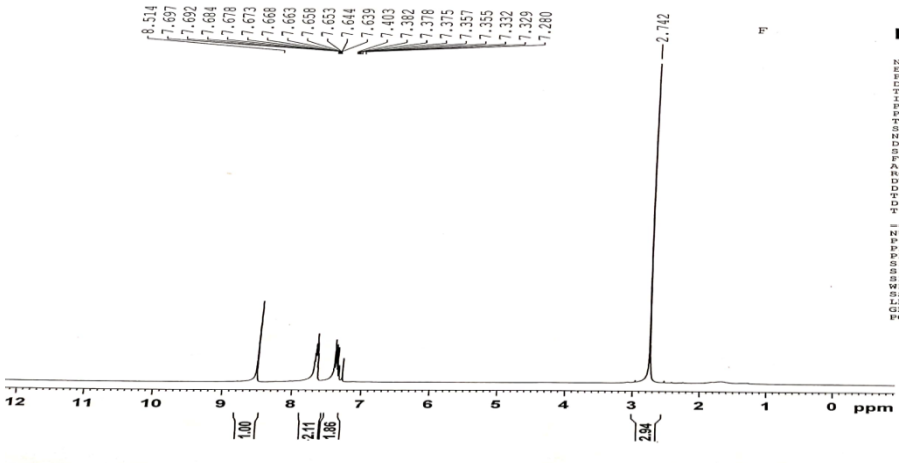
2-hydroxybenzaldehyde ethyl3-oxobutanoate 3-acetyl-2H-chromen-2-one

Tabular column:

Compound	Molecular Weight	Quantity	Moles	Equivalent Weights
Salicilaldehyde	122gram/mole	2gm	0.016	1eq
Ethylaceto acetate	130.1gram/mole	2.1gm	0.016	1eq
Piperidine	2-3 drops	-	-	-
Ethanol	10ml	-	-	-



- C=O stretching at 1720 cm^{-1}
- C-H stretching at 3028 cm^{-1}
- C=C stretching at 1453 cm^{-1}, 1556 cm^{-1}
- C-O-C stretching at 1202 cm^{-1}

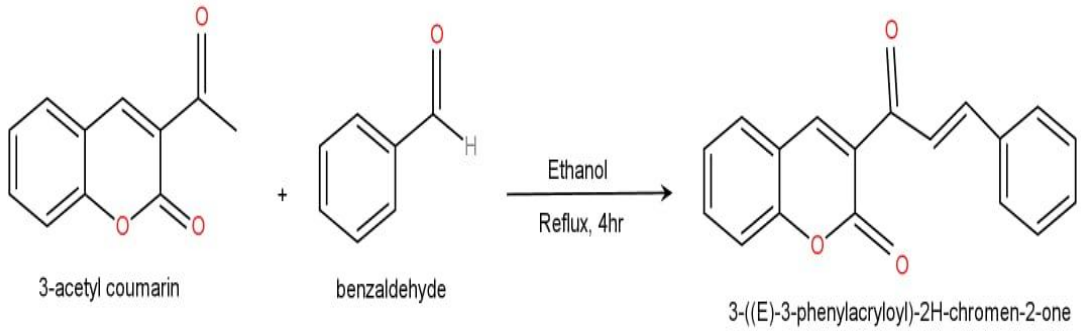


- δ 2.742 (s, 3H) of (O=C-CH_3)
- δ7.35 (double triplet 2H) at (6,7 position)
- δ7.77 (s, 2H) at (5,8 position)
- δ8.64(s, 1H) at (4 position)

2.2) Synthesis of 3-Cinnamoylcoumarins:

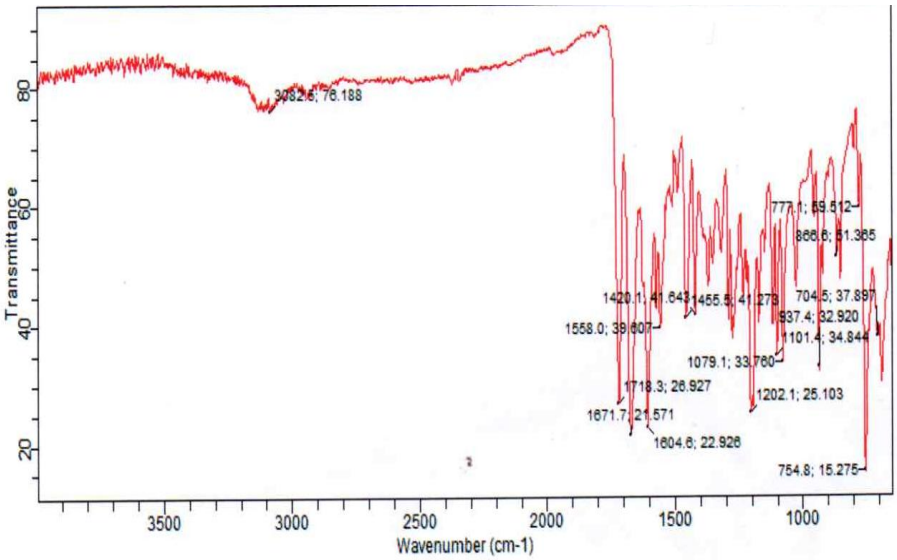
A mixture of 3-acetylcoumarin (1eq.) and corresponding benzaldehyde (1.2eq.) in EtOH was stirred with a few drops of piperidine under reflux during 2-12 hours. Mixture was cooled and the resulting solid was filtered and purified by recrystallization. Purification of compounds was made by recrystallization in MeOH.

- **Reaction:**



- **Tabular Column:**

Compound	Molecular weight	Quantity	Moles	Equivalent weight
3-acetyl Coumarin	188.182 gram/mole	0.632gm	0.00335	1eq
Benzaldehyde	106.13 gram/mole	0.4277gm	0.00403	1.2eq
Piperidine	2-3 drops	-	-	-
Ethanol	10ml	-	-	-

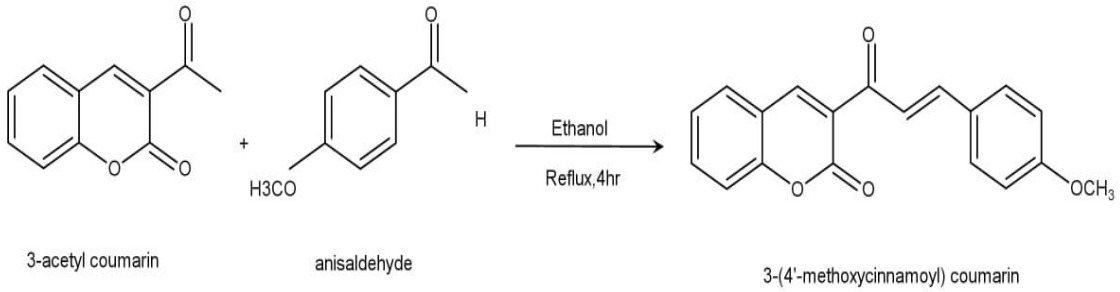


- C=O stretching at 1750 cm^{-1},
- C=O stretching at 1680 cm^{-1},
- C=C stretching at 1550 cm^{-1}.

2.3) General procedure for the synthesis of 3-(4'-Methoxycinnamoyl) coumarin:

A mixture of 3-acetylcoumarin (1eq.) and corresponding Anisaldehyde(1.2 eq.) in EtOH was stirred with a few drops of piperidine under reflux for 2-12 hours. Mixture was cooled and the resulting solid was filtered and purified by recrystallization. Purification of compounds was made by recrystallization in MeOH.

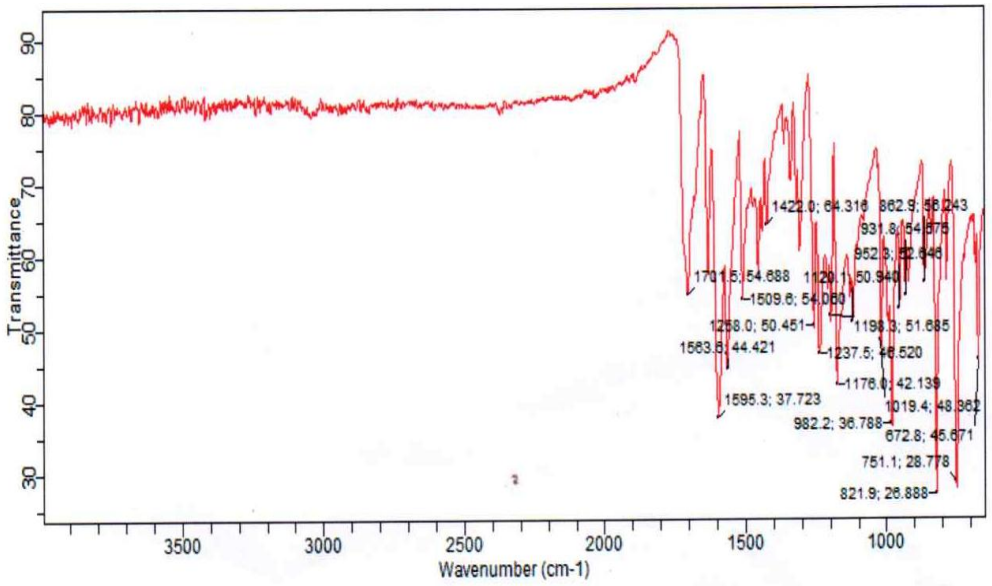
Reaction:



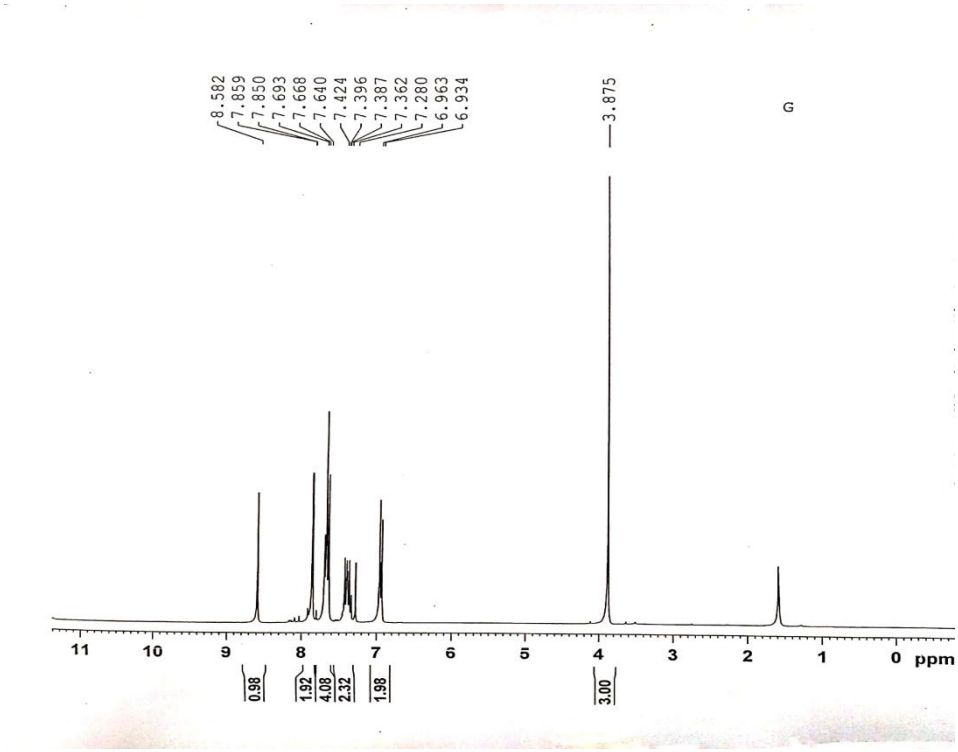
3-acetyl coumarin anisaldehyde 3-(4'-methoxycinnamoyl) coumarin

Tabular Column:

Compound	Molecular weight	Quantity	Moles	Equivalent weight
3-acetyl coumarin	188.182 gram/mole	2.803gm	0.0148	1eq
Anisaldehyde	136.15 gram/mole	2.43163gm	0.01786	1.2eq
Piperidine	3-4 drops	-	-	-
Ethanol	10ml	-	-	-



- C=O stretching at 1720 cm^{-1},
- C=O stretching at 1625 cm^{-1},
- C=C stretching at 1570 cm^{-1}.
- C-H stretching at 3030 cm^{-1}
- C-O-C stretching at1237 cm^{-1}



- δ 3.825 (s, 3H) of ($–OCH_3$)
- δ 6.95(d. 2H) of (CH=CH)
- δ 7.55(double doublet 4H) of(Aromatic)
- δ 7.35(double triplet 2H) at (6,7 position)
- δ 7.77(s. 2H) at (5,8 position)
- δ 8.64(s.1H) at (4 position)

III. Result &Discussion

Sl. No.	Name of the Product	Practical yield	M.P
1.	3-acetyl coumarin	61.68%	112°-114°C
2.	3-cinnamoyl coumarin	58.195%	198°-200°C
3.	3-(4'-methoxycinnamoyl) coumarin	48.45%	196°-199°C

3-acetyl coumarin is synthesized from Salicylaldehyde and Ethyl-acetoacetate by using piperidine as a base. Further 3-acetyl Coumarin was converted to Coumarin Chalcone Hybrid (3-Cinnamoyl Coumarin). It was prepared by refluxing 3-acetyl Coumarin and Benzaldehyde in presence of piperidine using Ethanol as a solvent. Similarly 3, (4'-methoxy Cinnamoyl Coumarin). Was also synthesized by refluxing 3-acetyl Coumarin a Anisaldehyde in presence of piperidin as a base. .

IV. Conclusion:

In this study, we synthesized various substituted Coumarins and they were converted to Coumarin-Chalcone hybrids. All the compounds that we synthesized were characterized by H^1NMR, IR and their melting points. We have confirmed the considerable antioxidant activity of new hydroxylated coumarin-chalcone hybrid compounds. Their antioxidant activity is affected by the introduction of a benzoyl moiety at the C3 position regarding to the coumarin ring. A very interesting finding is that compounds are very reactive and presents good antioxidant capacity against hydroxyl radicals as well as low oxidation potential. In spite of the moderate trypanocidal activity of coumarin-chalcone hybrids, they have been proved to be very good antioxidants. Based on these results, we can conclude that compounds are potential candidates for the studies of their antioxidant activity.

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