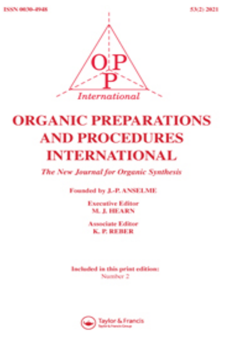

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One-Pot Expeditious Synthesis of 2-Amino-4,6-(disubstituted)nicotinonitriles Using Activated Fuller's Earth as Catalyst

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Pyridine is a ubiquitous privileged heteroaromatic found in numerous natural products, including vitamins B3 and B6, nicotine, and nicotinamide adenine dinucleotide phosphate (NADP).[1–4] The pyridine ring is a key structural motif present in numerous drugs with important biological activities such as anti-HIV,[5] antimicrobial,[6] and anti-inflammatory[7] properties. Pyridine-containing molecules are potent phosphodiesterase 4 (PDE4) inhibitors that are beneficial for treating asthma and chronic obstructive pulmonary disease (COPD).[8,9] In addition, pyridines are applied in coordination chemistry,[10] materials science,[11] supramolecular structures,[12] organocatalysis,[13] polymers, nonlinear optical materials, electrical materials and agrochemicals.[1] Among the pyridine substructures, 2-amino-4,6-(disubstituted)nicotinonitriles (**5**, Scheme 1) are known for their remarkable pharmacological activities, including anti-tumor,[14] cardiotonic,[15] and anti-inflammatory[16] characteristics.

2-Amino-4,6-(disubstituted)nicotinonitriles have conventionally been synthesized by the four-component reactions of aldehydes, acetophenone, malononitrile, and ammonium acetate[17] in the presence of such promoters as diphenyl ammonium triflate,[18] montmorillonite K-10,[9] $[EtNH_3]NO_3$,[19] microwave irradiation,[20,21] $Yb(PFO)_3$,[22] $FePO_4$,[23] urease,[24] $Ti(dpm)(NMe_2)_2$,[25] $[Bmim][BF_4]$,[26] cellulose-SO_3H,[27] ultrasonic irradiation,[28,29] SBTETASA,[30] and trifluoroethanol.[31]

In keeping with our previous experiments in catalysis,[32–51] we structured the present work to investigate the suitability of activated Fuller's earth[34] as a catalyst for the synthesis of 2-amino-4,6-(disubstituted)nicotinonitriles. For our initial model reaction, we chose benzaldehyde (2 mmol), acetophenone (2 mmol), malononitrile (2 mmol), and ammonium acetate (2.4 mmol) as reactants, and investigated solvent-free conditions in the production of compound **5a** (Tables 1 and 2).

The significance of catalyst loading on yields and time of the reaction was assessed (Table 1, entries 2-6). In the absence of a catalyst at 90 °C, the reaction occurred with only traces of the product, even after 120 minutes (Table 1, entry 1). An increase of the catalyst loading increased the product yield with a reduction in reaction time (Table 1,

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Scheme 1. Schematic representation of activated Fuller's earth catalyzed synthesis of 2-amino-4,6-diphenylnicotinonitrile.

Table 1. Effect of catalyst loading.[a]

Entry	Catalyst (wt %)	Temperature (°C)	Time (min.)	Yield[b] (%)
1	–	90	120	Traces
2	5	90	15	35
3	10	90	15	58
4	15	90	15	78
5	20	90	15	93
6	30	90	15	93
7	20	rt	120	Traces
8	20	50	15	60
9	20	70	15	68
10	20	110	15	94

[a] Reaction conditions: benzaldehyde (2 mmol), acetophenone (2 mmol), malononitrile (2 mmol), and ammonium acetate (2.4 mmol).

[b] Isolated yield.

Table 2. Effect of the solvents for the synthesis of 2-amino-4,6-diphenylnicotinonitrile.[a]

Entry	Solvent	Temperature (°C)	Time (min.)	Yield[b] (%)
1	Solvent-free	90	15	93
2	H_2O	90	120	29
3	EtOH	reflux	120	68
4	THF	reflux	120	53
5	MeCN	reflux	120	61
6	Toluene	90	120	25
7	DMF	90	120	78

[a] Reaction conditions: benzaldehyde (2 mmol), acetophenone (2 mmol), malononitrile (2 mmol), ammonium acetate (2.4 mmol), and activated Fuller's earth (20 wt. %).

[b] Isolated yield.

entries 2-5). However, catalyst loading beyond 20 wt. % was not advantageous (Table 1, entries 5 and 6), so 20 wt. % catalyst loading was chosen for further work. Temperature also played an important role in our model reaction. The best results were obtained at 90 °C (Table 1, entry 5).

The effect of several solvents on time and yield of the reaction was ascertained (Table 2). None of the solvents had an advantage of time and yield over the solvent-free conditions. Hence, the solvent-free protocol was regarded as the best for cost and environmental acceptability.

Table 3 shows a comparison of catalysts reported in the literature for the preparation of the title compounds. The present method ranks among the best in terms of reaction conditions, workup procedure, time and yields.

Table 3. Comparison of the efficiency of activated Fuller's earth with literature catalysts.

Entry	Catalyst	Reaction conditions	Time	Yield[a] (%)	Ref.
1	Activated Fuller's earth	Solvent free/90 °C	15 min.	93	This work
2	Fe_3O_4@Niacin) microwave	H_2O/70 °C	10 min.	86	52
3	Fe_3O_4@TiO_2@$O_2PO_2(CH_2)NHSO_3H$	Solvent free/90 °C	20 min.	90	53
4	Perchlorated Al-MCM-41 nanoparticles	Solvent free/100 °C	20 min.	73	54
5	Ultrasound irradiation	CH_3COONH_4	25 min.	91	28, 29
6	{Fe_3O_4@SiO_2@$(CH_2)_3$Im}C(CN_3	Solvent free/100 °C	40 min.	86	55
7	Nano-Cu_2O–MFR	EtOH/ H_2O/reflux	1 h	92	56
8	TBAB	H_2O/reflux	2 h	92	57
9	Cellulose–SO_3H	H_2O/60 °C	2.5 h	95	27
10	$FePO_4$	EtOH/reflux	4 h	93	23
11	SnO_2/SiO_2 nanocomposite	EtOH/reflux	4 h	91	58
12	[Yb(PFO)$_3$]	EtOH/reflux	4 h	90	22
13	Graphene oxide	H_2O/80 °C	5 h	96	59
14	Copper nanoparticles on charcoal (Cu/C)	MeCN/80 °C	6 h	91	60

[a]Isolated yield.

Table 4. Substrate scope.

Entry	Aldehydes (R_1)	Ketones (R_2)	Product	Time (min)	Yield[a] (%)
1	C_6H_5	C_6H_5	**5a**	15	93
2	4-CH_3O-C_6H_4	C_6H_5	**5b**	15	87
3	4-HO-C_6H_4	C_6H_5	**5c**	20	85
4	4-Br-C_6H_4	C_6H_5	**5d**	15	92
5	4-Cl-C_6H_4	C_6H_5	**5e**	15	91
6	4-CH_3-C_6H_4	C_6H_5	**5f**	15	90
7	4-NO_2-C_6H_4	C_6H_5	**5g**	15	89
8	3-Br-C_6H_4	C_6H_5	**5h**	20	87
9	3-NO_2-C_6H_4	C_6H_5	**5i**	25	86
10	2-Cl-C_6H_4	C_6H_5	**5j**	30	82
11	2-NO_2-C_6H_4	C_6H_5	**5k**	30	80
12	Thien-2-yl	C_6H_5	**5l**	40	85
13	C_6H_5	4-CH_3O-C_6H_4	**5m**	20	86
14	C_6H_5	4-HO-C_6H_4	**5n**	20	84
15	C_6H_5	4-Br-C_6H_4	**5o**	15	92
16	C_6H_5	4-Cl-C_6H_4	**5p**	15	90
17	C_6H_5	3-Br-C_6H_4	**5q**	20	88
18	C_6H_5	3-NO_2-C_6H_4	**5r**	25	86
19	4-CH_3O-C_6H_4	4-Br-C_6H_4	**5s**	20	91
20	4-Cl-C_6H_4	4-Br-C_6H_4	**5t**	15	92
21	4-CH_3-C_6H_4	4-Br-C_6H_4	**5u**	15	89
22	4-CH_3O-C_6H_4	3-Br-C_6H_4	**5v**	20	90
23	4-CH_3-C_6H_4	3-Br-C_6H_4	**5w**	20	92
24	3-Br-C_6H_4	3-Br-C_6H_4	**5x**	25	86

[a]Isolated yield.

Table 4 shows the results of applying our procedure more broadly. We prepared numerous 2-amino-4,6-(disubstituted)nicotinonitriles in very good to excellent yields (82-93%) in short reaction times. The nature of substitution has no significant effect on reaction time and yields. This protocol tolerates a variety of substituents on substituted aldehydes and substituted acetophenones. Table 5 provides melting points and literature references.

The reusability of the catalyst in the model reaction under solvent-free conditions at 90 °C was evaluated. We found that the recovered catalyst could be recycled as many as four times with no significant loss in catalytic activity (fourth run 89% yield).

In summary, the present work describes the scope of activated Fuller's earth as a catalyst for the formation of 2-amino-4,6-(disubstituted)nicotinonitriles in the reaction of

Table 5. Physical constants of 2-amino-4,6-(disubstituted)nicotinonitriles, **5a-x**.

Entry	Observed M. P. (°C)	Literature M. P. (°C)	Entry	Observed M. P. (°C)	Literature M. P. (°C)
5a	183-185	184-186[53]	**5m**	168-170	167-169[59]
5b	178-180	177-178[53]	**5n**	231-233	233-235[55]
5c	219-221	221[61]	**5o**	172-174	174-176[63]
5d	197-198	195-197[55]	**5p**	248-250	247-249[53]
5e	228-229	228-230[59]	**5q**	145-147	NA
5f	168-170	170-172[59]	**5r**	190-192	190-193[53]
5g	231-233	230-232[53]	**5s**	181-183	180-182[27]
5h	166-168	165-167[53]	**5t**	112-114	112[58]
5i	190-192	190-193[53]	**5u**	235-237	236-237[56]
5j	193-195	194-197[55]	**5v**	180-182	NA
5k	140-142	NA	**5w**	178-180	NA
5l	189-191	190-192[62]	**5x**	170-172	NA

NA = not available in the literature.

aldehydes, acetophenones, malononitrile, and ammonium acetate products in very good to excellent yields under solvent-free conditions.

Experimental section

All the chemicals and solvents used were of LR grade and purchased from SD Fine, Avra Synthesis, and Spectrochem and used as received. The purity determination of the starting materials and the reaction monitoring was accomplished by thin-layer chromatography (TLC) on Merck silica gel G F_{254} plates, with ethyl acetate:hexane as the eluting solvent. Melting points of all the compounds were recorded on an Analab ThermoCal melting point apparatus in open capillary tubes and are uncorrected. ^{1}H NMR spectra were recorded on an MR400 Agilent Technology NMR spectrometer using tetramethylsilane as an internal standard and DMSO-d6 or $CDCl_3$ as a solvent. HRMS was recorded on a Shimadzu LCMS-QTOF-9030 instrument by the direct infusion method. All compounds were characterized by melting points, ^{1}H NMR and HRMS spectroscopy. Characterization data are provided below for the novel compounds. For the novel compounds, complete copies of the original spectra were submitted for review and are available in the supplementary material in the online version of this article or from the corresponding author upon request. Covid-19 restrictions precluded reliance on the customary elemental analysis. Known compounds were identified by matching their melting points with the values in the literature references cited in Table 5.

Preparation of activated Fuller's earth catalyst

Commercial grade Fuller's earth (10 g) was stirred with 100 ml of distilled water to separate any water-soluble material. The suspended solid was filtered, washed with distilled water, and dried in an oven at 100 °C for 1h. To a 250 ml round bottom flask containing 50 ml of 5% HCl solution was added 5 g of solid obtained from the above treatment. The mixture was heated at 100 °C for 4h, cooled to room temperature, and filtered; the residue was washed with distilled water to neutrality. The resultant activated Fuller's earth was dried at 100 °C to constant weight.

General procedure for the synthesis of 2-Amino-4,6-(disubstituted)nicotinonitriles (5a-x)

A mixture of substituted aldehyde (2 mmol), substituted acetophenone (2 mmol), malononitrile (2 mmol), ammonium acetate (2.4 mmol), and activated Fuller's earth (20 wt. %) was heated at 90 °C in an oil bath. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the reaction mass was treated with hot ethanol (5 mL) dissolving the organic product and leaving the Fuller's earth insoluble. The Fuller's earth was removed by hot filtration. The filtrate was evaporated to get the product. The product was purified by recrystallization from ethanol.

2-Amino-4-(2-nitrophenyl)-6-phenylnicotinonitrile (5k)

^{1}H NMR (400 MHz, DMSO-d6) δ 8.23 (d, J = 8.2 Hz, 1H), 8.12 – 8.02 (m, 2H), 7.94 – 7.85 (m, 1H), 7.79 (dd, J = 17.7, 9.6 Hz, 1H), 7.64 (d, J = 7.6 Hz, 1H), 7.45 (d, J = 3.1 Hz, 3H), 7.24 (s, 1H), 7.10 (s, 2H). HRMS calculated for $C_{18}H_{12}N_4O_2$: 316.09602; found: (MH$^+$) 317.10234 *m/z.*

2-Amino-6-(3-bromophenyl)-4-phenylnicotinonitrile (5q)

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.33 (s, 1H), 8.11 (d, J = 8.0 Hz, 1H), 7.65 (m, 3H), 7.52 (d, J = 6.8 Hz, 3H), 7.41 (t, J = 7.6 Hz, 1H), 7.32 (s, 1H), 7.04 (s, 2H). HRMS calculated for $C_{18}H_{12}BrN_3$: 349.02145; found: (MH$^+$) 350.02764 *m/z.*

2-Amino-6-(3-bromophenyl)-4-(4-methoxyphenyl)nicotinonitrile (5v)

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.32 (m, 1H), 8.10 (s, 1H), 7.65-7.63 (m, 2H), 7.43-7.41 (m, 1H), 7.08-6.98 (m, 4H), 3.81 (s, 3H). HRMS calculated for $C_{19}H_{14}BrN_3O$: 379.03202; found: (MH$^+$) 382.03616 *m/z.*

2-Amino-6-(3-bromophenyl)-4-(p-tolyl)nicotinonitrile (5w)

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.35 (s, 1H), 8.13 (d, J = 7.8 Hz, 1H), 7.67 (d, J = 7.9 Hz, 1H), 7.59 (d, J = 7.9 Hz, 2H), 7.45 (t, J = 7.9 Hz, 1H), 7.36 (d, J = 7.9 Hz, 2H), 7.32 (s, 1H), 7.06 (s, 2H), 2.40 (s, 3H). HRMS calculated for $C_{19}H_{14}BrN_3$: 363.03710; found: (MH$^+$) 364.04324 *m/z.*

2-Amino-4,6-bis(3-bromophenyl)nicotinonitrile (5x)

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.34 (s, 1H), 8.13 (d, J = 8.8 Hz, 1H), 7.86 (s, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.65 (t, J = 7.2 Hz, 2H), 7.50-7.42 (m, 2H), 7.35 (s, 1H), 7.09 (s, 2H). HRMS calculated for $C_{18}H_{11}Br_2N_3$: 426.93196; found: (MH$^+$) 429.3570 *m/z.*

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