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Synthesis and Characterization of Some Chromene Derivatives

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ABSTRACT

The current work is focuses on the synthesis ofpharmaceutically importantsubstituted chromene derivatives through one pot reaction multi-component reaction between salicylaldehyde, malononitrile in presence of water as solventusing nano-catalyst at ambient conditions with moderate to good yield. In the present day research age, 4H-chromene and their functional analogues are biologically potent and have attracted significant attentionA survey of the literature revealed that though there are many synthetic approaches exists however most of these are linked to challenges such as harsh reaction condition, extraction, purification and extended reaction time. This fact inspired us to look for a straightforward, green, simple, environmentally friendly work-up synthesis approach. Graphitic carbon nitride (GCN) doped polyaniline composite was prepared and employed to prepare some 4H-chromene derivatives. The synthesized compounds characterized by 1H-NMR, 13C-NMR, MS and FT-IR techniques.

Keywords: Multicomponent, Synthesis, Chromene,Malononitrile, Nano-catalyst.

I. INTRODUCTION

Nowadays heterocyclic compounds attained numerous attention for the synthesis of natural as well as synthetic molecules. Chromene is commonly occur in nature. A frequently utilized component in heterocyclic chemistry is chromene. It has been employed to construct chemical probes for therapeutic and diagnosis purposes[1].Currently versatile field application of chromene like anti-inflammatories[2], antitubercular agents[3], antitumor agent[4], enzyme inhibitors[5], potassium channel activators[6], antimicrobial[7], antiviral[8]were shown by the different group of researcher.

Multicomponent reaction (MCR) efficiently used by Nagao research group. Knoevenagel condensation of malononitrilefor the synthesis of 2-amino-5-carboxy-4-aryl-4H-pyran-3-carbonitriles. This reaction is supposed to proceed through 2-benzylidenemalononitrile intermediate which on ring closure result in 4H-pyran[9].

In addition to looking for an alternate, eco-friendly reaction medium, green chemistry aims to lower reaction temperatures and boost reaction rates[10].Organic synthesis assisted with nano-catalyst attracting much attention due to its ease of functionalization, eco-friendly, recyclability, toxic free, commercially acceptable and most important easy extraction from the reaction mixture[11].Polyaniline has suffering with certain drawbacks for the actual commercial application like low surface area, mechanical degradation, limited catalytic activity. Doping is one of the efficient way to alter the physicochemical, morphological features of PANI. Pd and Pt doped PANI mediated organic transformation like oxidation of methanol[12], glycerol[13]etc. showed the useful application in various organic synthesis. Therefore in the present work we highlighted the application of GCN doped polyaniline nano-catalyst for the synthesis of 4H-chromene derivatives. Some substituted 2 - amino - 3 - cyano - 4H - chromenehave been prepared by one - pot two - component reaction between salicylaldehyde, malononitrilein water usingnano-catalyst. This synthesis protocol was found the convenientmethod for the synthesis of the variety of 4H-chromenederivatives which were characterized by 1H-NMR,MS and FT-IR.

Origin of Problem :

The above literature review reveals that despite usefulness of various catalyst, some of them uses harsh conditions, several steps, complex synthetic procedure. Hence it was interested to develop the green protocol to prepare 2-amino-4H-chromene derivatives using GCN doped polyaniline nano-catalyst and water as solvent.

II. METHODS AND MATERIALS

AR gradechemicals of Merkhave been used for synthesis. FT-IR spectra have been recorded on a Bruker Germany 3000 Hyperion microscope. The 1H NMR spectraat 500 MHzfromaBrukerAvance Neo NMRSpectrometer.Mass spectra collected from SYNAPT-XS DBA064 instrument.

III.EXPERIMENTAL

General procedure forsynthesis of substituted 4H-chromenederivatives:

In an oven-dried round bottom flask, salicylaldehyde (1 mmol), malononitrile (2 mmol), catalytic amount of doped PANI and water (5 mL) were transferred sequentially at optimum conditions[14]. This reaction mixture was then stirred continuously and vigorously for hours.The reaction progress was monitored by TLC[15].After reaction completion, a solid mass precipitated out when distilled water was added into the resulted mixture.It was filtered, washed with aqueous ethanol (Fig.1). The structure of synthesized compound was confirmed by 1HNMR, 13CNMR MS and FT-IR.

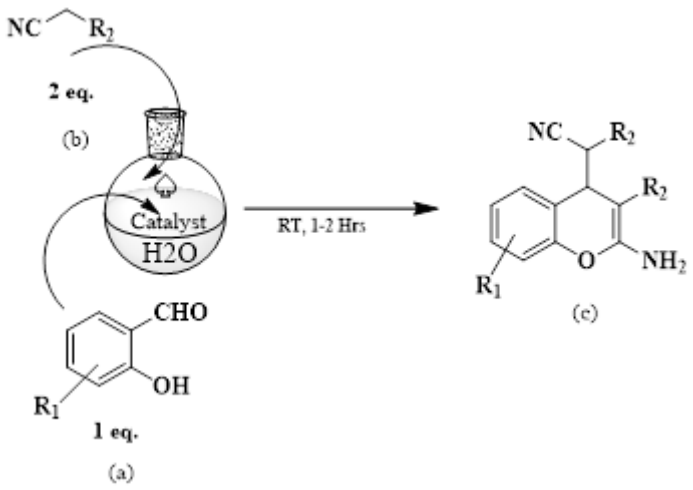
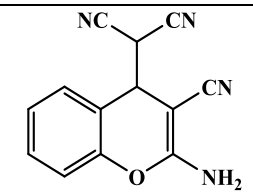
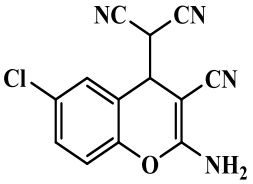
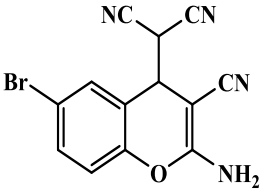


Fig.1 Theschematic representation ofsynthesis of 4H-chromene derivatives

IV. RESULT AND DISCUSSION:

There are some substituted 4H-chromenederivatives have been synthesized and theyfoundto havethe goodyield.

Table 1: Representative data of synthesized 4H-pyrans

SrNo	Product	R_1 in (a)	R_2 in (b)	Time (Hrs.)	Isolated Yield (%)
1		-H	-CN	1	89
2		-Cl	-CN	1.5	86
3		-Br	-CN	2	78

The Representative Analytical Data of Synthesized Compounds:

2-(2-amino-3-cyano-4H-chromen-4-yl) malononitrile (Table Entry 1)

Molecular Formula- **C13H8N4O;** Mol.Weight-236.23; Yellowish solid; M.P. 150 - 152° C; Yield: 89%;1H NMR (500 MHz, DMSO): δ 6.7 (s, 2H, Ar—NH2), 7.3 (d, 2H, Ar—H), 3.8 (d, 1H, C—H), 7.5 (t, 2H, Ar—H), 3.6 (1, 1H, —C4H pyran), FT-IR(υ max: cm-1); 3502 (-NH2), 1550 (C≡N), 1218 (CO), 1488 (C=C).

2-(2-amino-6-chloro-3-cyano-4H-chromen-4-yl)malononitrile(Table Entry 2)

Molecular Formula- **C13H7ClN4O;** Mol.Weight-270.68; Yellow solid; M.P. 154 - 156° C; Yield: 86%; 1H NMR (500 MHz, DMSO): δ 6.80 (s, 2H, NH2), 7.14 (d, 1H, Ar—H), 7.86 (s, 1H, Ar—H), 7.57 (d, 1H, Ar—H), 5.08 (d, 1H, (CN)2-CH), 3.37 (1, 1H, —C4 H pyran), FT-IR (υ max: cm-1); 3420 (-NH2), 1760 (C≡N), 1215 (CO), 1541 (C=C), 728 (C-Cl).

2-(2-amino-6-bromo-3-cyano-4H-chromen-4-yl)malononitrile (Table Entry 3)

Molecular Formula- **C13H7BrN4O;** Mol.Weight- 315.53;Yellowish brown solid; M.P. 170 - 172° C; Yield: 78%;1H NMR (500 MHz, DMSO): δ .90 (s, 2H, -NH2), 8.72 (s, 1H, Ar-H), 8.42 (d, 1H, Ar—H), 8.15 (d, 1H, Ar—H), 3.32 (d, 1H, (CN)2-CH), 2.50 (1, 1H, —C4 H pyran), FT-IR (υ max: cm-1); 3525 (-NH2), 1558 (C≡N), 1216 (CO), 1489 (C=C), 636 (C-Br).

V. CONCLUSION

This paper focus on the GCN doped polyaniline catalyzed synthesis of 4H-chromene derivatives using one pot multicomponent method. The nano-catalyst has feasible synthetic condition, safe, easily operated andefficient.It is worth mentioning that the reaction time was appreciably reduced by using novel nano-catalyst.

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