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Mont-K10 anchored Fe(II) Schiff-Base Complex: an efficient catalyst for Hydration of Nitriles to Amides

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Abstract:

Amides are one of the important functional groups in nature with wide range of pharmaceutical, biological and industrial applications. These compounds constitute the building blocks of peptides and polymer synthesis and can also be used as raw materials in detergents, lubricants, biologically active molecules, agrochemicals etc. Hence, chemical reactions involving the formation of amides are among the most important transformations in organic synthesis. Therefore, development of highly economic, efficient, cost effective, environment friendly and sustainable synthetic protocols for the formation of amides is of great concern. The objective of this work is to develop and investigate the catalytic activity of Fe(II) Schiff base complex anchored on mont-K10 (Fe@imine-mont-K10) for hydration of nitriles under ambient reaction conditions. The complex has been synthesized and characterized by FT-IR, powder X-ray diffraction, SEM-EDX and ICP-AES analysis. The catalytic activity of the complex was investigated for hydration of nitriles in *i*-PrOH under mild reaction conditions. The results indicated that the present protocol is quite effective towards hydration of aromatic nitriles and afforded moderate to excellent percentage conversion. The products were identified by FT-IR, GC-MS and compared with authentic samples.

Keywords: Schiff-Base, Fe@imine-mont-K10, Mont-K10, Hydration.

Introduction

Amides are one of the most important and prolific classes of organic compounds that have excellent biological and synthetic significance and play an essential and extensive role in many pharmaceutical and industrial applications. These are the key intermediates in organic synthesis and therefore have wide range of applications in organic chemistry. The amide group is also present in proteins and is also used as building blocks of peptide and polymer synthesis and therefore is very essential for sustaining life. It is known as synthetically versatile intermediate which is distributed in some specific biologically active molecules. Amide can be used as starting material for production of several detergents, fertilizer, lubricants and drug stabilizers. Because of the huge applications of amides such as in materials (e.g. Kevlar), biopolymers (e.g. proteins), and biologically active compounds, development of efficient and effective synthetic protocols for the synthesis of amides is one of the interesting topics of current research. In general, amides are synthesized by acid or base catalyzed hydration of nitriles. Among all the reported methods for the synthesis of amides, hydration of nitriles is one of the most widely used methods to obtain primary amides in industry [1-4]. Hydration of nitriles to amides is a simple, convenient, inexpensive and attractive transformation in synthetic organic chemistry. It is one of the most straightforward ways to synthesize amides by using corresponding organonitriles. Transition metal catalysed hydration of nitriles is an atom-economic and well-known sustainable method for the synthesis of versatile amides. Due to its operational simplicity, cost effectiveness and high atom efficiency, it has attracted the interests of the researchers. Conventionally, amides are produced by the hydration of nitriles with H_2SO_4 or NaOH, that causes the generation of plenty salts in the neutralization process, which is unfavourable and can cause over hydrolysis and formation of by-product in case of sensitive substrates along with the formation of carboxylic acids due to functional group incompatibility. Even the rate constant for amide hydration is usually larger than that of nitrile hydration, so it is difficult to stop the hydration at the amide stage. Generally, amides are synthesized by activating the carboxylic acid and then coupling with the amines. Although these methodologies have tremendous applicability in industry but they are still struggling due to their low efficiency and economically these techniques are not acceptable because large amount of waste is produced during the reactions. Previously, there was no concern about the solvent although as it has accounted the major part of any chemical reaction in pharmaceutical and chemical industry. The ultimate solution of the solvent issue is to replace the organic solvents with the water for its environmental impact, safety, availability and cost. Therefore, exploring metal ions based catalytic hydration of nitriles to the corresponding amides has attracted much attention, and various transition metal catalysed hydrations have been reported [5,6]. At present, several works have reported the use of metal nanoparticles for the hydration of nitriles. Despite all the efforts, most of the methods reported need high temperature, long reaction time, large excess of reagents or the use of expensive catalysts. Therefore, researchers have been trying to explore a new methodology to obtain amides from nitriles that require milder and environmentally benign synthetic conditions to reduce the wastes and environmental pollutions to achieve the green chemistry protocols. Scientist and researchers have developed the protocols for homogeneous catalysis which is frequently used in an industrial setting. Nevertheless, there are some disadvantages, such as a relatively short catalyst lifetime, a low tolerance for harsh conditions (high pressure and temperature), issues with catalyst-product separation and the cost of the catalyst itself. On the other hand, hydration with a heterogeneous catalyst is a valid alternative with the important advantage that the catalyst can be easily separated. The surface of heterogeneous materials can provide an ideal framework for the disposition of catalytically active functionality in order to achieve selectivity, especially with base metal oxides which can have both Lewis acidic and Brönsted basic sites. Heterogeneous catalysts have been successfully applied to the hydration of nitriles, although they are not widely used. With this concern, recently considerable efforts have been given to develop the new transition metal-catalysed hydration based on heterogeneous protocols. These protocols proved to be very efficient and have less environmental impact because isolation of the product and catalyst is very easy. The catalysts are also recyclable and reusable for many times without losing their reactivity significantly and they can be used for the hydration of the nitriles to produce amides at relatively lower temperatures as compared to previous systems. In that sense, the use of metal catalysts such as Ru [7-10], Pt [11,12], Pd [13], Os [14], Au [15], Ag [16], Mn [17], Cu [18], Ni [19] etc. have been crucial for the development of milder and more efficient transformations. Among the metal catalysts, Fe based catalysts have been received comparatively more attention for the hydration of nitriles to amides, since iron is readily available and low cost metal. Both homogeneous as well as heterogeneous Fe based catalysts for the synthesis of amides from nitriles have been reported [1]. Thus, in continuation of our previous work on catalytic activity of mont-K10 immobilized Fe (II) Schiff base complex [6] herein we wish to report the development of environmentally benign Fe (II) Schiff base complex immobilized on mont-K10 which catalyze the hydration of nitriles to amides effectively under mild reaction condition.

Experimental:

General Information: All chemicals were obtained commercially which were chemically pure and used as received without further drying or purification. Mont-K10 and APTES (3-Aminopropyl triethoxysilane) were purchased from Sigma-Aldrich, $FeSO_4.7H_2O$ was purchased from SRL (Sisco Research Laboratories Pvt. Ltd), *iso*-propanol, acetonitrile and potassium hydroxide were procured from MERCK and benzonitrile was obtained from Tokyo Chemical Industry. Acetone, toluene, dichloromethane, methanol and 2, 5-dimethoxybenzaldehyde were obtained from Rankem.

Methods: The characterization of Mont K-10 supported Fe (II) Schiff base complex has been carried out only by some limited spectroscopic techniques such as FT-IR, XRD, SEM-EDX and ICP-AES etc. FTIR spectra of APTES@mont-K10, imine@mont-K10 and Fe@imine-mont-K10 were recorded as KBr pellets on a Shimadzu IR prestige-21 FTIR spectrophotometer (500-4000 cm^{-1}). The SEM-EDX image of Fe@imine-mont-K10 was recorded using JEOL, JSM IT-300 operating at 20 kV. Powdered X-ray diffraction pattern of Fe@imine-mont-K10 was recorded using Rikagu Ultima IV diffractometer with Cu-Kα (1.541Å radiation). The Inductively Coupled Plasma Atomic Emission Spectrometric (ICP-AES) analysis of Fe@ imine-mont-K10 was done on ARCOS, Simultaneous ICP Spectrometer. GC-MS of the products were performed on Agilent Technologies GC system 7820 coupled with a mass detector 5975 and SHRXI-5MS column.

Syntheses: The mont-K10 supported APTES@mont-K10, imine@mont-K10 and Fe@iminemont-K10 were prepared following the reported procedure [6] which has been shown in Scheme.1.

a) Synthesis of APTES Functionalized Montmorillonite (APTES@mont-K10)

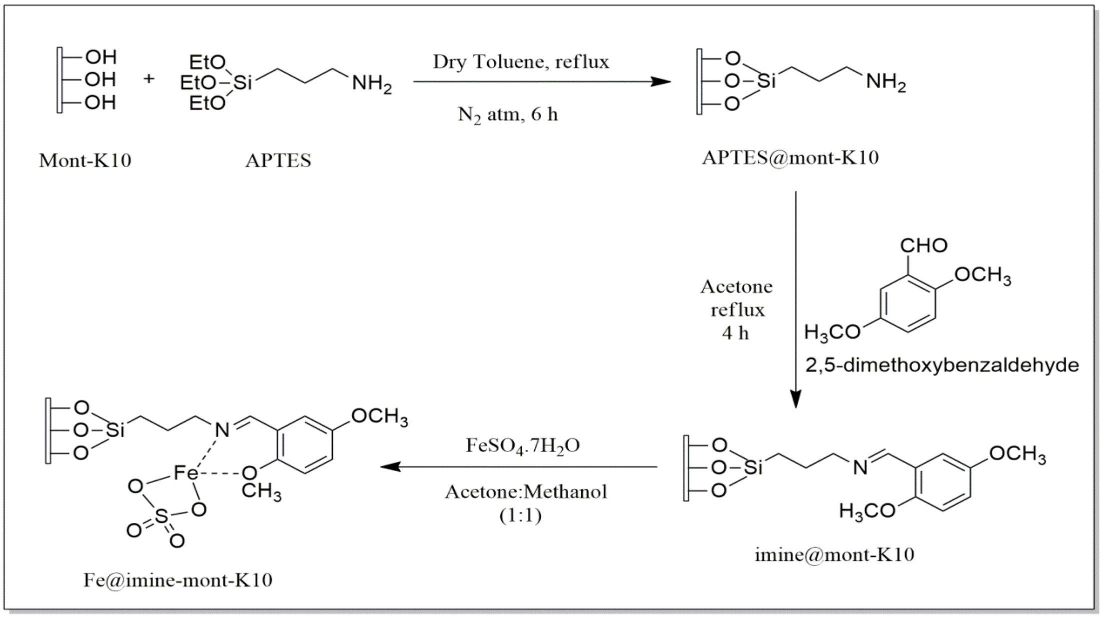
To a suspension of montmorillonite (500 mg) in dry toluene (100 mL), 0.11 g APTES (0.5 mmol) was added and then refluxed under nitrogen atmosphere for 6 h. The resulting mixture was allowed to cool, filtered and then washed with toluene repeatedly through Soxhlet extraction. The product obtained was dried (designated as APTES@mont-K10) and collected.

b) Synthesis of Imine Functionalized APTES@mont-K10 (imine@mont-K10)

To a suspension of APTES@mont-K10 (400 mg) in acetone (60 mL), 0.070 g of 2,5-dimethoxybenzaldehyde (0.17 mmol) was added, followed by refluxing for 4 h. The mixture obtained was filtered and then the residue was washed repeatedly with dichloromethane through Soxhlet extraction. It was dried (designated as imine@mont-K10) and then was kept in a desiccator.

c) Synthesis of Metal Functionalized imine@mont-K10 (Fe@imine-mont-K10)

To a solution of $FeSO_4.7H_2O$ (0.01725 g, 0.062055 mmol) in methanol and acetone (1:1), imine@ mont-K10 (100 mg) was added, stirred for 24 h at room temperature and then filtered. The residue was washed repeatedly with acetone through Soxhlet extraction and then allowed to dry (designated as Fe@ imine-mont-K10).



Scheme.1: Method of preparation of Fe@ imine-mont-K10

General Procedure for Hydration of Nitrile using Fe@imine-mont-K10 as the Catalyst:

The typical procedure for hydration of nitrile was carried out by taking a mixture of nitrile (1 mmol), catalyst (10 mg of Fe@imine-mont-K10, 1.877 mol %), KOH (1 mmol) and *i*-PrOH (10 ml) and stirring the mixture at room temperature for 4 h. The progress of the reaction was monitored continuously after regular intervals of time by thin layer chromatography (TLC) using 5% ethyl acetate-hexane as the eluent. As the reaction completes (monitored by GC-MS), stirring was stopped, the resulting mixture was centrifuged and the catalyst was separated by simple filtration. The residue was extracted from the filtrate using water-ethyl acetate mixture (1:1) followed by washing with brine and drying over anhydrous Na_2SO_4.

Results and Discussion

Characterization:

FT-IR study

The FT-IR spectrum of mont-K10 (Figure.1) shows peaks at 3343.75 cm^{-1} (broad) and 1640.53 cm^{-1} (sharp) which may be due to -OH stretching and bending, respectively. The sharp peak at 1100 cm^{-1} could be assigned to asymmetric stretching frequency of siloxane (Si-O-Si) framework. In the FT-IR spectrum of APTES@mont-K10 (Figure.2), a new band at 1540.23 cm^{-1} (γNH_2) is observed which indicates successful grafting of APTES onto the surface of mont-K10. The FTIR spectrum of imine@mont-K10 (Figure.3) shows the presence of a new band observed at 1646 cm^{-1} which may be attributed to νC=N stretching of imine group indicating the formation of Schiff base by condensation of amine group of APTES@mont-K10 and C=O group of 2,5-dimethoxybenzaldehyde. In the FT-IR spectrum of Fe@imine-mont-K10 (Figure.4), the presence of new bands at 470.65 cm^{-1} and 521.77 cm^{-1}, are assigned to bending vibrations of Fe–O and Fe–N respectively, which confirms that Fe is coordinated through oxygen and nitrogen respectively, indicating successful grafting of Fe(II) onto the surface of imine@mont-K10 through chemical bonding [6, 20-21] .

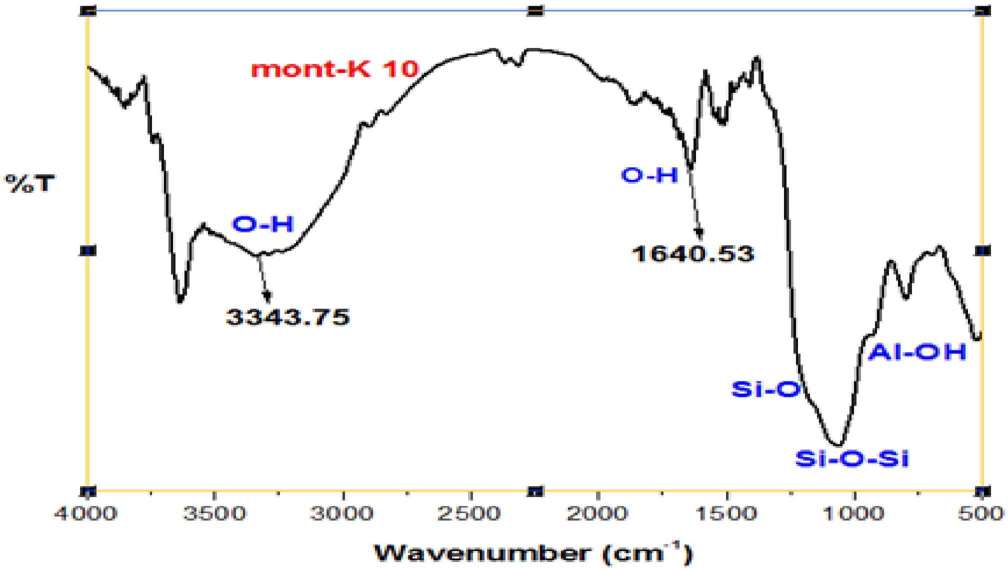
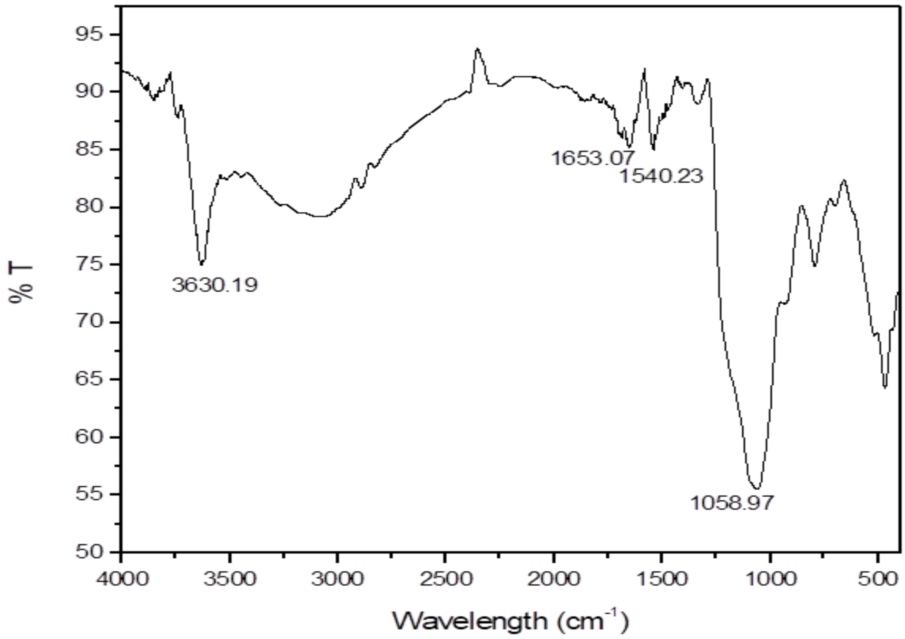


Figure.1: FT-IR spectrum of mont-K10



Date of submission 17-05-2026 Date of acceptance 02-06-2026

Figure.2: FT-IR spectrum of APTES@mont-K10

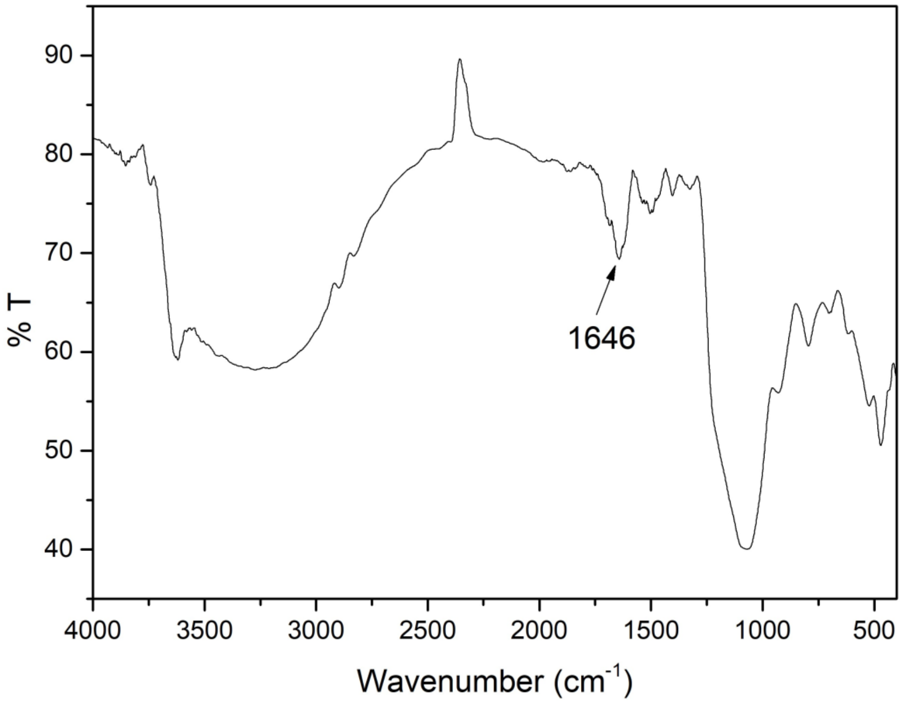


Figure.3: FT-IR spectrum of imine@mont-K10

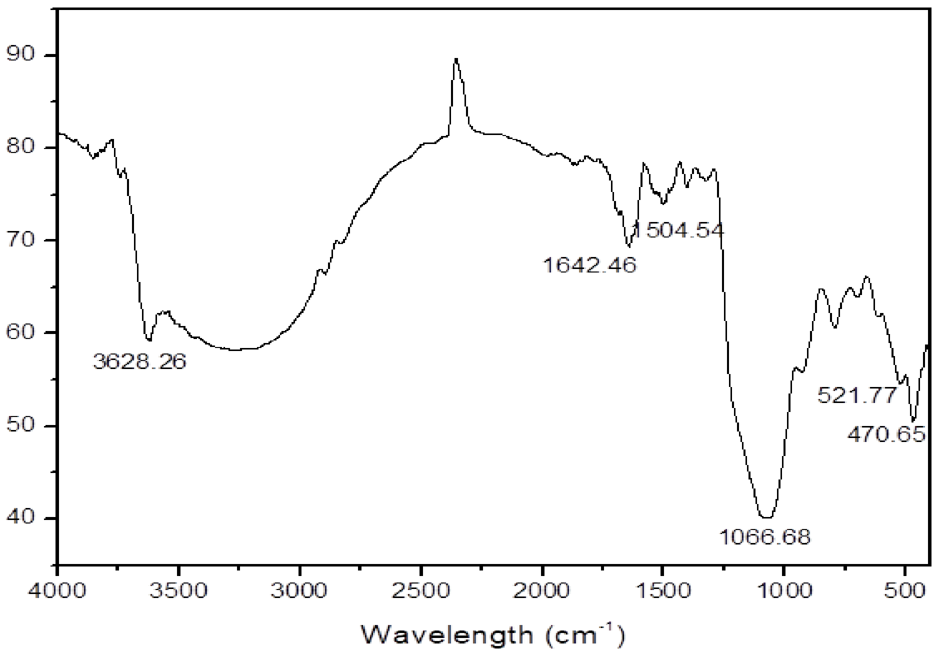


Figure.4: FT-IR spectrum of Fe@imine-mont-K10

XRD study

The powdered X-ray diffraction pattern in the range of 2θ = 5° to 80°of mont-K10 and Fe@ imine-mont-K10 have shown in Figure.5 and Figure.6, respectively. The difference in the peaks indicates successful grafting of Fe on the surface of mont-K10. The sharp peaks at 26.57°, 34.89° and 37.44° (Figure.6) corresponds to the {110},{130} and {111} crystal planes of Fe (+2) in Fe@imine-mont-K10.

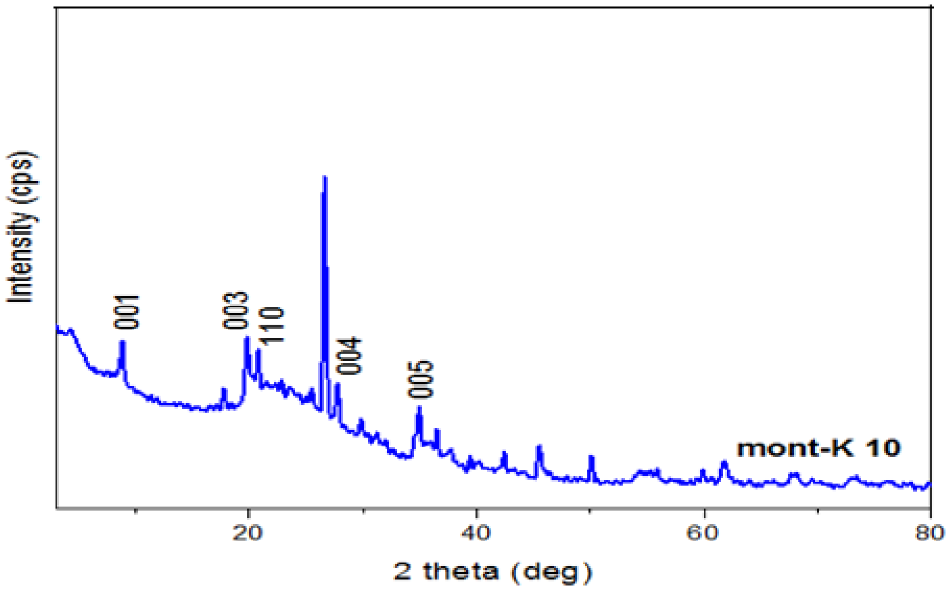


Figure.5: X-ray diffraction pattern of mont-K10

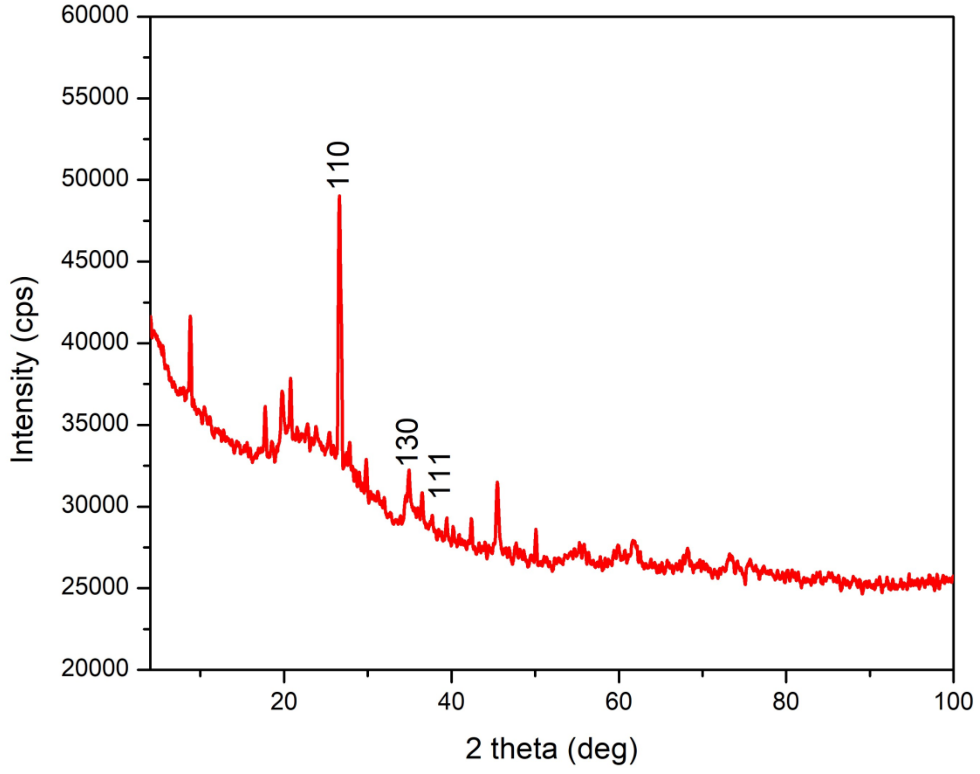


Figure.6: X-ray diffraction pattern of Fe@imine-mont-K10

SEM-EDX analysis

The SEM image of Fe@imine-mont-K10 has shown in Figure.7. In the EDX (Figure.8) of Fe@imine-mont-K10, the signals of Fe content along with C, O, N and Si proportions has been recorded suggesting that the metal complex have been successfully grafted with the anchored ligand on the surface of mont-K10.

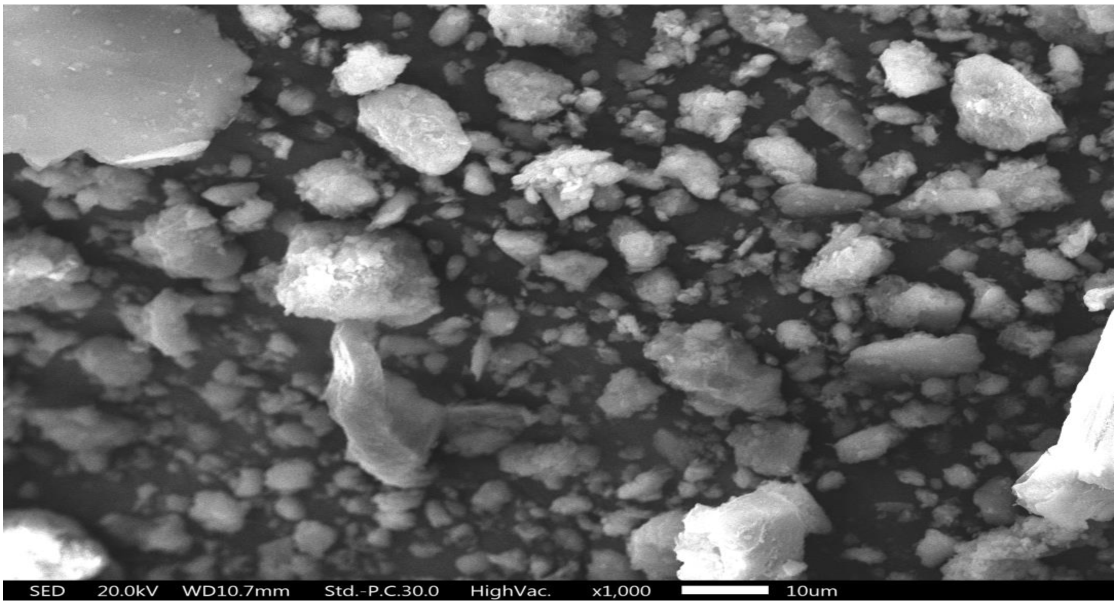


Figure.7: SEM image of Fe@imine-mont-K10

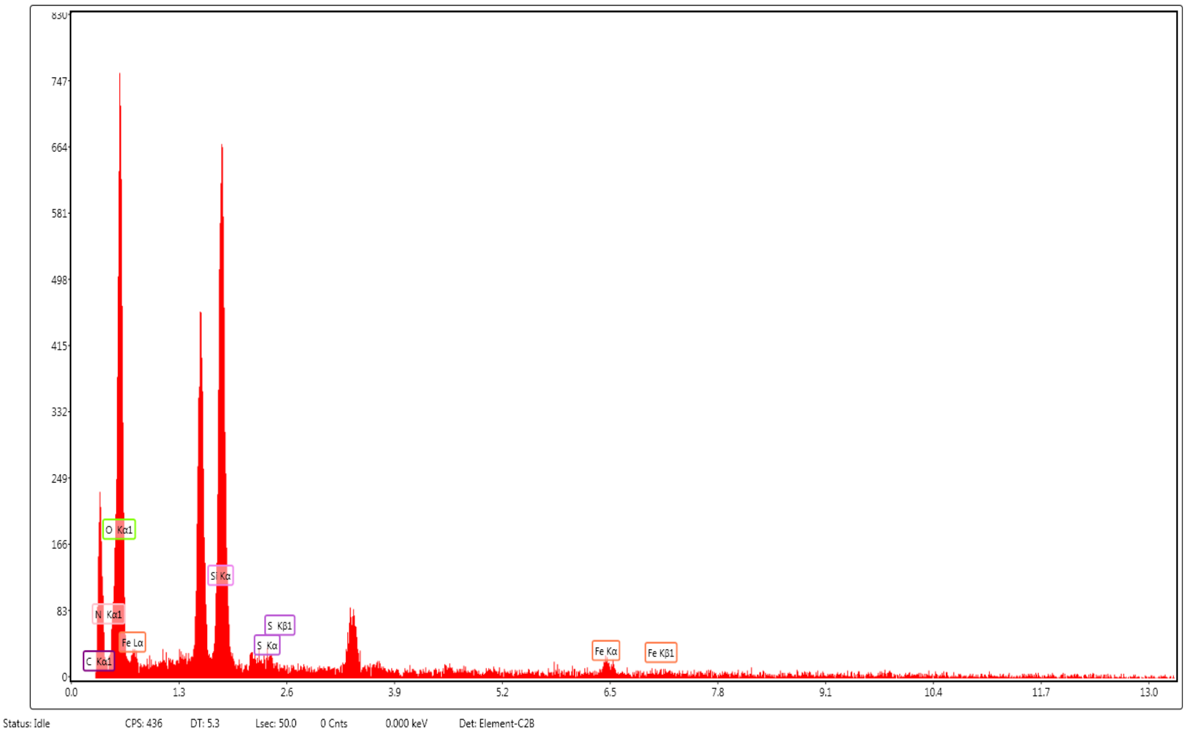


Figure.8: SEM-EDX of Fe@imine-mont-K10

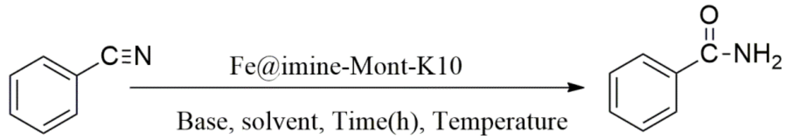
ICP-AES analysis

The ICP-AES analysis of Fe@imine-mont-K10 reports that it contains 1.877 mol % of Fe per 10 mg of the catalyst.

Hydration of Nitrile Compounds Catalyzed by Fe@imine-mont-K10

In order to study the catalytic activity and determine the suitable reaction condition for the hydration of nitriles, benzonitrile has been chosen as the model substrate to carry out the optimization studies (Table. 1). Initially, we have taken benzonitrile (1 mmol) with *i*-PrOH (10 ml) as the solvent and KOH (1 mmol) as the base and carried out the reaction in the absence of catalyst at room temperature for 24 h (Table. 1, Entry 1) which gives no product formation. In the next run, the catalyst (Fe@imine-mont-K10) has been introduced and the reaction was performed for 24 h in absence of any base (Table. 1, Entry 2) which also showed negative results. Our investigations showed that the presence of the catalyst as well as the base is essential in order to carry out this reaction. We examined the reaction at room temperature using various solvents such as *i*-PrOH, acetonitrile, ethanol, DMF and DMSO (Table. 1, Entry 1-8) and found out that *i*-PrOH gave the best results at room temperature (Table. 1, Entry 3 & 4). Moreover, being a green solvent *i*-PrOH has been chosen as the best solvent for the reaction.

Table. 1: Optimization of the reaction conditions for the hydration of benzonitrile using Fe@imine-mont-K10 as the catalyst[a]



Entry	Solvent	Base(mmol)	Catalyst(mg)	Time(h)	Isolated Yield (%)
1	*i*-PrOH	1	-	24	-
2	*i*-PrOH	-	10	24	-
3	*i*-PrOH	1	10	4	99
4	*i*-PrOH	1	20	3.5	99
5	CH_3CN	1	10	4	93
6	Ethanol	1	10	4	94
7	DMF	1	10	4	12
8	DMSO	1	10	4	-
9	*i*-PrOH	1	10	3	99[b]

[a]Reaction condition: benzonitrile (1 mmol), solvent (10 ml), base (KOH), catalyst (Fe@imine-mont-K10), r.t=22 °C,[b]at 40 °C

Now, since the presence of base is necessary for hydration of benzonitrile, we have performed the reaction using different bases such as KOH, NaOH, Na_2CO_3, $NaHCO_3$, K_2CO_3 and Et_3N (Table. 2) and examined. Upon investigation, we found out that KOH (1 mmol) exhibited the best performance at room temperature in 4 h (Table. 2, Entry 1).

Table. 2: Optimization of the base for the hydration benzonitrile[a]:

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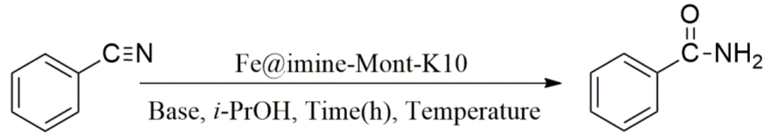
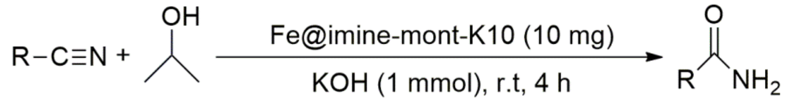
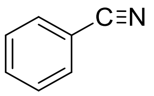
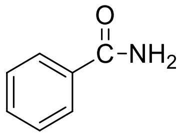
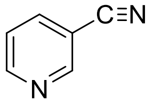
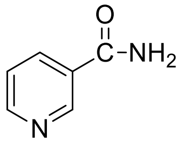
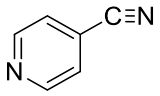
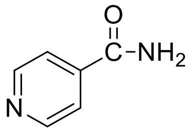
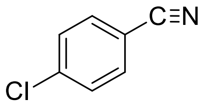
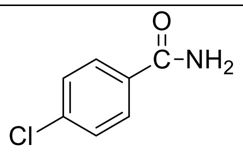
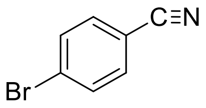
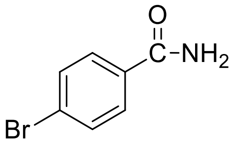
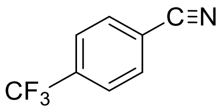
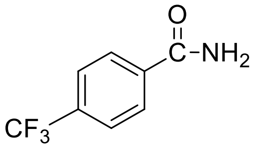
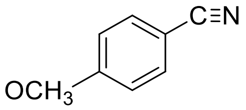
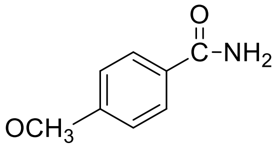
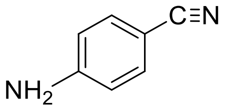
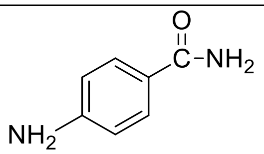
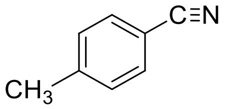
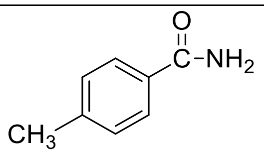
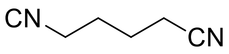
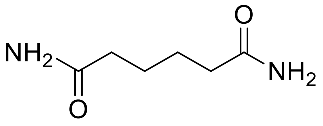
<tr><td colspan="4"> </td></tr>
<tr><th>Entry</th><th>Base(1 mmol)</th><th>Time(h)</th><th>Isolated Yield (%)</th></tr>
<tr><td>1</td><td>KOH</td><td>4</td><td>99</td></tr>
<tr><td>2</td><td>KOH</td><td>3.5</td><td>99[b]</td></tr>
<tr><td>3</td><td>NaOH</td><td>4</td><td>78</td></tr>
<tr><td>4</td><td>Na_2CO_3</td><td>4</td><td>-</td></tr>
<tr><td>5</td><td>$NaHCO_3$</td><td>4</td><td>-</td></tr>
<tr><td>6</td><td>K_2CO_3</td><td>4</td><td>-</td></tr>
<tr><td>7</td><td>Et_3N</td><td>4</td><td>-</td></tr>
<tr><td>8</td><td>KOH (2 mmol)</td><td>4</td><td>89</td></tr>
<tr><td colspan="4">[a]Reaction condition: benzonitrile (1 mmol), solvent(*i*-PrOH, 10ml), catalyst (Fe@imine-mont-K10, 10 mg), r.t=23, [b]at 40 °C</td></tr>
</table>

In order to determine the scope of this reaction, we have applied these optimized reaction conditions, i.e. the catalyst (Fe@imine-mont-K10, 10 mg), base (KOH, 1 mmol) and solvent (*i*-PrOH, 10 ml) at room temperature for hydration of various nitriles with aromatic and hetero-aromatic ring having electron withdrawing or electron donating group. The results are summarized in Table. 3. It has been observed that the hydration reaction in case of benzonitrile (Table. 3, Entry 1) and aromatic heterocyclic nitrile (Table. 3, Entry 2 & 3) produces excellent to moderate isolated yield. However, effective results have been observed in case of benzonitrile with electron withdrawing groups, such as Cl, Br, and CF_3 (Table. 3, Entry 4 - 6) giving higher yields of product formation, but no desired product fornmation has been observed for benzonitrile with electron donating groups, such as $-OCH_3$, $-NH_2$ and $-CH_3$ (Table. 3, Entry 7 - 9). The present protocol was not suitable to aliphatic nitriles (Table. 3, Entry 10). But to the best of our knowledge, the present protocol provided the best reaction condition system for hydration of nitriles to amides till date.

Table. 3: Hydration of various nitriles using *i*-PrOH, KOH and Fe@imine-mont-K10[a]



Entry	**Nitrile**	**Product**	**Isolated Yield (%)**
1			99
2			78
3			87
4			49
5			32
6			58
7			-
8			-
9			-
10			-

[a]Reaction conditions: Nitrile (1 mmol), *i*-PrOH (10 ml), KOH (1 mmol), Fe@imine-mont-K10 (10 mg), Time = 4 h.

Recycling:

Since, in case of heterogeneous catalysis reusability and recyclability are important parameters in determining its stability and efficiency. Hence, taking benzonitrile as the model substrate we have carried out the reaction following the optimized condition. As the reaction completes, the solid catalyst was easily retrieved from the reaction mixture by simple centrifugal precipitation, washed thoroughly with distilled water, dried and then used for the next run (Figure. 9). It has been observed that there is a slight decrease in the isolated yield of the product. It might be either because of some physical loss of catalyst during the separation from the reaction mixture or some decomposition of the real catalyst during the reaction.

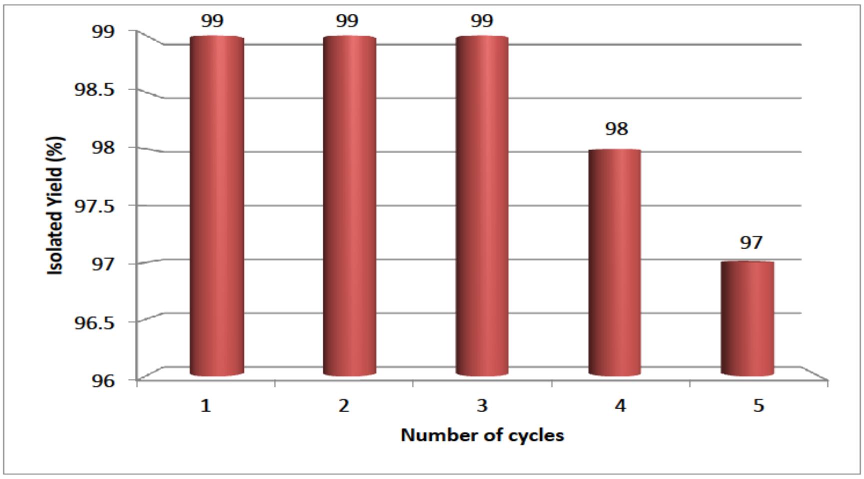
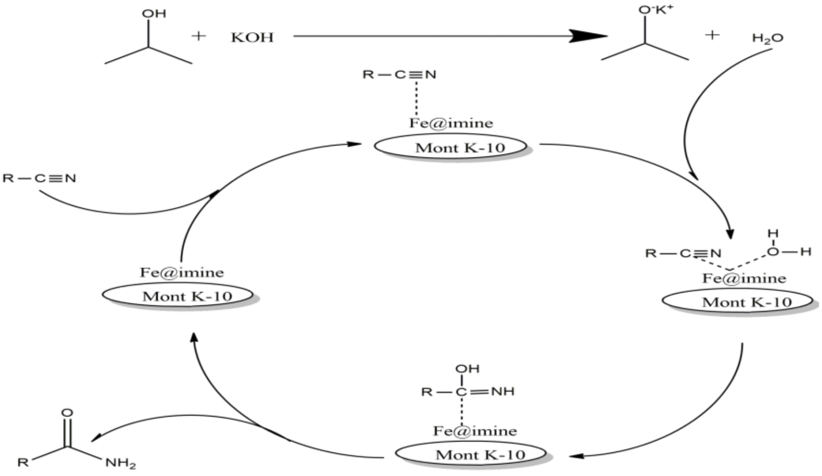


Figure. 9: Recycling of the catalyst

Probable Mechanism:

The plausible mechanism for hydration of nitriles to amides has shown in Scheme. 1.



Scheme. 1: Plausible mechanism for hydration of nitriles to amide

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Conclusion:

Herein, we report the synthesis of a new Fe(II) Schiff base complex by immobilizing $FeSO_4$ on the surface of imine-mont-K10. It was found as an efficient, reusable and heterogeneous catalyst for hydration of aromatic nitriles in *i*-PrOH at room temperature under mild reaction condition. This new protocol will offer new scope for the hydration of nitriles to amides that will provide wide applications in organic transformations and industrial purposes.

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