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Synthesis of 2-amino-6-methoxy-4-(3-nitrophenyl) quinoline-3-carbonitrile by using dealuminated mesolite as a heterogenous catalyst

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Abstract
In order to investigate optimum loading of catalyst and reaction condition, (1mmol) 4-OCH_3 aniline and (1mmol) 3-NO_2 benzaldehyde were reacted with (1mmol) malononitrile as model reaction with different amount of dealuminated mesolite as catalyst at reflux conditions and the results are summarised in (Table 1.1). In the absence of a catalyst the reaction did not gave satisfactory desired product (3a), which indicate crucial role of catalyst, 0.1 g of delaminated mesolite is suitable to catalyse 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 compared to non-protic solvents such as tetrahydrofuran and acetonitrile which gave lower yield.

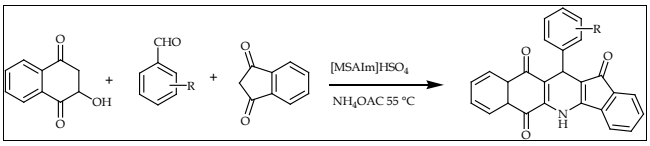
Keywords: Synthesis, mesolite, quinoline-3-carbonitrile derivatives, one-pot cyclocondensation and Yield etc

Introduction

One-pot synthesis methods have played a significant role in organic synthesis because of its several advantages, such as minimum reaction time, high selectivity and atom economy. Additionally, it delivers fewer by-products as compared to classical synthesis, with lower cost, time and energy [1]. Quinoline derivatives are building blocks of a number of nitrogen-containing alkaloids [2]. In recent years quinoline derivatives especially quinoline-3-carbonitrile derivatives have received strong research interest because of their potent biological and pharmacological properties such as antimicrobial, anticancer, antimalarial, cytotoxic, anti-proliferative, DNA binding properties, and inhibitors for phosphodiesterase type 4B enzyme [3, 8]. Besides this, 8-hydroxy quinoline and its derivatives form stable electroluminescent complexes with Al and Zn [9]. Furthermore tetrahydrobenzo [h] quinoline derivatives are well-known dyes [10]. Considering these versatile applications of the quinoline scaffold several methods were developed using various homogeneous and heterogeneous catalyst for the synthesis of quinoline derivatives.

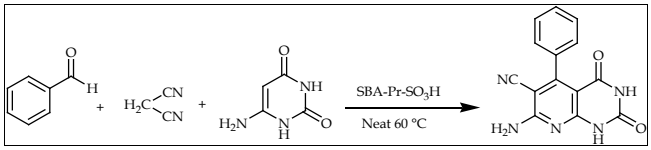
Literature review on quinoline-3-carbonitrile.

N. G. Khaligh *et al* [13], have developed solvent free 3-methyl-1-sulphonic acid imidazolium hydrogen sulfate [MSAIm] HSO4-catalysed one-pot synthesis of benzo [g] indeno[2,1-b] quinoline derivatives via four-component condensation of 2-hydroxynaphthalene-1,4dione, aromatic aldehyde, 2H-indene-1,3-dione and ammonium acetate under grinding condition at 55 °C with 85-86% yield in 40-50 min. (Scheme-1)



Scheme-2

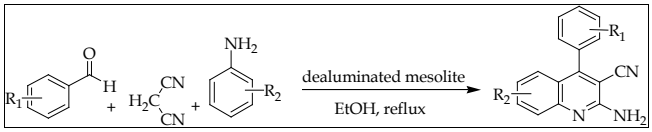
G. M. Ziarani *et al* [14], reported one pot synthesis of pyrido[2,3-d]pyrimidine derivatives by cyclocondensation of aromatic aldehyde, malononitrile and 6-amino uracil by using sulphonic acid functionalized SBA-15 as solid heterogeneous catalyst under solvent-free conditions at at 60 °C with 70-80% yield in 25-30 min. (Scheme-3)



Scheme-3

Present Work

In present work, dealuminated mesolite was found to be active catalyst for one pot synthesis of quinoline -3-carabonitrile derivatives via cyclocondensation of aromatic aldehydes, malononitrile and aromatic amines 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

Result and Discussion

In order to investigate optimum loading of catalyst and reaction condition, (1mmol) 4-OCH_3 aniline and (1mmol) 3-NO_2 benzaldehyde were reacted with (1mmol) malononitrile as model reaction with different amount of dealuminated mesolite as catalyst at reflux conditions and the results are summarised in (Table 1.1). In the absence of a catalyst the reaction did not gave satisfactory desired product (3a), which indicate crucial role of catalyst, 0.1 g of dealuminated

mesolite is suitable to catalyse 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 compared to non-protic solvents such as tetrahydrofuran and acetonitrile which gave lower yield (Table 1.1)

Table 1: Optimisation of catalyst loading with different solvents for the synthesis of 3a

Entry	Solvent	Catalyst amount (g)	Time (min)	Yield(%)[a]
1	Ethanol	-	120	35
2	Methanol	-	120	30
3	Acetonitrile	-	120	25
4	1,4-dioxane	-	120	28
5	Tetrahydrofuran	-	120	23
6	Water	-	120	28
7	Ethanol	0.05	30	80
8	Ethanol	0.1	30	90
9	Ethanol	0.15	30	93
10	Ethanol	0.2	30	93

[a] isolated yield

In order to explore the scope and importance of the present method different aromatic aldehydes and substituted anilines were tested under optimum reaction conditions for synthesis of quinoline-3-carbonitrile and results are summarized in (Table 1.2). After optimizing reaction conditions, efforts have been made towards recovery and reusability of the catalyst. The catalyst was separated by diluting reaction mixture by hot ethanol followed by filtration. The recovered catalyst was washed with acetone and dried at 100 °C for 1 h before the next catalytic run. Reusability of the catalyst was investigated for two times and it was found that the catalyst has retained almost consistent activity (Table 1.2, entry 3a).

Table 2: Dealuminated mesolite catalyzed cyclocondensation of aromatic aldehyde, malononitrile and aniline.

Entry	(R_1)	(R_2)	Time (min)	Yield (%)[a]	Melting point (°C)	
					Observed	Literature
3a.	3-NO_2	4-OCH_3	30	90	82-85	83-88
3b.	4-NO_2	4-CH_3	30	93	95-96	97-98

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

General Procedure

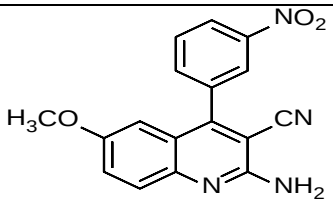
A mixture of aromatic aldehyde (1 mmol), aniline (1 mmol), malononitrle (1 mmol), and catalytic amount dealuminated mesolite (0.1g) was refluxed in ethanol (15ml) for the time shown in (Table. 1.2). The progress of the reaction was monitored by TLC (petroleum ether: ethyl acetate =7:3 as eluent). After completion of the reaction, the reaction mass was filtered, the filtrate was concentrated under reduced pressure, and the crude product obtained was recrystallized from ethanol to afford pure product.

Spectral Data of Representative Compound

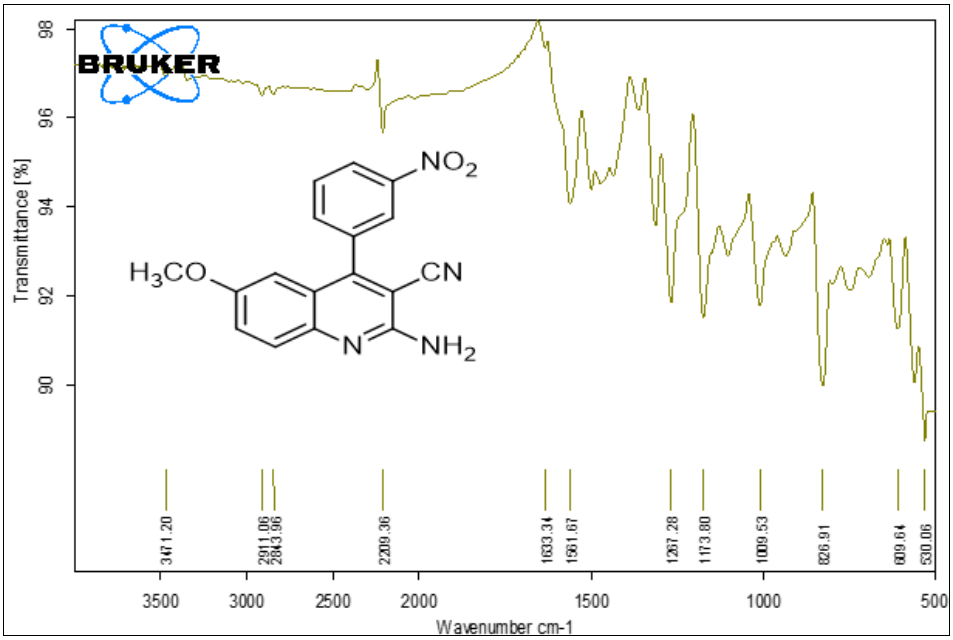
2-amino-6-methoxy-4-(3-nitrophenyl)quinoline-3-carbonitrile (3a) :

Yellow solid, M. P. 95-97 °C

FT-IR (cm^{-1}) 3471 (NH_2), 2209 (CN), 2911, 1561.



2-amino-6-methoxy-4-(3-nitrophenyl)quinoline-3-carbonitrile



FT-IR spectrum of 2-Amino-6-Nitro-4-(4-methoxyphenyl)-quinoline-3-carbonitrile (3a)

Conclusions

- In summary, an efficient catalytic system has been developed for the synthesis of quinoline-3-carbonitrile derivatives via one-pot cyclocondensation of substituted benzaldehyde, aniline and malononitrile and using dealuminated mesolite-type natural zeolite as a green catalyst.
- The reaction takes a short time for completion with an excellent yield of the product.
- The present method shows several advantages over the reported method such as such as inexpensive, non-toxic and non-corrosive nature of the catalyst. Simple recovery and reusability of the catalyst make the reaction more successful under environmentally benign conditions.

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