MCM-41 mesoporous silica: a highly efficient and recoverable catalyst for rapid synthesis of α-aminonitriles and imines

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ABSTRACT
In this work, pure MCM-41 mesoporous silica with active mesoporous sites has been successfully applied, as a highly efficient and recoverable catalyst, for the rapid and convenient synthesis of α-aminonitriles and imines. Various imines, as the intermediate of the Strecker reaction, were simply prepared from condensation of a wide range of aldehydes and amines in the presence of low loading of MCM-41 mesoporous silica under solvent-free conditions at room temperature in high to quantitative yields. Furthermore, the corresponding α-aminonitrile derivatives were prepared through the three-component Strecker reaction using trimethylsilylcyanide catalyzed by MCM-41 as a bifunctional heterogeneous mesoporous solid catalyst.

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Introduction

Bifunctional compounds such as α-aminonitriles are extremely useful synthetic intermediates. α-Aminonitriles serve as significantly valuable precursors for the synthesis of natural or synthetic α-amino acids, diamines and various nitrogen containing heterocycles such as thia diazoles, imidazoles and indoles (7–7). Indeed, these bifunctional compounds have received considerable attention due to their remarkable biological activities such as anticancer, antibacterial, antifungal, antibiotic and antiviral properties (5, 6, 8, 9).

The Strecker reaction which was first reported in 1850 (10) is the simplest and the most efficient method for the formation of carbon–carbon and carbon-heteroatom bonds required for the preparation of non-proteinogenic α-amino acids from corresponding α-aminonitriles in both lab and industrial scales. Furthermore, it has been known as the oldest reaction in the field of multicomponent reactions (MCRs) which is an attractive area of research in the modern organic chemistry (11, 12).

Some typical pharmacologically useful compounds which can be prepared through the Strecker reaction,
as one of the key steps, include saframycin A (9, 13–15), Phthalascidin (16–18), (R)-phenylglycinol derivatives (19), Ecteinascidin 743 (14, 20), (S)-Clopidogrel (Plavix®) (21, 22) and Captopril (Figure 1) (23, 24). Accordingly, different catalytic methods have been developed for synthesis of α-aminonitrile derivatives through the Strecker MCR strategy (25).

Several modifications of the traditionally Strecker reaction have been reported using a variety of alternative cyanide sources (10). Indeed, to overcome problems encountered with the use of NaCN, KCN and HCN, other cyanide sources such as trimethylsilylcyanide (TMSCN) (26), Bu2SnCN (27), Et2AlCN (28), Me2C(OH)CN (29), (EtO)2P(O)CN (30) and K4[Fe(CN)6] (31) have been examined. Among these alternative cyanide sources, TMSCN is a safer and more easily handled reagent compared to other cyanide sources (4). However, TMSCN requires a catalyst to promote the Strecker reaction. Therefore, different homogeneous and heterogeneous Lewis or Bronsted acids, metal complexes and biopolymers have been introduced as catalysts for the Strecker reaction. For example, K2PdCl4 or the NHC-amidate palladium(II) complex (32), NiCl2 (33), RhL3.H2O (34), InL3 (35), NbCl5 (36), BiCl3 (4), BiBr3 (37), Fe2O4 (38), Fe2O4@ZrO2/$SO4^2−$ (39), Fe3O4@SiO2@Me/Ph-HSO3H or Fe2O4@SiO2@PrNH2SO3H (19), Fe(Cp)2PF6 (40), Cu(OTf)2 (41), Yb(OTf)3 (42), Ga-TUD-1 (43), Ga(OTf)3 (44), GaCl3 (45), CeCl3 (46), Pr(OTf)3 (47), RuCl3 (48), La(NO3)3.6H2O (49), diethylphosphorocyanidated (30), I2 (50), FeCl3 (51), NH2SO3H (52), L-proline (53), p-toluenesulfonic acid (54), nano-sized TiO2 (55), O-benzenedisulfonimide (57), sulfuric acid-modified PEG-6000 (PEG-OSO3H) (58), xanthate sulfuric acid (59), cellulose sulfuric acid (60), Nafion-SAC-13 and Nafion-H (61), and chitosan (62) in conventional organic solvents have been developed in past two decades. Furthermore, a few heterogeneous mesoporous nano-ordered silica modified by transition metal cations or strong Bronsted acid centers such as Co/SBA-15 (63, 64), Zr-MCM-41 (65), B-MCM-41 (66), SBA-15-Ph-Pr-SO3H (67) and MCM-41-SO3H (68) have been introduced in recent years. However, most of these homogeneous or heterogeneous catalytic systems and modifications require the use of expensive reagents, harsh reaction conditions, lengthy reaction times, the use of toxic organic solvents, low yields as well as tedious work-up leading to the generation of a large amount of toxic waste (32, 34–37, 42, 43, 50, 61). Consequently, the use of cost-effective and recyclable catalysts as well as avoiding toxic or volatile organic solvents can be a major effort to improve clean and green synthesis of α-aminonitriles (62–64, 67).

In general, one of the fundamental aspects of development of new instrumental alternative processes is minimizing waste in chemical synthesis leading to environmental protection from pollution. For this purpose, attempts to find alternative and environmentally benign synthetic routes are one of the major aims in the protection of the environment and essential for the development of life and life processes (69, 70). The development of porous materials with large specific surface areas (>1000 m2.g−1) such as silica-based mesoporous has attracted significant attention with regard to their potential applications in the adsorption, chromatography, gas storage, sensor technology and recoverable solid catalysts in organic synthesis (71). In the case of solid-activated silica-based mesoporous catalysts, mesoporous materials were used as solid support in previous studies performed by our or other research groups (63–68). Hence, different methods including replacement of Si atoms in the matrix by metal ions such as Al (72), B or Fe (66), and Zr (65) or anchoring acidic organic groups such as 3-sulfonic propyl (67, 73) and inorganic sulfonic acid (−SO3H) (68) have been described. In continuation of our interest to develop more environmentally

Figure 1. Chemical structure of some pharmaceutically important compounds prepared by the Strecker reaction.
benign catalytic systems for different organic transformations (62, 66, 74, 75), we wish herein to report the pure MCM-41 (1a) as an efficient catalyst for rapid and convenient Strecker reaction of different aldehydes under solvent-free conditions to afford corresponding α-amino-nitriles in high to excellent yields (Scheme 1, A). Also, the corresponding imines, as the Strecker reaction intermediates, were easily prepared and isolated under the same reaction conditions in the absence of TMSCN (Scheme 1, B).

### Results and discussion

In order to optimize the reaction conditions, condensation of 4-chlorobenzaldehyde (2a, 1 mmol), aniline (3a, 1 mmol) and TMSCN (1.2 mmol), as the model reaction, was investigated in the presence of both silica-based mesoporous materials (MCM-41 and SBA) as well as commercial silica (SiO2) under solvent-free conditions at room temperature. The results are summarized in Table 1. It is noteworthy that only a trace amount of 2-(4-chlorophenyl)-2-(phenylamino)acetonitrile (4a) was

| Entry | Catalyst (1) | Loading (mg) | Time (min) | Yield (%)b |
|-------|--------------|--------------|------------|------------|
| 1     | –            | –            | 10         | Trace      |
| 2     | MCM-41 (1a)  | 10           | 10         | 97         |
| 3     | SBA-15 (1b)  | 10           | 10         | 89         |
| 4     | SiO2 (1c)    | 10           | 10         | 63         |
| 5     | MCM-41 (1a)  | 6            | 3          | 95         |
| 6     | MCM-41 (1a)  | 3            | 10         | 51         |

aReaction conditions: 4-chlorobenzaldehyde 2a (1 mmol), aniline 3a (1 mmol) and TMSCN (1.2 mmol) under solvent-free conditions at r.t.
bIsolated yield.

Scheme 2. Proposed mechanism for the Strecker reaction of aldehydes 2 and amines 3 with TMSCN or the formation of imines (5) catalyzed by pure MCM-41 (1a).
Table 2. Three-component Strecker reaction of various aldehydes 2 and amines 3 with TMSCN catalyzed by pure MCM-41 (1a) under optimal conditions. 

| Entry | Aldehyde 2 | Amine 3 | Product 4 | Time (min) | Yield (%) | Mp °C (Obsd/Lit) |
|-------|------------|---------|-----------|------------|-----------|-----------------|
| 1     | 4-(Cl)C₆H₄CHO (2a) | Aniline (3a) | ![4a](image) | 3 | 95 | 111–113/114–116 (68) |
| 2     | 2-(Cl)C₆H₄CHO (2b) | Aniline (3a) | ![4b](image) | 120 | 77 | 69–70/67–70 (39) |
| 3     | 4-(Br)C₆H₄CHO (2c) | Aniline (3a) | ![4c](image) | 30 | 95 | 90–92/87–87 (68) |
| 4     | 4-(CN)C₆H₄CHO (2d) | Aniline (3a) | ![4d](image) | 15 | 90 | 113–115/113–114 (46) |
| 5     | 4-(NO₂)C₆H₄CHO (2e) | Aniline (3a) | ![4e](image) | 20 | 94 | Oil/ Oil (66) |
| 6     | 3-(NO₂)C₆H₄CHO (2f) | Aniline (3a) | ![4f](image) | 25 | 90 | 95–97/89–92 (76) |
| 7     | PhCHO (2g) | Aniline (3a) | ![4g](image) | 5 | 95 | 78–79/80–82 (60) |
| 8     | 4-(CH₃)C₆H₄CHO (2h) | Aniline (3a) | ![4h](image) | 25 | 93 | 83–85/80–81 (40) |
| 9     | 4-(OH)C₆H₄CHO (2i) | Aniline (3a) | ![4i](image) | 25 | 91 | 124–126/120–122 (32) |
### Table 2. Continued.

| Entry | Aldehyde 2 | Amine 3 | Product 4 | Time (min) | Yield (%) | Mp °C (Obsd/Lit) |
|-------|------------|---------|-----------|------------|-----------|-----------------|
| 10    | 4-(CH₃O)C₆H₄CHO (2j) | Aniline (3a) | (4j) | 30 | 94 | 95–97/93–94 (40) |
| 11    | Vaniline (2k) | Aniline (3a) | (4k) | 60 | 89 | 113–115/106–108 (32) |
| 12    | PhCHCHCHO (2l) | Aniline (3a) | (4l) | 5 | 94 | 124–126/123–125 (40) |
| 13    | Furfural (2m) | Aniline (3a) | (4m) | 5 | 92 | 70–72/67–69 (68) |
| 14    | Thiophen-2-carbaldehyde (2n) | Aniline (3a) | (4n) | 10 | 90 | 97–100/98–100 (68) |
| 15    | 4-(Cl)C₆H₄CHO (2a) | p-Toluidine (3b) | (4a) | 25 | 94 | 83–85/83–85 (68) |
| 16    | PhCHO (2g) | p-Toluidine (3b) | (4p) | 5 | 95 | 105–107/104–107 (53) |
| 17    | 4-(CH₃)C₆H₄CHO (2h) | p-Toluidine (3b) | (4q) | 20 | 93 | 116–118/104–106 (62) |
| 18    | PhCHCHCHO (2l) | p-Toluidine (3b) | (4r) | 10 | 90 | 103–105/108–110 (78) |
| 19    | Furfural (2m) | p-Toluidine (3b) | (4s) | 20 | 87 | 74–76/67–68 (30) |
obtained in the absence of any catalyst. Interestingly, the yield of the desired product 4a was significantly improved in the presence of catalytic amount of MCM-41 (1a), SBA-15 (1b) and the commercial SiO2 (1c). Because of providing higher specific surface area by both MCM-41 (1a) and SBA-15 (1b), they provided a higher yield than the commercial SiO2 (1c) at same catalyst loading. On the other hand, MCM-41 (1a) afforded a higher yield compared to SBA-15. Therefore, the reaction conditions were optimized with regard to various MCM-41 (1a) loading in the next experiments (entries 5 and 6).

Encouraged by these results, the optimized conditions (MCM-41, 6 mg; solvent-free conditions; r.t.) were developed to different carbocyclic or heterocyclic aromatic aldehydes 2a–o and different amines 3a–c to afford desired α-aminonitriles 4a–x. The results are summarized in Table 2. The desired α-aminonitriles 4a–n were successfully prepared using aniline 3a with a variety of aldehydes (entries 1–14). The reaction also proceeded smoothly using other amines such as p-toluidine 3b and benzylamine 3c to afford the corresponding α-aminonitriles 4o–x in high to excellent yields (entries 15–24). In general, solid products are precipitated out from the initial liquid mixture containing the suspended MCM-41. On the other hand, the oily products afforded viscose mixtures.

After completion of the reaction, the catalyst 1a can be easily separated from the crude products by suspending the reaction mixture in hot EtOH and subsequent filtration. Indeed, aldehydes containing electron-withdrawing groups (2a,d–f) react faster than those bearing electron-donating groups (2h–k) depending on the used amine. Exceptions are 2-chlorobenzaldehyde (2b) and 4-bromobenzaldehyde (2c) which require longer reaction times (entries 2, 3). The long reaction times required for 2-chlorobenzaldehyde (2b) and 4-bromobenzaldehyde (2c) can be related to steric hindrance around the aldehyde functional group and lower electron-withdrawing of the bromo substituent compared to chloro at the para-position, respectively (62, 71). Furthermore, acid-sensitive substrates such as cinnamaldehyde (2l), furfural (2m), and thiophene-2-carbaldehyde (2n) reacted smoothly under optimized reaction conditions to provide the corresponding products 4l–4n or 4r–s in high to excellent yields without the formation of any polymerization products (entries 12–14, 18, 19) (49, 68).

To our delight, products such as 4l–v and 4x, which have low melting points and their physical state is generally oil at room temperature, were obtained at optimal reaction conditions in high to excellent yields and short to reasonable times (entries 12–22 and 24). Interestingly, these products and other oily products were
According to the observed results summarized in Tables 2 and 4, it can be deduced that the rate-determining step is addition of cyanide nucleophile on the activated imines (IV) where amine component 3 has been combined with aldehydes 2 before. Therefore, amines 3a-c with different nucleophilicities do not show significant effects on the reaction rate. On the other hand, it should be noted that the formation of imines (5) from aldehydes (2) and amines (3) and subsequent addition of cyanide anion of complexes IV or V to afford the corresponding α-aminonitriles (4) are faster than direct cyanation of carbonyl compounds. Therefore, MCM-41 (1a) can be considered as a simple and efficient bifunctional inorganic catalyst for the Strecker reaction.

The reusability of the separated MCM-41 (1a) was also investigated for the model reaction in further runs. The recovered catalyst after each run was washed three times with EtOAc and then dried at 50 °C for 1 h. Good conversions for the consecutive model reaction with a little loss of its activity was observed. The loss of reactivity of the catalyst (1a) can be attributed to the blocking of its channels by grafting of the trimethylsilyl group from the TMSCN reagent (see intermediate IV in Scheme 2, Figure 2).

The catalytic activity of the pure MCM-41 (1a) has also been compared with other previously reported heterogeneous catalysts to show the advantages of the present protocol. The results are summarized in Table 3. Comparison of data in Table 3 clearly demonstrates that a superior environmentally benign method in terms of the catalyst loading, avoiding the use of active Lewis or Bronsted acid reagents and toxic solvents, and required reaction time has been developed.

Furthermore, a plethora of catalytic systems have been introduced to promote imines (5) formation from corresponding carbonyl compounds (2) and primary amines (3) through a reversible condensation reaction. Imines (5) are also known as the Strecker reaction intermediates (58, 62, 68, 77). Therefore, we decided to investigate the imine formation from carbonyl compounds (2) and amines (3) in the presence of MCM-41 (1a) under similar conditions to the Strecker reaction. The results

| Entry | Catalyst | Solvent | Catalyst loading (mg) | Temp.(°C) | Time/min | Yield (%) | Ref. |
|-------|----------|---------|-----------------------|-----------|----------|-----------|------|
| 1     | MCM-41   | –       | 6                     | r.t       | 5        | 95        | This work |
| 2     | Chitosan  | –       | 6                     | r.t       | 10       | 95        | (62)  |
| 3     | SBA-Ph-Pr-SO₂H | – | 93                               | 50        | 15       | 100       | (67)  |
| 4     | Sn-Mont   | –       | 95                    | r.t       | 45       | 96        | (56)  |
| 5     | Nafion-SAC-13 | CH₂Cl₂ | 200                   | r.t       | 360      | 80        | (61)  |
| 6     | MCM-41-SO₂H | EtOH 96% | 5                     | r.t       | 70       | 97        | (68)  |
| 7     | PEG-OSO₂H | H₂O     | 180                   | r.t       | 10       | 91        | (58)  |
| 8     | Cellulose sulfuric acid | CH₂CN | 100                   | r.t       | 45       | 94        | (60)  |
| 9     | XSA       | CH₂Cl₂  | 100                   | r.t       | 65       | 97        | (59)  |
| 10    | PVP-SO₂   | CH₂Cl₂  | 100                   | r.t       | 360      | 86        | (77)  |
Table 4. Preparation of different imines 5 by the reaction of aldehydes 2 and amines 3 catalysed by MCM-41 (1a).a

| Entry | Aldehyde 2 | Amine 3 | Product 5 | Time (min) | Yield* (%) | Mp °C (Obsd/Lit) |
|-------|-----------|---------|-----------|------------|-------------|------------------|
| 1     | 4-(Cl)C6H4CHO (2a) | Aniline (3a) | (5a)      | 2          | 96          | 63–65/62–64 (62)  |
| 2     | 4-(NO2)C6H4CHO (2e) | Aniline (3a) | (5b)      | 3          | 91          | 87–89/86–87 (79)  |
| 3     | 3-(NO2)C6H4CHO (2f) | Aniline (3a) | (5c)      | 8          | 93          | 67–69/62–64 (39)  |
| 4     | PhCHO (2g) | Aniline (3a) | (5d)      | 5          | 92          | 52–54/52–54 (79)  |
| 5     | 4-(CH3)C6H4CHO (2h) | Aniline (3a) | (5e)      | 6          | 89          | 43–45/42–45 (39)  |
| 6     | 4-(OH)C6H4CHO (2i) | Aniline (3a) | (5f)      | 10         | 90          | 189–191/190–192 (62) |
| 7     | 2-(OH)C6H4CHO (2o) | Aniline (3a) | (5g)      | 8          | 91          | 54–56/52–54 (62)  |
| 8     | Vaniline (2k) | Aniline (3a) | (5h)      | 3          | 91          | 152–153/155–157 (80) |
| 9     | PhCHCHCHO (2l) | Aniline (3a) | (5i)      | 2          | 94          | 99–101/102–104 (53) |
| 10    | Furfural (2m) | Aniline (3a) | (5j)      | 5          | 95          | 58–60/59–60 (87)  |
| 11    | Thiophen-2-carbaldehyde (2n) | Aniline (3a) | (5k)      | 7          | 90          | Oil/ Oil (82)    |
| 12    | 4-(CH3)C6H4CHO (2h) | p-Toluidine (3b) | (5l)     | 1          | 90          | 83–84/84–86 (62)  |
| 13    | PhCHCHCHO (2l) | p-Toluidine (3b) | (5m)     | 1          | 93          | 78–79/76–77 (83)  |
| 14    | Thiophen-2-carbaldehyde (2n) | p-Toluidine (3b) | (5n)     | 8          | 90          | 59–61/62–64 (84)  |
| 15    | 3-(NO2)C6H4CHO (2f) | Benzylamine (3c) | (5o)     | 4          | 95          | 60–62/60–62 (62)  |
| 16    | Benzaldehyde (2g) | Benzylamine (3c) | (5p)     | 5          | 92          | 52–54/47–49 (76)  |
| 17    | 4-(OH)C6H4CHO (2i) | Benzylamine (3c) | (5q)     | 5          | 93          | 98–100/97–98 (85) |

*aReaction conditions: Aldehyde 2a (1 mmol), aniline 3a (1 mmol) and MCM-41 (1a, 6 mg) under solvent-free conditions at r.t.

bAll products are known compounds and their structures were established from their spectral data and melting points as compared with the authentic samples or literature values.

cIsolated yields.
of our experiments for this part of our studies about the synthesis of imines (5) are summarized in Table 4.

Conclusions

The three-component Strecker reaction catalyzed by the mesoporous MCM-41 without any modification using active Lewis or Bronsted acid reagents, as an efficient bifunctional inorganic catalyst, has been reported for the first time. The most important advantages of the present methodology in the Strecker reaction compared to other methodologies are low catalyst loading, excellent yields, short reaction times, avoiding the use of toxic solvents, clean reaction profile, simple work-up procedure and catalyst recyclability.

Experimental section

General

All chemicals and reagents were purchased from Merck or Aldrich and used without further purification except for benzaldehyde, for which a fresh distilled sample was used. MCM-41 was prepared according to the literature (68). Tetraethylorthosilicate (TEOS) and cetyltrimethylammonium bromide (CTAB) were used as the source of silicon and structure directing agent, respectively. Analytical TLC was carried out using Merck 0.2 mm silica gel 60 F-254 Al-plates. All yields refer to the isolated products after purification. FTIR spectra were recorded as KBr pellets on a Shimadzu FTIR-8400S spectrometer. $^1$H NMR (500 MHz) spectra were obtained using a Bruker DRX–500 AVANCE spectrometer in CDCl$_3$ at ambient temperature. Melting points were determined using an Electrothermal 9100 apparatus and are uncorrected. All products are known compounds and their structures were established from their spectral data and melting points as compared with the authentic samples or literature values (62, 66, 68).

General procedure for preparation of MCM-41 (1a)

A typical MCM-41 preparation is as follows: Diethylamine (2.7 g) was added to deionized water (42 mL) in a 200 mL beaker while the mixture was stirred at room temperature. After 10 min, CTAB (1.47 g) was added to the above solution under stirring step by step for 30 min until a clear solution was obtained. Then, TEOS (2.1 g) was added drop-wise to the solution. The pH of the reaction mixture was adjusted to 8.5 by the slow addition of hydrochloric acid solution (1 M). At this stage, the precipitate was formed. After 2 h, under slow stirring, the solid product was filtered from the mother liquor and washed with deionized water. The sample was dried at 45 °C for 12 h. The obtained white powder was calcined at 550 °C for 5 h to remove any remaining surfactant (68).

Typical procedure for the synthesis of α-amino nitriles 4a–x catalyzed by MCM-41 (1a)

A mixture of aldehyde (1 mmol), amine (1 mmol), and TMSCN (1.2 mmol, 0.15 mL) in the presence of the MCM-41 (6 mg) was stirred under solvent-free conditions at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the organic product was dissolved in hot EtOH (3 mL) and the mixture was simply filtered to remove the MCM-41, as a heterogeneous catalyst. The solid products were easily recrystallized from EtOH. In the case of oil products, EtOH was evaporated under reduced pressure to afford the essentially pure products (62, 66, 68).

Typical procedure for the synthesis of imine 5a–q catalyzed by MCM-41 (1a)

A mixture of aldehyde (1 mmol) and amine (1 mmol) in the presence of the MCM-41 (6 mg) was stirred under solvent-free conditions at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the organic product was dissolved in hot EtOH (3 mL) and the mixture was simply filtered to remove the MCM-41 as a heterogeneous catalyst. The solid products were easily recrystallized from EtOH. In the case of oil products, EtOH was evaporated under reduced pressure to afford the essentially pure products (62, 81–87).

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Disclosure statement

No potential conflict of interest was reported by the authors.

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