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ORIGINAL PAPER

An efficient, green solvent-free protocol for the synthesis of 2,4,6-triarylpyridines using reusable heterogeneous activated Fuller's earth catalyst

Deelip S. Rekunge[1] · Ishwari A. Kale[1] · Ganesh U. Chaturbhuj[1]

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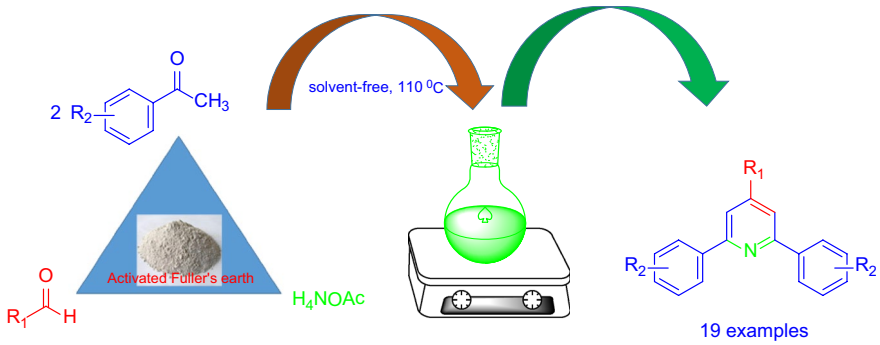
Abstract

A simple, efficient, and green method of preparation for the synthesis of highly substituted pyridines by multicomponent reaction of acetophenones, aldehydes, and ammonium acetate using activated Fuller's earth as an effective and reusable heterogeneous catalyst is described. The advantages of the present protocol include simple procedure with an easy workup procedure, mild reaction conditions, and high yields of the products. The performance of this reaction under solvent-free conditions using heterogeneous catalysts, such as activated Fuller's earth, could enhance its efficiency from an economic as well as ecological point of view.

Graphical abstract



- Recyclable and non-corrosive catalyst
- Metal-free synthesis
- No column chromatography

Keywords Activated Fuller's earth · Triarylpyridines · Ammonium acetate · Solvent-free condition

Introduction

The *N*-heterocyclic pyridine compounds, mainly 2,4,6-triarylpyridine, are of enormous attention owing to their wide range of biological and pharmaceutical properties such as anti-convulsant, anesthetic, anti-malarial, vasodilator, anti-epileptic character (Fig. 1), and they are also used in

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✉ Ganesh U. Chaturbhuj
gu.chaturbhuj@gmail.com

[1] Department of Pharmaceutical Sciences and Technology, Institute of Chemical Technology, Mumbai, Maharashtra 400019, India

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agro-chemicals as pesticidal, fungicidal, and herbicidal [1–3]. Nowadays, this molecule has made it a prime target for scientific research because of the presence of the pyridine ring system in natural products, such as NAD nucleotides, pyridoxal (vitamin B_6), pyridine alkaloids, and a number of pharmacologically significant molecules [4]. They are important in supramolecular chemistry due to their π-stacking ability and directional H-bonding capacity [5–8]. In addition, due to excellent thermal stabilities of these pyridines, they are used as monomeric building blocks in organometallic polymers and thin films [9–12]. 2,4,6-Triarylpyridines have been directed for photodynamic cell-specific cancer therapy having structure similarities with symmetrical triaryl-thiopyrylium, triarylselenopyrylium, and triaryl-telluropyrylium photosensitizers [13], also these molecules found to be useful for the synthesis of DNA binding ligands, as a possible target in cancer therapy targeting G-quadruplex DNA [14–18].

Traditionally, 2,4,6-triarylpyridines (Kröhnke-pyridine) synthesis occur through the reaction of *N*-phenacyl pyridinium salts with α, β-unsaturated ketones in the presence of ammonium acetate (NH_4OAc) [19, 20], but this method is relatively expensive, time-consuming, and harmful as ecological point of view, also the pyridinium salts and unsaturated ketones have to be synthesized first, in this method. Recently, several improved methods have been developed for the synthesis of 2,4,6-triaryl pyridines, viz., reaction of α-ketoketene dithioacetals with methyl ketones in the presence of NH_4OAc [21], reaction of *N*-phosphinylethanimines with aldehydes [22], solvent-free reaction of chalcones with ammonium acetate [23], solvent-free reaction between acetophenones and benzaldehydes, and NH_4OAc in the presence of various catalysts such as PEG-400 [24], $HClO_4{\cdot}SiO_2$ [25], catalytic amount of acetic acid [26], $H_{14}[NaP_5W_{30}O_{110}]$ [27], molecular iodine [28], L-proline [29], microwave irradiation without catalyst [30], [BmIm] $[BF_4]$ [31], wet 2,4,6-trichloro-1,3,5-triazine [32], pentafluorophenyl ammonium triflate [33], trichloroisocyanuric acid [34], bismuth triflate [35], and $MgAl_2O_4$ nanocrystals [36] have been employed to promote this transformation. Among these, the multicomponent one-pot reaction of aromatic ketones, aldehydes, and ammonium acetate is one of the simplest methods.

However, many of these protocols suffer from drawbacks such as the use of costly and corrosive catalysts, acidic media, and toxic organic solvents, undesired side products of the reaction with harsh reagents, long reaction time, cumbersome product isolation procedures, and environmental pollution. To avoid such drawbacks, the development of greener, simple, economical, and efficient protocols are still in demand.

A green chemistry protocol involves considerations, such as atom economy, non-hazardous catalysts, solvent-free conditions, and process simplicity. Therefore, to overcome this problem associated with the synthesis of pyridines, it is highly desirable to develop a powerful method of synthesis to meet the requirement of green chemistry. In this regard, Fuller's earth is a commercially available, non-toxic, eco-friendly, economic material. It played a pivotal role under heterogeneous conditions by catalyzing various organic transformations; this is due to tangible benefits such as non-corrosiveness, ease of preparation, handling, regeneration, low cost, and insolubility in most of the organic solvents. The catalytic activity of activated Fuller's earth is due to their Bronsted as well as Lewis acidic characters in their natural form. It has been used to analyze color additives in food products and as an adsorbent in pharmaceutical and cosmetics. These qualities make it safer and suitable for both laboratories as well as industrial processes. In connection with our continuing studies recently, our lab has prepared, characterized stable, and easily handled clay-based catalyst, such as activated Fuller's earth and sulfated polyborate, as well as aluminized polyborate, and demonstrated its effectiveness as an acid catalyst for the development of novel methodologies to synthesize important heterocycles [37–54, 67]. Herein, we wish to report an efficient, and green method for the synthesis of 2,4,6-triarylpyridines by one-pot three-component reaction of acetophenone, aryl aldehydes, and ammonium acetate using activated Fuller's earth catalyst under solvent-free conditions. To the best of our knowledge, there is no report in the literature on the use of activated Fuller's earth in the synthesis of 2,4,6-triarylpyridines.

Experimental

All the chemicals and solvents used were of LR grade and purchased from SD fine, Avra Synthesis, and Spectrochem, and used as received. Melting points of all the compounds were recorded by Analab ThermoCal melting point apparatus in the open capillary tube and are uncorrected. The FTIR spectra (KBr) were recorded on Shimadzu FTIRAffinity-1 Fourier Transform infrared spectrophotometer. ^{1}H NMR spectra were recorded on MR400 Agilent Technology NMR spectrometer using tetramethylsilane (TMS) as an internal standard and $CDCl_3$ as a solvent. X-ray diffractograms (XRD) were recorded on Shimadzu X-ray diffractometer. The SEM-EDAX characterization was performed on a JEOL JSM-638DLA scanning electron microscope equipped with energy dispersive X-ray spectrometer. The purity determination of the starting materials and reaction monitoring was accomplished by thin-layer chromatography (TLC) on Merck silica gel G F_{254} plates. All the products are known compounds and were identified by ^{1}H NMR spectroscopy for structural identification.

Preparation of activated Fuller's earth

The activated Fuller's earth catalyst was prepared as per procedure reported in the literature [37].

General procedure for the synthesis of 2,4,6-triphenylpyridine

A mixture of benzaldehyde (2 mmol), acetophenone (4 mmol), ammonium acetate (2.4 mmol), and activated Fuller's earth (10 wt%) was heated at 110 °C in an oil bath. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the insoluble product was dissolved in hot ethanol to separate activated Fuller's earth by filtration, and the filtrate was evaporated to get the product. The product was technically pure subject to purification by recrystallization for spectral analysis. The products obtained were known compounds and identified by their melting point and ^{1}H NMR spectroscopy, and the analytical data were compared with the literature values.

Procedure for the recyclability study

Recyclability of the catalyst is an important attribute for the industrial suitability. Therefore, reusability of the catalyst in the model reaction of acetophenone, benzaldehyde, and ammonium acetate under a solvent-free condition at 110 °C was evaluated. After completion of the reaction, the insoluble crude product was dissolved in hot ethanol, and activated Fuller's earth was recovered by filtration. After filtration catalyst was washed with ethanol to remove the organic traces. The catalyst was dried at 60 °C for 1 h and reused for successive four cycles with no remarkable loss of yield.

Representative spectral data

^{1}H NMR spectra of 2,4,6-triphenylpyridine (Table 3, entry 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 2H), 8.11 (d, J = 8.4 Hz, 2H), 7.87 (s, 2H), 7.74 (d, J = 6.8 Hz, 2H), 7.59–7.49 (m, 5H), 7.39 (t, J = 7.8 Hz, 2H).

^{1}H NMR spectra of 4-(4-chlorophenyl)-2,6-diphenylpyridine (Table 3, entry 2)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 8.4 Hz, 4H), 7.84 (s, 2H), 7.68 (d, J = 7.6 Hz, 2H), 7.52–7.49 (m, 8H).

^{1}H NMR spectra of 4-(4-nitrophenyl)-2,6-diphenylpyridine (Table 3, entry 4)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, J = 8.8 Hz, 2H), 8.21 (d, J = 7.2 Hz, 4H), 7.98–7.88 (m, 4H), 7.55–7.46 (m, 6H).

^{1}H NMR spectra of 4-(4-methoxyphenyl)-2,6-diphenylpyridine (Table 3, entry 5)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 7.2 Hz, 4H), 7.88 (s, 2H), 7.71 (d, J = 8.8 Hz, 2H), 7.51 (t, J = 7.2 Hz, 4H), 7.45 (d, J = 7.2 Hz, 2H), 7.05 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H).

^{1}H NMR spectra of 4-(3-bromophenyl)-2,6-diphenylpyridine (Table 3, entry 7)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, J = 5.6 Hz, 4H), 7.88 (s, 1H), 7.84 (s, 2H), 7.52–7.38 (m, 9H).

^{1}H NMR spectra of 2,6-diphenyl-4-styrylpyridine (Table 3, entry 9)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 7.6 Hz, 4H), 7.89 (s, 2H), 7.75 (d, J = 8.0 Hz, 2H), 7.53–7.44 (m, 11H).

^{1}H NMR spectra of 2,6-diphenyl-4-(thiophen-2-yl) pyridine (Table 3, entry 11)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 7.6 Hz, 4H), 7.87 (s, 2H), 7.63–7.62 (m, 1H), 7.54–7.45 (m, 7H), 7.19–7.17 (m, 1H).

^{1}H NMR spectra of 4-phenyl-2,6-di-*p*-tolylpyridine (Table 3, entry 13)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, J = 8.0 Hz, 4H), 7.83 (s, 2H), 7.74 (d, J = 7.6 Hz, 2H), 7.54–7.46 (m, 3H), 7.31 (d, J = 8.0 Hz, 4H), 2.43 (s, 6H).

^{1}H NMR spectra of 2,6-bis(3-bromophenyl)-4-phenylpyridine (Table 3, entry 14)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 2H), 8.11 (d, J = 7.6 Hz, 2H) 7.87 (s, 2H), 7.73 (d, J = 6.8 Hz, 2H), 7.59–7.49 (m, 5H), 7.39 (t, J = 7.8 Hz, 2H).

^{1}H NMR spectra of 2,4,6-tri-*p*-tolylpyridine (Table 3, entry 17)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, J = 8.0 Hz, 4H), 7.81 (s, 2H), 7.63 (d, J = 8.0 Hz, 2H), 7.30 (t, J = 7.2 Hz, 6H), 2.41 (s, 9H).

^{1}H NMR spectra of 2,6-bis(3-bromophenyl)-4-(*p*-tolyl)pyridine (Table 3, entry 18)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 2H), 8.10 (d, J = 7.6 Hz, 2H), 7.85 (s, 2H), 7.64 (d, J = 7.6 Hz, 2H), 7.57 (d, J = 7.6 Hz, 2H), 7.46–7.33 (m, 4H), 2.45 (s, 3H).

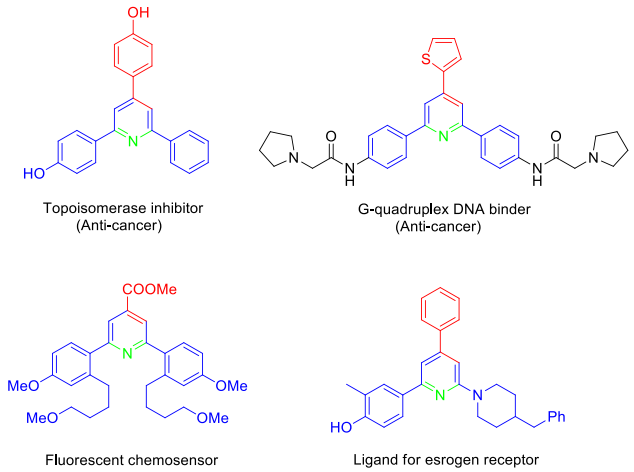


Fig. 1 Pharmacologically active 2,4,6-trisubstitutedpyridines

Table 1 Effect of catalyst loading for the synthesis of 2,4,6-triphenylpyridine

Entry	Catalyst (wt%)	Temperature (°C)	Time (min)	Yield[a] (%)
1	–	110	360	Traces
2	5	110	180	60
3	7.5	110	120	79
4	10	110	90	91
5	15	110	90	91
6	10	60	120	45
7	10	90	120	75
8	10	rt	360	NR[b]

[a]Isolated yield

[b]No reaction

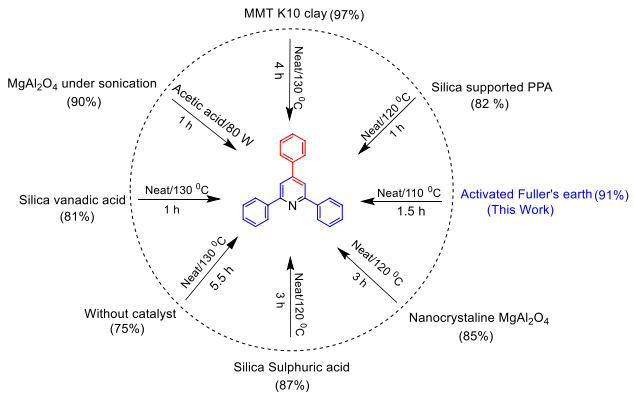


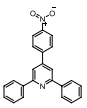
Fig. 2 Comparison of the efficiency of activated Fuller's earth with the literature reported catalysts for the synthesis of 2,4,6-triphenylpyridine

Table 2 Effect of the solvents for the synthesis of 2,4,6-triphenylpyridine

Entry	Catalyst (wt%)	Solvent	Temperature (°C)	Time (min)	Yield[a] (%)
1	10	Solvent free	110	90	91
2	10	H_2O	Reflux	240	22
3	10	EtOH	Reflux	240	54
4	10	MeCN	Reflux	240	30
5	10	DMF	110	240	68

[a]Isolated yield

Table 3 Synthesis of 2,4,6-triarylpyridine using a variety of aldehydes and acetophenones

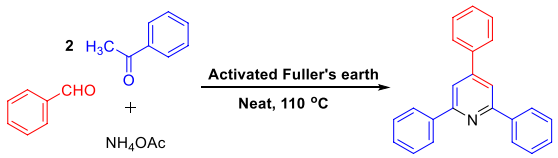
Entry	Aldehydes	Acetophenones	Products	Time (h)	Yield[a] (%)	Melting point (°C)	
						Obs.	Lit.
1.				1.5	91	135–136	136–137 [60]
2.				1.5	90	128–130	127–128 [60]
3.				1.5	89	135–137	136–138 [61]
4.				2.5	87	198–200	199–201 [62]
5.				2.5	87	101–102	100–101 [62]
6.				2.5	86	117–118	116–119 [61]
7.				2	86	105–107	106.3–107.5 [63]
8.				2.5	84	110–112	109–112 [61]
9.				3	84	122–124	123 [64]
10.				3	83	76–78	76 [65]
11.				2.5	86	160–162	162–164 [66]
12.				2	88	178–179	177–179 [60]
13.				2.5	85	155–157	156–158 [60]
14.				2	86	173–175	172–174 [60]
15.				2	89	266–267	268–270 [62]
16.				2	87	130–132	–
17.				2	84	178–180	179–181 [62]
18.				2	84	120–122	–
19.				2.5	85	124–126	–

[a]Isolated yield

Results and discussion

We structured our study to investigate the suitability of activated Fuller's earth as a catalyst for synthesis of 2,4,6-triarylpyridines, for this benzaldehyde (2 mmol), a representative substrate, acetophenone (4 mmol), and ammonium acetate (2.4 mmol) were used to afford 2,4,6-triphenylpyridine (Tables 1, 2).

Effect of the catalyst loading on time and yields of the reaction was assessed (Table 1, entries 2–5). The reaction does proceed at 110 °C with low product yield in the absence of a catalyst (Table 1, entry 1). An increase in the catalyst loading increased the product yield with a reduction in reaction time (Table 1, entries 2–5). The catalyst loading beyond 10 wt% was not advantageous (Table 1, entries 4 and 5), hence a 10 wt% catalyst loading was chosen for further study. Temperature played an important role in the synthesis of 2,4,6-triphenylpyridine (Table 1, entries 6 and 7). The temperature effect was examined at ambient, 60 and 90 °C under the solvent-free condition with activated Fuller's earth as a catalyst. The reaction does not proceed at room temperature. Further increasing temperature to 110 °C resulted in increased product yield in shorter reaction time (Table 1, entry 5). Therefore, this was the optimum temperature for performing the reaction (Scheme 1).

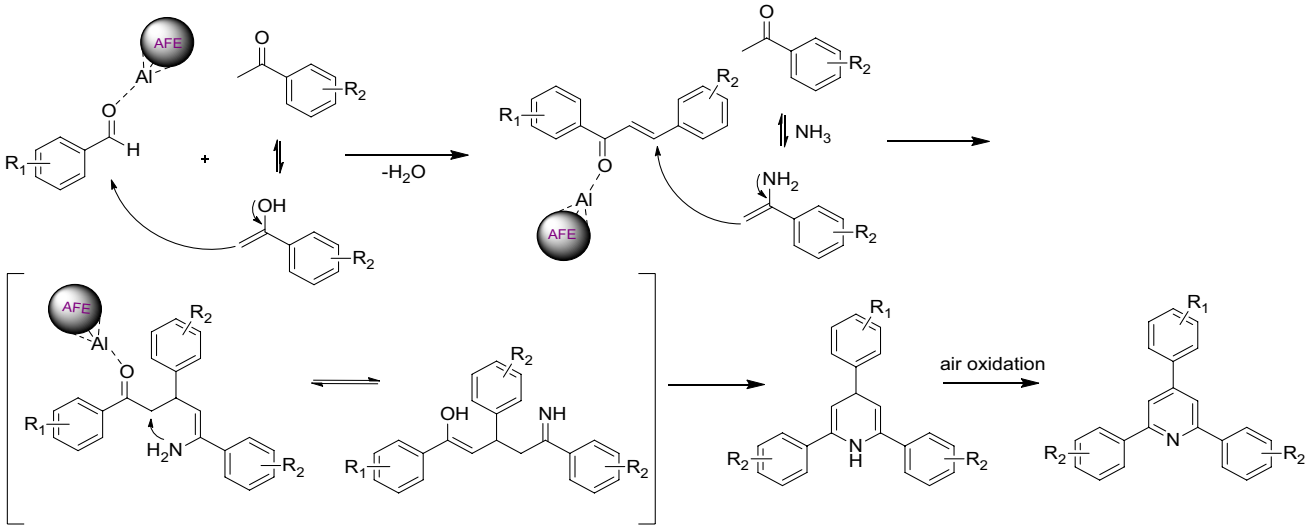


Scheme 1 Schematic representation of activated Fuller's earth catalyzed the synthesis of 2,4,6-triphenylpyridine

The effect of various solvents in model reaction on time and yield of the reaction was ascertained (Table 2, entries 2–5). None of the solvents presented the advantage of time and yield over solvent-free condition. Hence, the solvent-free condition was regarded as the best for the cost and environmental acceptability.

In comparison with the literature reported other catalysts such as PPA–SiO_2 [55], silica sulphuric acid [56], silica vanadic acid [57], mesoporous nanocrystalline $MgAl_2O_4$ [36], ultrasound-mediated nanocrystalline $MgAl_2O_4$ [58], and montmorillonite K10 Clay [59] used for the synthesis of 2,4,6-triphenylpyridine, activated Fuller's earth catalyst showed an advantage with respect to reaction condition, workup procedure, time, and yields (Fig. 2).

To investigate the substrate scope, optimized reaction conditions were applied to substituted aromatic/heterocyclic/α,β-unsaturated aldehydes, and acetophenones. All the substrate variants reacted well and afforded high yields of the corresponding 2,4,6-triarylpyridines within short reaction time (Table 3). Several electron-releasing or electron-withdrawing substituents at *ortho* and *para* positions of aromatic aldehydes have been examined. However, for 4-nitro, 4-methoxy, and 4-methyl substrates, the reaction time was longer with comparable product yield (Table 3, entries 4–6). This protocol was also applicable to cinnamaldehyde, phenylacetaldehyde, and thiophene 2-carboxaldehyde (Table 3, entries 9–11). On the other hand, the applicability of this protocol on substituted acetophenones was also examined (Table 3, entries 12–14). All the acetophenone variants reacted well and afforded good yields in shorter reaction time.



Scheme 2 Proposed mechanism for the synthesis of 2,4,6-triphenylpyridine

The scope of the present protocol has been explored for the synthesis of 2,4,6-triarylpyridine using substituted aldehyde and substituted acetophenones, wherein, electron-withdrawing substituents variants showed comparable yield and time to the substituted aldehyde or substituted acetophenones 2,4,6-triarylpyridine. (Table 3, entries 15–19).

The postulated mechanism for the synthesis of 2,4,6-triphenylpyridine is shown in Scheme 2; the reaction involves four steps aldol condensation, Michael addition, cyclisation, and finally air oxidation. Condensation of an aldehyde and acetophenone forms aldol product; on the other hand, a molecule of acetophenone with ammonia forms an enamine adduct. The addition of enamine to the aldol product followed by cyclisation gives dihydropyridine. Finally, air oxidation afforded the final product. Activated Fuller's earth aids aldol condensation, Michael addition, and precipitated oxidation of dihydropyridine to the final product. The reaction with electron-withdrawing groups of aromatic aldehydes is faster than the electron-donating one.

Conclusion

In conclusion, A mild, efficient, and ecological approach for the synthesis of 2,4,6-trisubstituted pyridines via the condensation of aromatic ketones with aromatic aldehydes and ammonium acetate in the presence of activated Fuller's earth clay as a recyclable heterogeneous solid acid catalyst has been developed. Due to the mild reaction conditions, good to excellent yields and easy workup procedure, the present protocol has edge over the other methods. Moreover, present method tolerates a wide variety of substituents. The trisubstituted pyridines were produced without formation of any other side product. Activated Fuller's earth is an inexpensive, eco-friendly, efficient heterogeneous catalyst. The catalyst can be easily prepared, used, recovered, and recycled with no loss of significant catalytic activity.

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