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# Synthesis of 2-Amino-7-hydroxy-4-(4-Bromophenyl)-4H-chromene-3-carbonitrile by using dealuminated mesolite as a heterogenous catalyst

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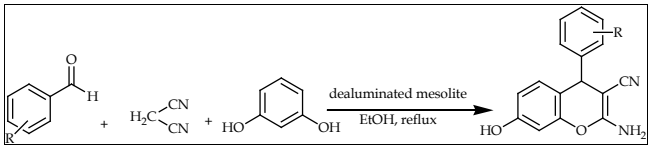
**Abstract**
In recent years 2-Amino-4H-chromenes and their derivatives have attracted strong research interest because of their potent biological and pharmacological significance such as antimicrobial, anti-angiogenesis, antioxidant and antitumor activities. In summary, an efficient catalytic system has been developed for the synthesis of 2-Amino-4H-chromene derivatives via one pot cyclocondensation of benzaldehyde, malononitrile and resorcinol using dealuminated mesolite type natural zeolite as heterogeneous catalyst. The reaction takes place in short time with excellent yield of the product. The present method shows several advantages over reported method such as such as inexpensive, non-toxic and non-corrosive nature of catalyst. Simple recovery and reusability of the catalyst makes the reaction more successful under environmental benign conditions.

**Keywords:** Synthesis, 2-Amino-4H-chromene derivatives, cyclocondensation, zeolite and benzaldehyde etc

## Introduction

In recent years 2-Amino-4H-chromenes and their derivatives have attracted strong research interest because of their potent biological and pharmacological significance such as antimicrobial, anti-angiogenesis, antioxidant and antitumor activities [1, 6]. 4H-chromenes are building blocks of number of oxygen contain heterocyclic natural products like tannin and polyphenol which is present in green tea, fruits and vegetables [7]. Chromene heterocycles is an important constituent in cosmetics, pigments and biodegradable agrochemicals [8]. In addition dihydropyranochromens are well known spasmolytic, diuretic, anti-coagulant agents; furthermore they are utilized as cognitive enhancers, for the treatment of neurodegenerative disease including Alzheimer's disease, Parkinson's diseases, Down's syndrome schizophrenia and myoclonus [9]. Besides this 2-amino-4H-chromenes and pyran derivatives are used as photoactive material to construct the solar cell. [10]. Considering these wide applications of chromene scaffold several catalytic methods were developed for synthesis of 2-amino-4H-chromene derivatives.

In present work, dealuminated mesolite was found to be an active catalyst for one pot synthesis of 2-amino-4H-chromene derivatives via cyclocondensation of aromatic aldehydes, malononitrile resorcinol in ethanol under reflux condition (Scheme 4). Present method offer several advantages over reported protocols such as simple work up procedure, short reaction time, mild reaction conditions, excellent yield and reusability of catalyst.



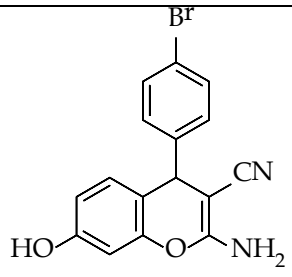

**Scheme 4**

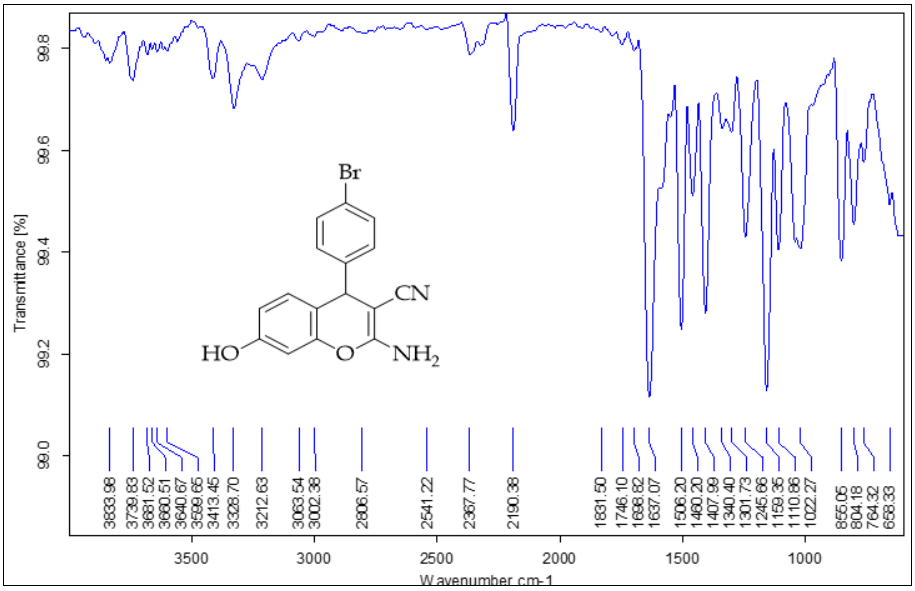
## General Procedure

A mixture of aromatic aldehyde (1 mmol), resorcinol 0.110 g (1 mmol), malononitrile 0.066 g (1 mmol), and catalytic amount dealuminated mesolite (0.1g) was refluxed in ethanol (15 ml) for the time shown in (Table 5.1.22). The progress of the reaction was monitored by thin layer chromatography using ethyl acetate: pet ether (7:3) as a solvent system. After completion of the reaction, the reaction mass was filtered, the filtrate was concentrated, and the crude product obtained was recrystallised from ethanol to afford pure products (4a-c)

## Spectral Data of Representative Compound

2-Amino-7-hydroxy-4-(4-Bromophenyl)-4H-chromene-3-carbonitrile (4c): *White solid, M. P. 110-112 °C+*

| | |
|---|---|
| FT-IR ($cm^{-1}$) 3413 (OH), 3328 ($NH_2$), 2190 (CN), 1506, 1159 |  |




**Results and Discussion**

In order to investigate optimum loading of catalyst and reaction condition, 4-Br benzaldehyde (1mmol), resorcinol (1mmol), were reacted with malononitrile (1mmol) as model reaction with different amount of dealuminated mesolite as catalyst under reflux condition and results are summarized in (Table 1.1). In the absence of a catalyst, the reaction did not yield a satisfactory amount of the desired product (4c), indicating a crucial role of the catalyst. 0.1 g of dealuminated mesolite is suitable for catalysing the reaction smoothly. The same reaction was carried out under different protic and aprotic solvents and it is observed that the reaction proceeds faster in protic solvent such as water and ethanol with maximum yield in reduced time as compare to non protic solvents such as terahydrofuran and actonitrile which gave lower yield.

**Table 1:** Optimization of catalyst loading with different solvents for the synthesis of (4a)

| Entry | Solvent | Catalyst amount (g) | Time (min) | Yield(%)[a] |
|---|---|---|---|---|
| 1 | Ethanol | - | 120 | 30 |
| 2 | Methanol | - | 120 | 25 |
| 3 | Acetonitrile | - | 120 | 23 |
| 4 | 1,4-dioxane | - | 120 | 27 |
| 5 | Tetrahydrofuran | - | 120 | 21 |
| 6 | Water | - | 120 | 40 |
| 7 | Ethanol | 0.05 | 30 | 80 |
| 8 | Ethanol | 0.1 | 30 | 85 |
| 9 | Ethanol | 0.15 | 30 | 85 |
| 10 | Ethanol | 0.2 | 30 | 85 |

In order to explore the scope and importance of the present method, different aromatic aldehydes were tested under optimum reaction conditions for the synthesis of 2-amino-4H-chromen derivatives and the results are summarised. After optimising reaction conditions, efforts have been made towards the recovery and reusability of the catalyst. The catalyst was separated after completion of reaction by diluting reaction mixture by hot ethanol and filtration. The recovered catalyst was washed with acetone and dried at 100 °C for 1 hour before the next catalytic run. Reusability of the catalyst was also investigated for three times and it was found that the catalyst has retained almost consistent activity.

**Table 2:** Dealuminated mesolite catalyzed cyclocondensation reaction of aromatic aldehyde, malononitrile and resorcinol. (°C

| Entry | R | Time (min) | Yield (%)[a] | Melting point (°C) | |
|---|---|---|---|---|---|
| | | | | Observed | Reported |
| 4a | 4-Br | 30 | 92 | 222-224 | 225-227 [17] |
| 4b | 3- $NO_2$ | 30 | 90 | 167-169 | 169-170 [17] |
| 4c | 4-F | 30 | 86 | 191-192 | 187-189 [17] |

Reaction conditions: benzaldehyde (1 mmol), resorcinol (1 mmol), malononitrile, (1 mmol), catalyst 0.1g, and ethanol 15 ml. [a] Isolated yields. [c]Yield after consecutive cycle.

To specify the advantages of proposed method, results of different reported methods are compared with our results. It is found from tabulated results that, dealuminated mesolite promotes reaction more effectively than other reported method.

**Table 3:** Catalytic performance of different catalyst for synthesis of 2-Amino-7-hydroxy-4-(4-methoxyphenyl)-4H-chromene-3-carbonitrile

| Entry | Catalyst | Conditions | Time (min) | Yield (%) | Lit. |
|---|---|---|---|---|---|
| 1 | Nano MgO | $H_2O$, R. T. stirring | 20 | 95 | [11] |
| 2 | (DAB-PPI-$G_1$) | Solvent free 110 °C | 03 | 95 | [13] |
| 3 | Clinoptilolite | $H_2O$, reflux | 20 | 90 | [14] |
| 4 | $Na_2CO_3$ (30 mol %) | $H_2O$: MeOH (95:5) 25 °C | 600 | 75 | [15] |
| 5 | PPI | $H_2O$, reflux | 20 | 96 | [17] |
| 6 | Polyamine/$SiO_2$ | $H_2O$:Ethanol, reflux | 80 | 88 | [18] |
| 7 | Dealuminated mesolite | reflux in ethanol | 30 | 85 | Our results |

**Conclusions**

- In summary, an efficient catalytic system has been developed for the synthesis of 2-Amino-4H-chromene derivatives via one pot cylocon cyclocondensation of benzaldehyde, malononitrile and resorcinol using dealuminated mesolite type natural zeolite as heterogeneous catalyst.

- The reaction takes place in short time with excellent yield of the product.
- The present method shows several advantages over reported method such as such as inexpensive, non-toxic and non-corrosive nature of catalyst. Simple recovery and reusability of the catalyst makes the reaction more successful under environmental benign conditions.

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