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THE SYNTHESIS OF HETEROCYCLIC COMPOUNDS BASED ON 3-FORMYLCHROMONE VIA ORGANIC REACTIONS

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Abstract – 3-Formylchromones are known as key heterocyclic scaffolds for the synthesis of various important organic compounds. The compounds with 3-formylchromone have attracted much attention among heterocyclic structures. This review article aims to provide an overview of multi-component reactions related to 3-formylchromone derivatives in the synthesis of various heterocyclic compounds covering the literature going back to 2015 until 2019.

CONTENTS

1. INTRODUCTION

3-Formylchromones are the most usable derivatives of chromones which used in the synthesis of heterocyclic compounds. They are also known as 4-oxo-4*H*-1-benzopyran-3-carboxaldehydes, 4-oxo-4*H*-chromene-3-carboxaldehydes, and chromone-3-carboxaldehydes. Among many methods which were used for the synthesis of 3-formylchromones, Vilsmeier-Haack[1] reaction on substituted 2-hydroxyacetophenones is the most applicable.[2-9] The compounds include 3-formylchromone scaffold

show the variety of pharmacological activities such as antitumor,[10] antimicrobial,[11,12] anti-tubercular,[2] anti-inflammatory[6], and antifungal.[14] Due to the specific structure of 3-formylchromone from a synthetic view point; it is a versatile moiety in the synthesis of novel heterocyclic compounds.[5] The existence of three electron-deficient sites, the carbon atom of aldehyde, the C–2 and the C–4 of the chromone leads to use the 3-formylchromones as heterodienes as well as a dienophile or a Michael[15] acceptor. In recent years, several review articles on the chemistry and the application of 3-formylchromones have been published.[16-18] In continuation of our studies toward investigation of synthetic heterocyclic compounds,[19-30] herein, the recent applications of 3-formylchromones in the synthesis of different types of heterocyclic compounds are reviewed during the period from 2015 to 2019.

According to Scopus database, there are many subject areas related to 3-formylchromones, and among them chemistry has the first rank about 41% as demonstrated in Figure. 1 which shows the high importance of this compound.

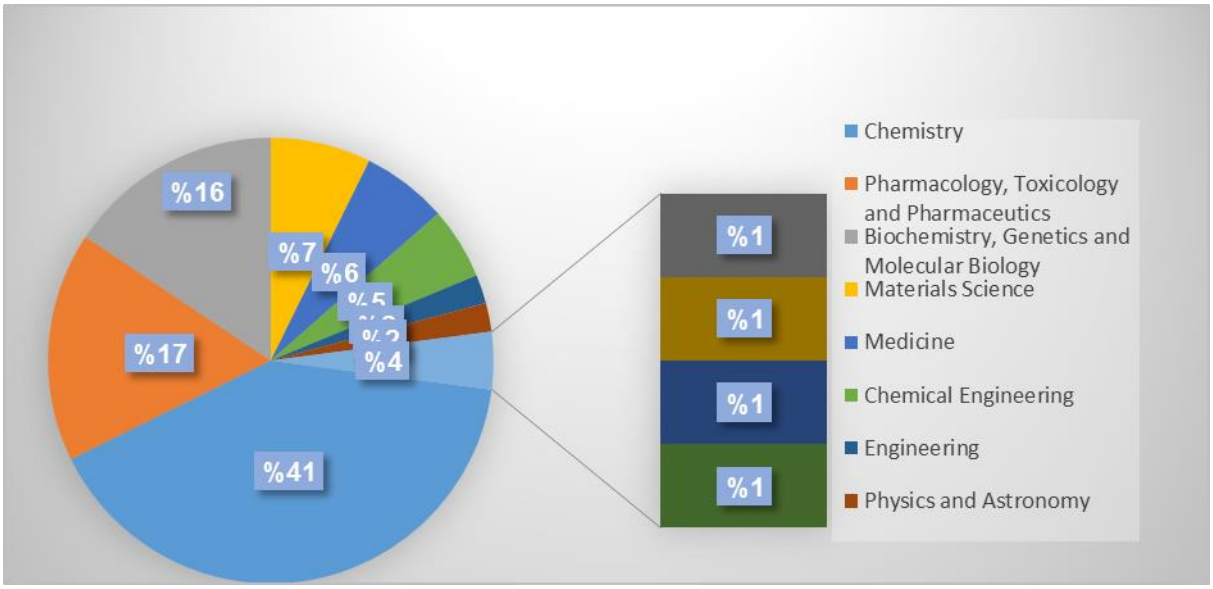


Figure 1. The percentage of publication of 3-formylchromone by the subject area

Base on Scopus resource type, the journal of BMC Gastroenterology gain the first step of the resource, which could be highlighted the importance of the 3-formylchromone about 55% in the medicinal chemistry due to providing biologically active products and in the second level the Journal of Heterocyclic Chemistry take the next level for publishing its papers (Figure 2).

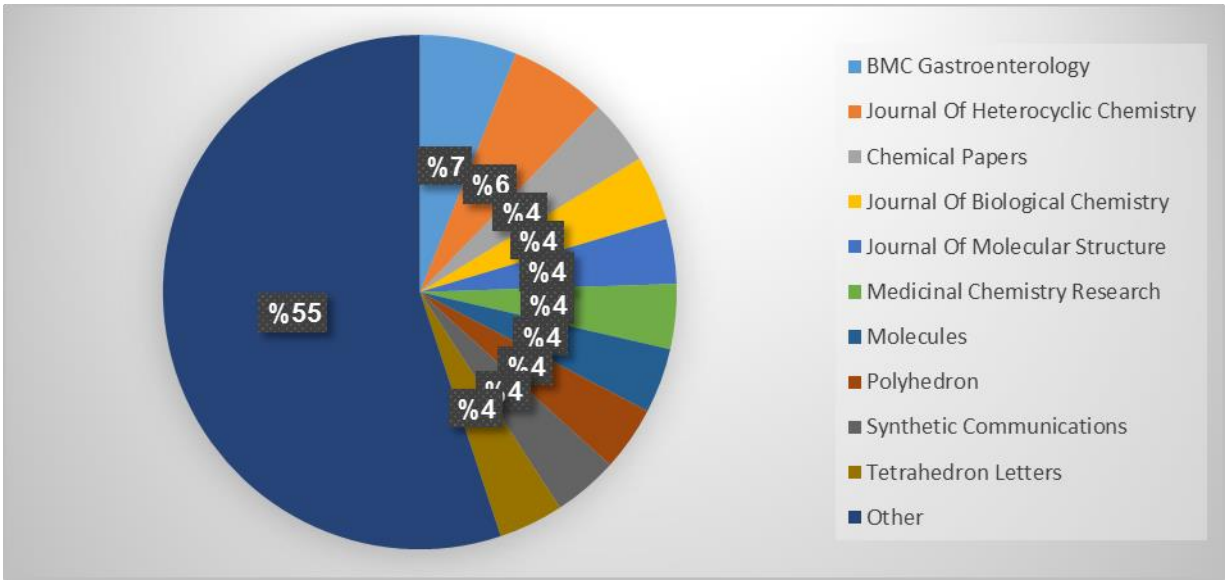


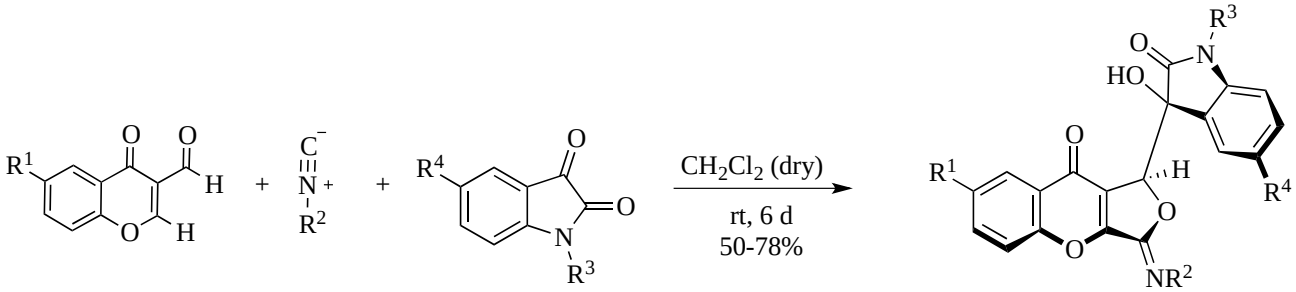
Figure 2. The publication percentage of 3-formylchromone articles based on the source type

2. APPLICATION OF 3-FORMYLCHROMONES IN THE SYNTHESIS OF THE HETEROCYCLIC COMPOUNDS *VIA* VARIOUS METHODS

2.1 Cycloaddition and annulation reactions based on 3-formylchromones

2.1.1. [4+1] Cycloaddition reactions

In 2017, Teimouri and co-workers[31] described the synthesis of furochromone-isatin conjugates *via* an uncatalyzed diastereoselective [4+1] cycloaddition/tautomerization/Friedel-Crafts hydroxyalkylation domino reaction of isocyanides **2**, 3-formylchromones **1** and isatin derivatives **3** in dry CH_2Cl_2 (Scheme 1).



1 **2** **3** **4**

R^1= H, Cl, Me

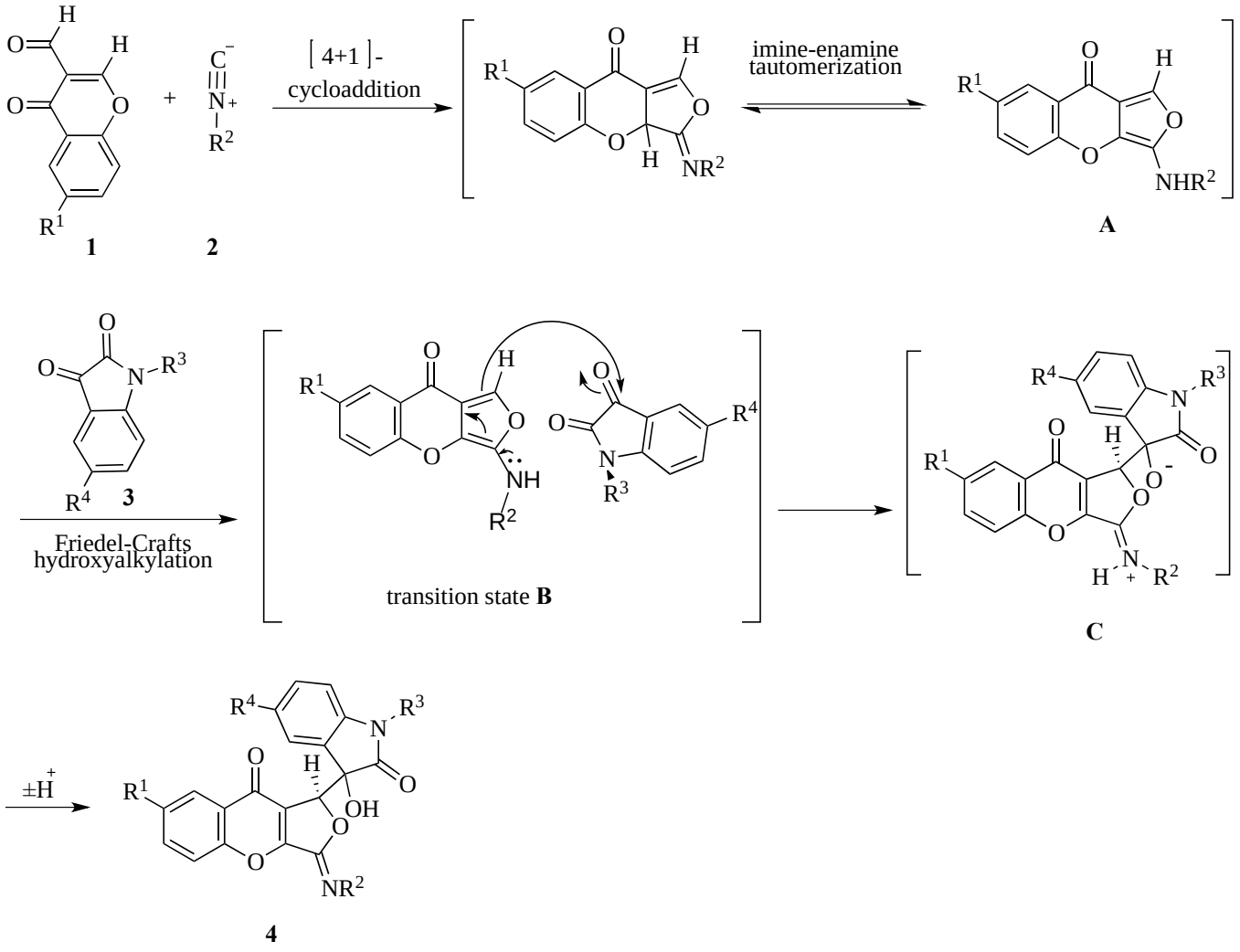
R^2= C_4H_9, C_6H_5, $C_6H_5CH_2$, C_8H_9,

R^3= H, Me, Et, $C_6H_5CH_2$

R^4= H, Br, NO_2

Scheme 1. Diastereoselective synthesis of furochromone-isatin conjugates

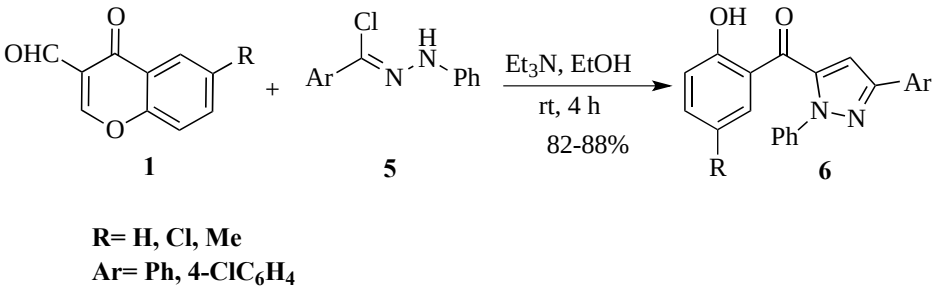
The proposed mechanism is initiated through the [4+1] cycloaddition of 3-formylchromones **1** with alkyl or aryl isocyanides **2** to generate 3-(alkylamino)-9*H*-furo[3,4-*b*]chromen-9-ones as intermediate **A**, which has imine-enamine tautomerization. Then, the intermediate **A** reacted with isatin *via* its activated carbonyl group through the electrophilic Friedel-Crafts hydroxyalkylation[32] to provide the transition state **B** which was converted to the intermediate **C** *via* electrophilic heteroaromatic substitution on the fused 2-aminofuran moiety. The last one affords furochromone-isatin conjugates **4** by hydrogen interchange (Scheme 2).



Scheme 2. Possible mechanism and intermediate **C** conformation for the formation of furochromone-isatin hybrid heterocycles **4**

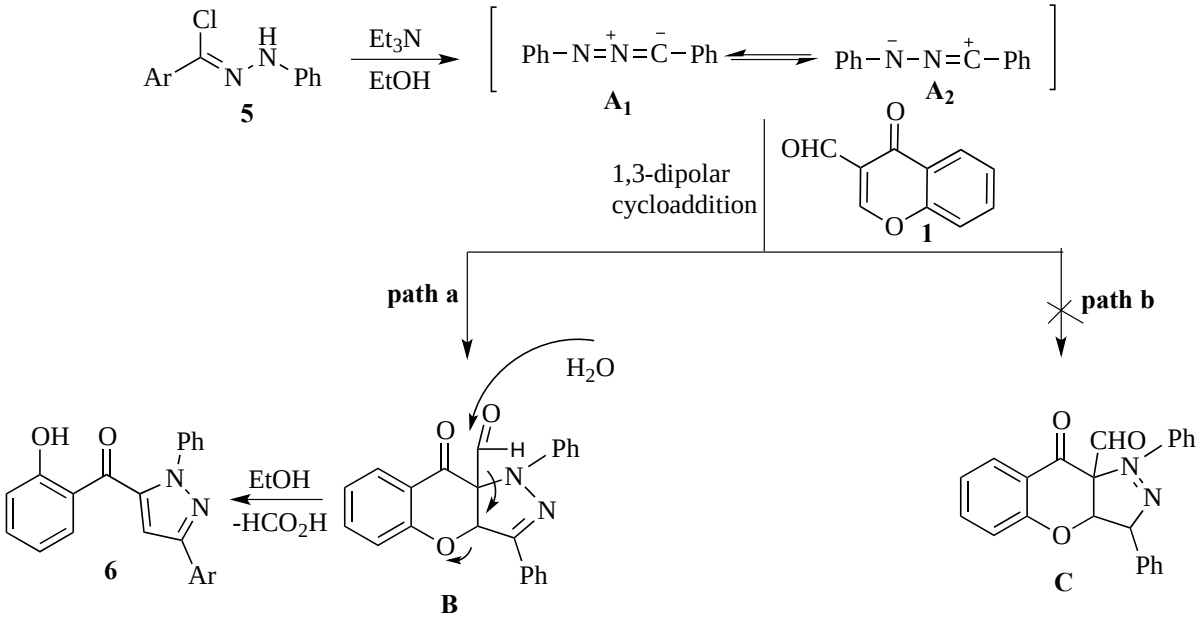
2.1.2. [3+2] Cycloaddition reactions

In 2016, Alizadeh and co-workers[33] synthesized the pyrazole derivatives **6** *via* a simple and regioselective 1,3-dipolar cycloaddition reaction (Scheme 3). The synthesis was included the reaction of **1** with hydrazonoyl chlorides **5** in the presence of Et_3N in ethanol at room temperature.



Scheme 3. The regioselective synthesis of pyrazole derivatives **6**

A mechanistic proposal was presented in Scheme **4**. At first, hydrazonoyl chloride **5** treated with Et_3N to afford *in situ* the nitrile imine **A**, which reacted with 3-formylchromone **1** *via* the 1,3-dipolar cycloaddition reaction through two pathways. In path **a**, the connection of electron-rich carbon of dipole structure nitrile imine $\mathbf{A_1}$ reacted with C–2 of 3-formylchromone to obtain the regioisomer **B**, which can treat with water to produce the target product **6**. In path b, the connection of electron-rich nitrogen of dipole structure nitrile imine $\mathbf{A_2}$ attacked to C–2 of 3-formylchromone to give the regioisomer **C**, which was not obtained by X-ray crystallographic analysis.[33]

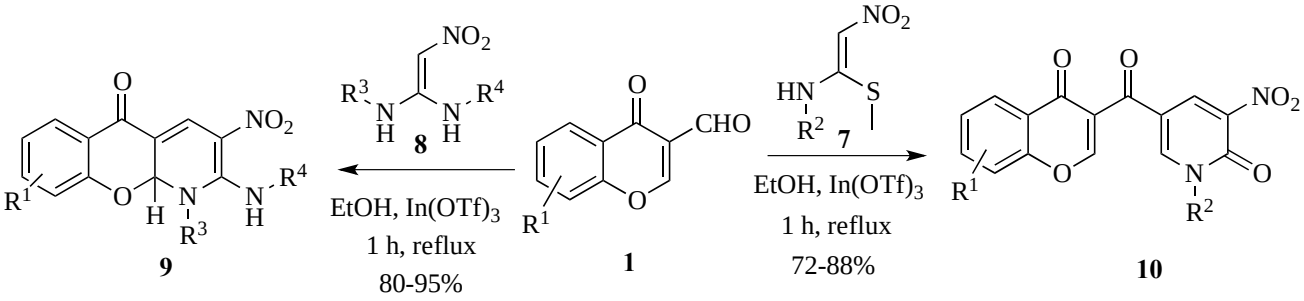


Scheme 4. Proposed mechanism for the synthesis of pyrazole derivatives **6**

2-1-3. [4+2] Cycloaddition reactions

Poomathi and co-workers[34] reported the green and one-pot method for the synthesis of 2-pyridone derivatives **10** by the reaction of 3-formylchromones **1** and nitroethenamines **7** using indium triflate as

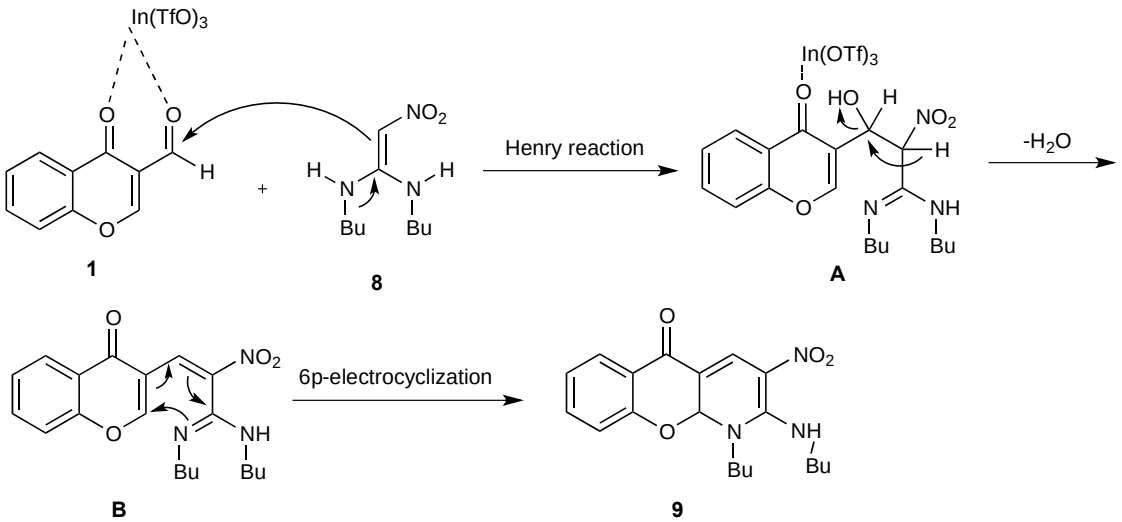
catalyst (Scheme 5). In the same condition, the compound **9** was produced from 3-formylchromones **1** and different cases of alkenes **8** as shown in Scheme 5.



R^1= H, Br, 6-Cl, 6,8-di-Cl, 7-F, F, Me, OMe
R^2= Me, *n*-propyl, *n*-butyl, cyclohexyl, benzyl
R^3= Me, *n*-butyl
R^4= Me, *n*-butyl, cyclohexyl

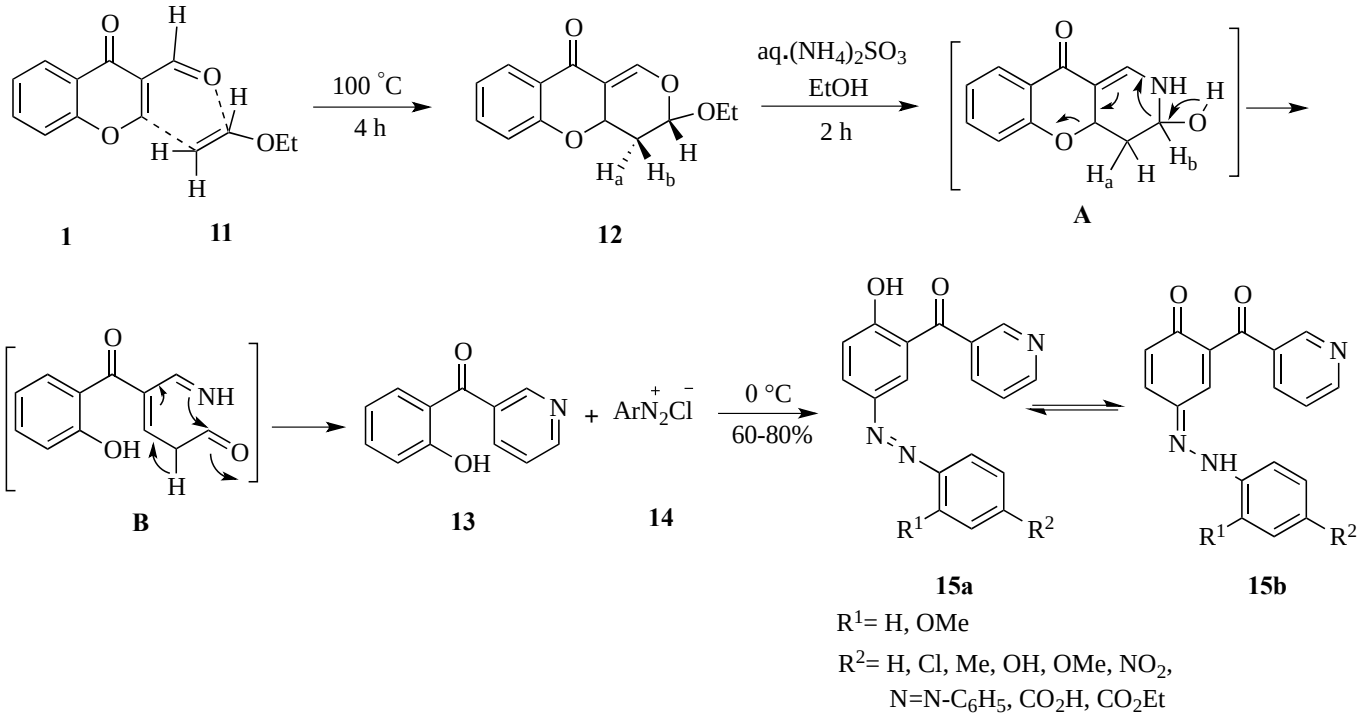
Scheme 5. The synthesis of 2-pyridones **10** and functionalized azaxanthones **9** *via* indium triflate

The rational mechanism was suggested for the reaction of 3-formylchromone **1** with *N*,*N*′-dimethyl-2-nitroethene-1,1-diamine **8** as shown in Schemes 6. In the first step, treatment of 3-formychromone **1** and *N*,*N*-dibutyl-2-nitroethene-1,1-diamine **8** provided the intermediate **A** through Henry reaction[35] then the later converted to intermediate **B** after elimination of water. In the following, 6π-electrocyclization of intermediate **B** formed the final product **9**.



Scheme 6. The mechanism of the synthesis of functionalized azaxanthone **9** frameworks *via* indium triflate catalyzed domino reaction

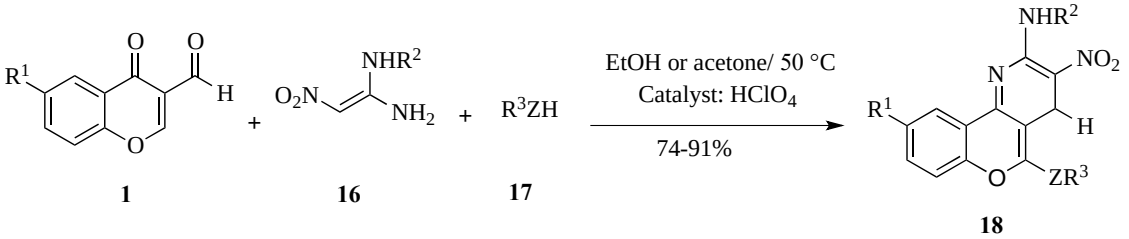
In 2016, Fadda and Abbas[36] synthesized a dyestuff precursor in three steps. This synthetic route was started from the reaction of 3-formylchromone **1** with ethyl vinyl ether **11** to obtain 3-ethoxy-4,4a-dihydro-3*H*,10*H*-pyrano[4,3-*b*]chromen-10-one **12**. In the next step, the treatment of **12** with aqueous ammonium sulfite in ethanol resulted in the intermediate **A** which was converted to the intermediate **B**. the latter was converted to 3-(2-hydroxybenzoyl)pyridine **13** by cyclization process**.** Finally, 4-arylazo-2-(nicotinoyl)phenols **15a** and **15b** were produced through coupling reaction of **13** with various aryl diazonium **14** salts (Scheme 7).



Scheme 7. Synthesis of 4-arylazo-(2-nicotinoyl)phenols **15**

2-2. Nucleophilic addition reactions

The biologically active chromenopyridines derivatives **18** were synthesized by 3-formylchromones **1**, (*Z*)-*N*-butyl-2-nitroethene-1,1-diamine **16** and ethanol or various amine derivatives **17** in the presence of $HClO_4$ as catalyst in ethanol at 50 ˚C (Scheme 8). It is important to know that three sites of 3-formylchromones **1** contributed to one-pot cascade reaction for the synthesis of bicyclic pyridines **18**.[37]

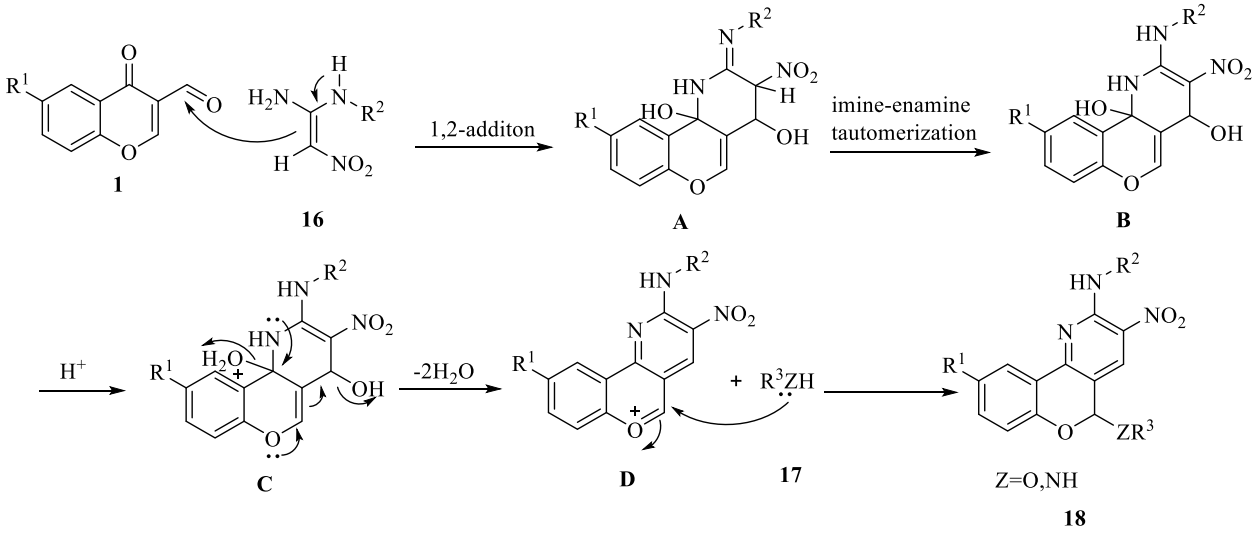


1 16 17 18

R^1= H, F, Cl, NO_2, Me
R^2= *n*- butyl, H, cyclohexyl, furan-2-ylmethyl, benzyl, 4-F-benzyl, 4-Cl-benzyl, phenethyl, 4-methoxyphenethyl
R^3= Et
Z= O, NH

Scheme 8. The synthesis of 5*H*-chromeno[4,3-*b*]pyridines **18**

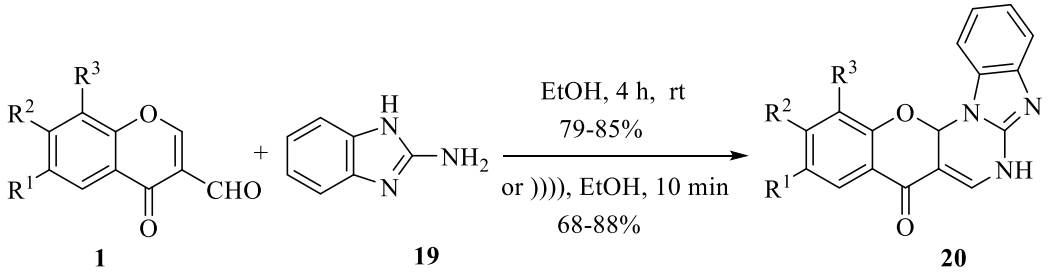
The proposed mechanism was shown for preparation of chromenopyridine **18** from 3-formylchromone **1**, 1,1-enediamine **16**, and ethanol/amine **17** in the presence of $HClO_4$ as catalyst. At first 1,1-enediamine **16** was reacted with the C4 of 3-formylchromone **1** by 1,2-addition reaction to form intermediate **A**, which was converted to intermediate **D** subsequently by the imine-enamine tautomerization, acid treatment and dehydration reaction. Finally, intermediate **D** was reacted with ethanol/amine **17** to produce 5*H*-chromeno[4,3-*b*]pyridines **18** (Scheme 9).[37]



1 16 A B C D 17 Z=O,NH 18

Scheme 9. Mechanism hypotheses for the synthesis of 5*H*-chromeno[4,3-*b*]pyridines **18**

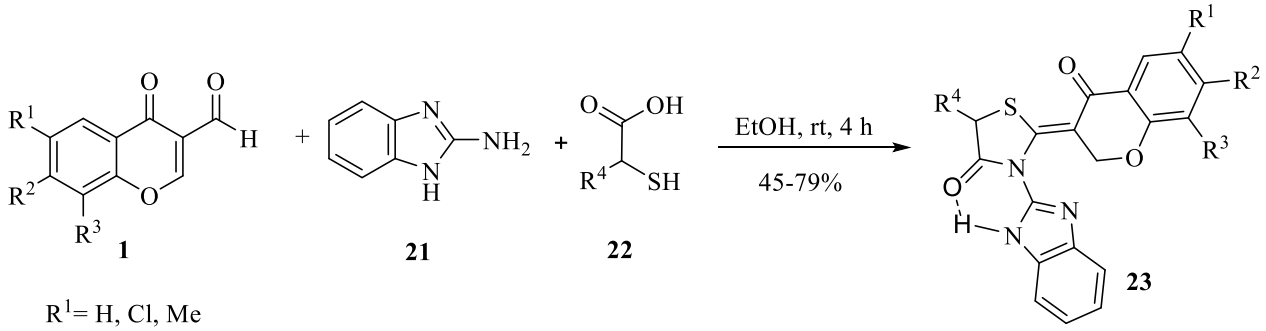
Chromenopyrimidobenzimidazolones **20** were prepared by the reaction of 3-formylchromone derivatives **1** and 2-aminobenzimidazole **19** for 4 h at room temperature in ethanol as solvent. Also, this reaction can be proceeded upon ultrasound condition at room temperature for **10** min in short reaction time as shown in Scheme 10.[38]



R^1= H, Br, Cl, *i*-Pr, Me
R^2= H, Me
R^3= H, Br

Scheme 10. The synthesis of chromenopyrimidobenzimidazolones **20**

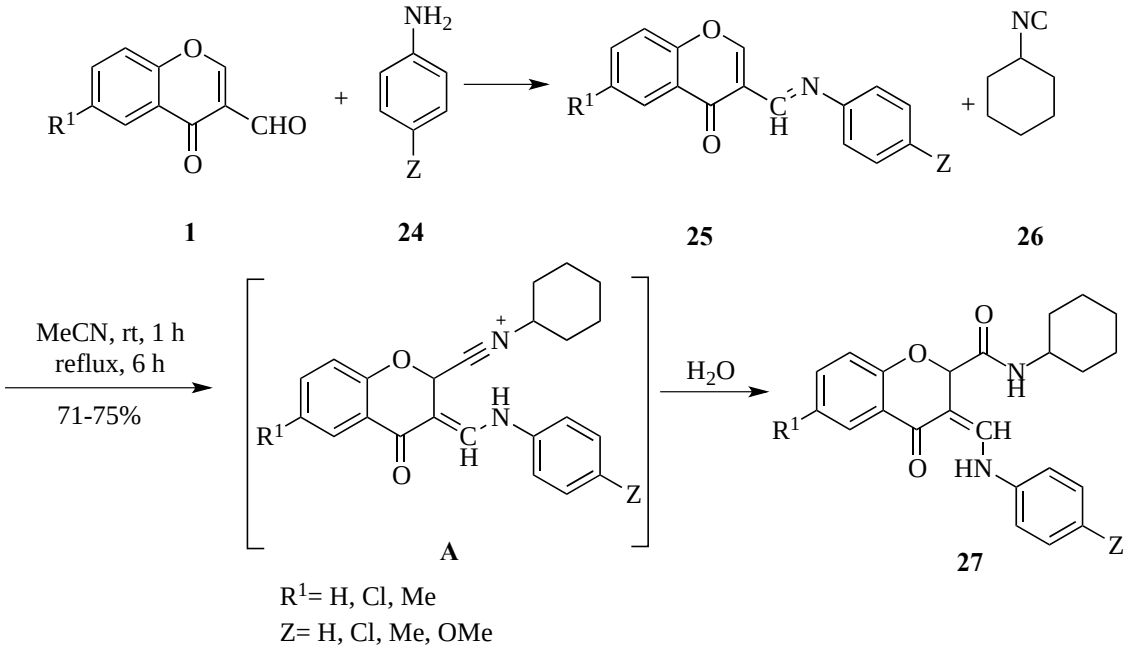
Polycyclic heterocyclic ring systems **23** were synthesized by the combination of different chromones **1**, benzimidazole **21** and thiol derivatives **22** through three-component reaction condition.[38] It is worth mentioning that the corresponding compounds categorized as pharmacophoric heterocycle scaffolds (Scheme 11).



R^1= H, Cl, Me
R^2= H, Me
R^3= H
R^4= H, Me

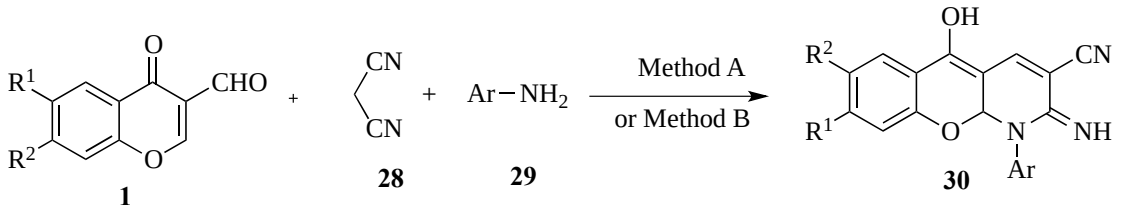
Scheme 11. The synthetic route of thiazolidinones **23**

In 2016, Ghosh and co-workers[39] described the three-component synthesis of 3-arylaminomethylidenechroman-2-carboxamide derivatives **27** as shown in Scheme 12. In this study, the reaction was proceed by the reaction of 3-formylchromones **1** and different aromatic amines **24** to provide Schiff-base **25** which was treated with cyclohexyl isocyanide **26** to obtain intermediate **A,** and then it was treated with water to produce the compound **27**.



Scheme 12. Synthesis of 3-aminomethylidenechroman-2-carboxamides **27**

In 2017, Gupta *et al.*[40] reported the synthesis of novel 5-hydroxychromeno[2,3-*b*]pyridines **30** from different 3-formylchromone **1**, various aromatic amines **29**, and malononitrile **28** *via* **A** and **B** method which was solvent and catalyst-free conditions at room temperature in both methods (Scheme 13).



Method A: Stiring at room temperature under neat condition, 10-15 min, 91-94%

Method B: Grinding under neat condition, 5-10 min, 90-94%

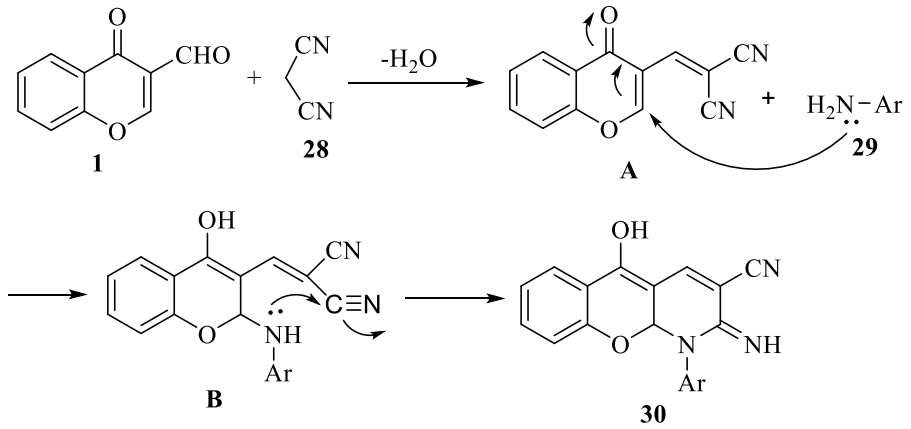
R^1= H, Br, Cl, Me

R^2= H, Me

Ar= 4-FC_6H_4, 4-ClC_6H_4, 4-Br-C_6H_4, 4-(OMe)C_6H_4, 4-(Me)C_6H_4, 2-(Me)C_6H_4, 3-C_5H_4N, 4-$(CO_2Me)C_6H_4$, 4-IC_6H_4, 3-BrC_6H_4, 2,3-$Cl_2C_6H_3$, 3-CN, 4-FC_6H_4, 2,4-$(OMe)_2C_6H_3$

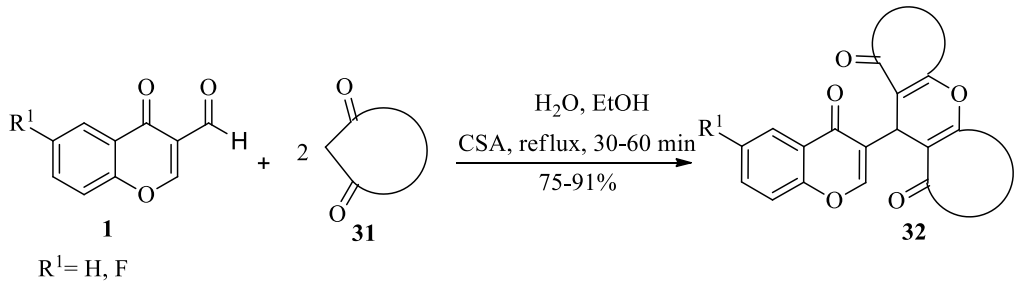
Scheme 13. The synthesis of 5-hydroxychromeno[2,3-*b*]pyridines **30** *via* two methods of **A** and **B**

Its proposed mechanism was demonstrated in Scheme 14**.** In the first step, malononitrile **28** reacted with 3-formylchromone **1** through Knoevenagel condensation reaction[41] to produce the adduct product **A**, which was treated with aromatic amine **29** *via* Michael-type addition to give the intermediate **B**. Finally the target molecule **30** was prepared through intramolecular cyclization of the intermediate **B**.



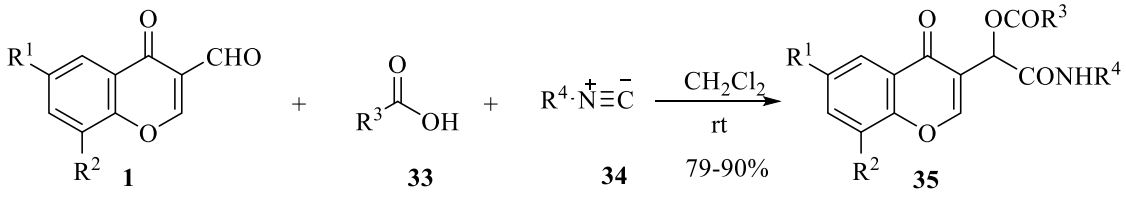
Scheme 14. Plausible mechanism for the synthesis of 5-hydroxychromeno[2,3-*b*]pyridines **30**

In 2016, Kumbhar and co-workers[42] disclosed the synthesis of some coumarin core derivatives **32** from substituted 3-formylchromone **1** and cyclic 1,3-diketone **31** using (±)-camphor-10-sulfonic acid (CSA) as catalyst in H_2O/EtOH as shown in Scheme 15.



Scheme 15. CSA catalyzed synthesis of coumarin core derivatives **32**

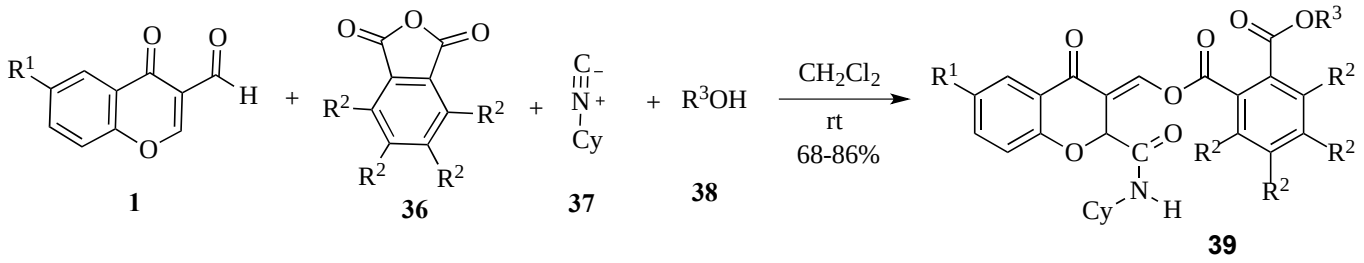
In 2016, Teimouri and co-workers[43] reported the synthesis of 2-(3-chromonyl)-2-acyloxycarboxamide derivatives **35** through the three-component reaction of 3-formylchromones **1**, different alkyl isocyanides **34** and various carboxylic acid derivatives **33** in CH_2Cl_2 at room temperature as shown in Scheme 16.



R^1= H, Cl, Me
R^2= H, Cl
R^3= Br, Cl, C_6H_5, C_8H_7, 2,4-diCl-C_6H_3, 4-NO_2-C_6H_4, C_4H_3O, C_4H_8Br
R^4= C_4H_9, C_6H_{11}, C_7H_7, C_8H_{17}

Scheme 16. The synthesis of 2-(3-chromonyl)-2-acyloxycarboxamides **35**

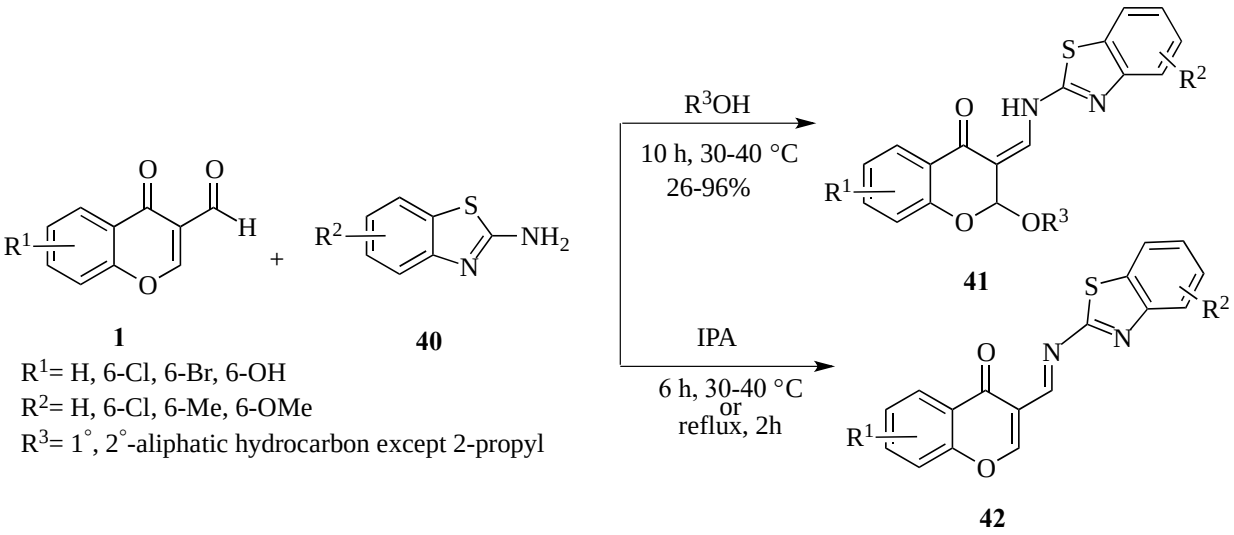
Teimouri and co-workers[44] in 2018 designed the synthesis of the chromone derivatives through the one-pot, four-component reaction of 3-formylchromone **1**, tetrahalophthalic anhydride **36**, cyclohexyl isocyanide **37** and alcohol **38** to synthesize the alkyl [2-[(cyclohexylamino)carbonyl]-4-oxo-2*H*-chromen-3(4*H*)-ylidene]methyl-3,4,5,6-tetrahalophthalates **39** in CH_2Cl_2 at room temperature (Scheme 17).



R^1= H, Cl, Me
R^2= Cl, Br
R^3= H, Et, $(CH_2)_3Me$, $(Me)_2CHCH_2$, $C_6H_5CH_2$, C_6H_{11}, Et, C_3H_5, Me

Scheme 17. The synthesis of desired compound **39**

Owais *et al.* reported a new protocol for the synthesis of 2-alkoxy-3-enamines **41** by the reaction of various 3-formylchromones **1** with different 2-aminobenzothiazoles **40** and primary alcohols in the absence of catalyst (Scheme 18).[45] In another study, it was found that the reaction with the simplest 2˚-alcohol, i.e., 2-propanol (IPA) gave imine **42** (Scheme 18).

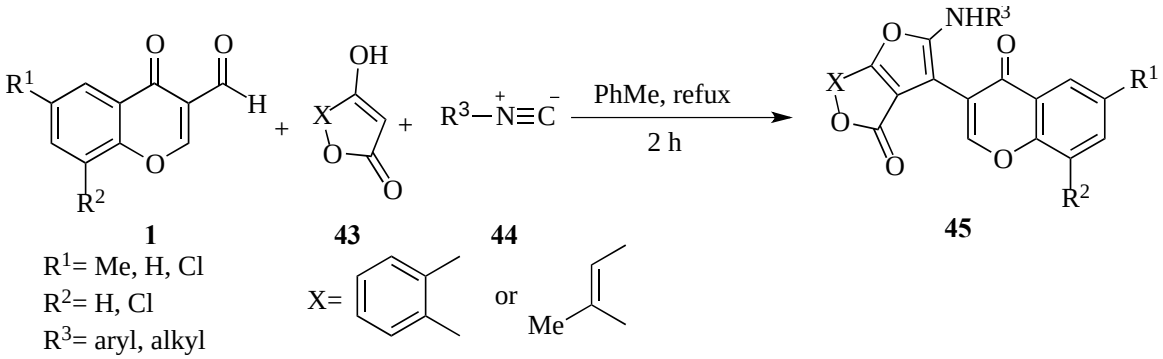


R^1= H, 6-Cl, 6-Br, 6-OH
R^2= H, 6-Cl, 6-Me, 6-OMe
R^3= 1˚, 2˚-aliphatic hydrocarbon except 2-propyl

Scheme 18. The synthesis of 2-alkoxy-3-enamines **41**

New series of furopyranone- and furocoumarin-chromone conjugates **45** were synthesized by Teimouri *et al. via* one-pot condensation of 3-formylchromone derivatives **1**, 4-hydroxycoumarin **43** and alkyl or aryl

isocyanides **44** in toluene under reflux condition for 2 h (Scheme 19). The most derivatives showed good binding tendency in molecular docking studies to cyclooxygenase enzymes. Also, all products revealed varied potencies inhibitory in killing cancerous cells.[46]



Scheme 19. Novel furopyranone- and furocoumarin-chromone conjugates **44**

3. CONCLUSIONS

Consequently, the chemistry of 3-formylchromone and its application have attracted much attention as a synthon for the synthesis of several heterocyclic compounds. All reactions related to 3-formylchromone derivatives in the synthesis of various heterocyclic compounds covering many literatures. This review is the latest researches on the application of 3-formylchromone in the organic reactions which was manifested to follow for further study.

ACKNOWLEDGEMENTS

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