Jurnal Serambi Engineering JSE
Volume XI, No.2, April 2026 Hal 19411 - 19414
p-ISSN : 2528-3561
e-ISSN : 2541-1934

Synthesis and Characterization of Fe-Doped MCM-41 Using the One-Pot Method: XRD and FTIR Study

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Received: April 12, 2026 Approved: May 2, 2026

Abstract
MCM-41 is a mesoporous solid material developed by Mobil Corporation, composed of amorphous silica with a regular pore structure and uniform pore size arranged in a hexagonal pattern. This material has a large surface area (700–1000 m^2/g) and good thermal stability. MCM-41 is often used for the removal of heavy metals from aqueous media and as a heterogeneous catalyst to accelerate chemical reactions due to its flexible and modifiable nature. This study examines the effect of varying Fe metal doping on the one-pot synthesis of MCM-41 and its catalytic performance. MCM-41 was synthesized via a one-pot method using the following composition: 0.999 g CTAB, 4.666 g TEOS, 0.30 g NaOH, 1.245 g $FeSO_4$, and 1.211 g $FeCl_3$. Characterization of the synthesized material was performed using X-Ray Diffraction (XRD) for crystal structure analysis and Fourier Transform Infrared Spectroscopy (FTIR) for functional group identification; therefore, characterization of morphology and porosity is not discussed.
Keywords: *MCM-41, one-pot, fe metal, catalyst*

Abstrak
MCM-41 merupakan material padatan berpori mesopori yang ditemukan oleh Mobil Corporation, tersusun dari silika amorf dengan struktur pori teratur dan ukuran seragam yang membentuk susunan heksagonal. Material ini memiliki luas permukaan besar (700-1000 m^2/g) dan stabilitas termal yang baik. MCM-41 sering diaplikasikan pada penghilangan logam berat dari media air dan sebagai katalis heterogen untuk mempercepat reaksi kimia karena sifatnya yang fleksibel dan dapat dimodifikasi. Penelitian ini mengkaji pengaruh variasi doping logam Fe pada sintesis one pot MCM-41 serta performa katalitiknya. MCM-41 disintesis dengan metode one pot menggunakan komposisi CTAB 0,999 gram, TEOS 4,666 gram, NaOH 0,30 gram, $FeSO_4$ 1,245 gram, dan $FeCl_3$ 1,211 gram. Karakterisasi material hasil sintesis dilakukan menggunakan X-Ray Diffraction (XRD) untuk analisis struktur kristal dan Fourier Transform Infrared Spectroscopy (FTIR) untuk identifikasi gugus fungsi, sehingga karakterisasi morfologi dan porositas tidak dibahas.
Kata Kunci: *MCM-41, one-pot, logam fe, katalis*

1. Introduction

Mesoporous silica materials have attracted significant attention in material science due to their unique structural characteristics such as high surface area, uniform pore size distribution, and good thermal stability [1][2]. One of the most widely studied mesoporous materials is MCM-41 (Mobil Composition of Matter No. 41), which possesses a highly ordered hexagonal pore structure[3]. These properties make MCM-41 suitable for various applications including catalysis, adsorption, and drug delivery systems[4].

However, pure MCM-41 consists mainly of amorphous silica, which exhibits weak Lewis acidity and lacks Brønsted acid sites. As a result, its direct application as a catalyst is limited. To enhance its catalytic performance, the incorporation of metal species into the silica framework is often required. Transition metals such as iron (Fe) are commonly used as dopants because they can introduce active catalytic sites and improve redox properties[5] [6].

Conventional methods for introducing metal species into MCM-41 generally involve post-synthesis impregnation. Although effective, this approach requires multiple steps and may reduce the structural order of the mesoporous framework. An alternative approach is the one-pot synthesis method, in which the formation of the silica framework and metal incorporation occur simultaneously in a single reaction system. This method simplifies the synthesis process, reduces energy consumption, and improves the dispersion of metal species within the silica matrix. To evaluate the success of the synthesis process and metal incorporation, structural characterization techniques are required. X-ray diffraction (XRD) is commonly used to analyze the crystallinity and structural order of mesoporous materials, while Fourier Transform

Infrared Spectroscopy (FTIR) is used to identify functional groups and investigate chemical interactions within the silica framework. Therefore, this study aims to synthesize Fe-doped MCM-41 using the one-pot method and evaluate its structural properties using XRD and FTIR characterization techniques [7].

2. Material and Methods

2.1. Materials

The materials used in this study include tetraethyl orthosilicate (TEOS) as the silica precursor, cetyltrimethylammonium bromide (CTAB) as the structure-directing agent, sodium hydroxide (NaOH) as the base catalyst, iron chloride ($FeCl_3$) and iron sulphate ($FeSO_4$) as iron precursors, and distilled water as the solvent.

2.2. Synthesis of Fe-Doped MCM-41

Fe-doped MCM-41 was synthesized using the one-pot synthesis method. First, 0.999 g of CTAB was dissolved in 480 mL of 15 mM NaOH solution under stirring. Subsequently, 4.666 g of TEOS was added dropwise into the solution while maintaining continuous stirring[8].

After homogenization, the iron precursor ($FeCl_3$ or $FeSO_4$) was added to the mixture. The reaction mixture was stirred at 80 °C for 2 hours to promote the formation of the mesoporous structure and incorporation of iron into the silica framework.

The resulting solid product was separated by filtration, washed with distilled water and methanol, and dried in an oven at 105 °C for 1 hour. Finally, the dried material was calcined in a furnace at 600 °C for 6 hours to remove the surfactant template and obtain the final Fe-doped MCM-41 catalyst[4].

2.3 Characterization Techniques

FTIR spectroscopy was used to identify functional groups and chemical interactions within the synthesized material. The measurements were conducted using an FTIR spectrometer equipped with an ATR accessory with a resolution of 4 cm^{-1} and 16 scan accumulations [9]. XRD analysis was performed to investigate the structural characteristics and crystallinity of the synthesized materials. The diffraction patterns were analyzed to determine the presence of crystalline phases and structural changes caused by Fe doping[10][11][12].

3. Result and Discussion

3.2. FTIR Analysis

FTIR analysis was performed to identify the functional groups present in MCM-41 and to evaluate the chemical interaction between Fe ions and the silica framework after doping. The FTIR spectrum of pure MCM-41 shows characteristic absorption bands associated with the silica network. The strong band around 1080 cm^{-1} corresponds to the asymmetric stretching vibration of Si–O–Si bonds, while the bands near 800 cm^{-1} and 460 cm^{-1} are attributed to symmetric stretching and bending vibrations of Si–O bonds. In addition, a broad band around 3400 cm^{-1} is associated with O–H stretching vibrations from adsorbed water, and a band near 1630 cm^{-1} corresponds to H–O–H bending vibrations [13].

After doping with $FeCl_3$, noticeable changes in the FTIR spectrum were observed, particularly in the region below 1000 cm^{-1}. These changes indicate interactions between Fe^{3+} ions and the silica framework. The increase in absorption intensity around 500–600 cm^{-1} suggests the formation of Fe–O bonds or Fe–O–Si linkages, which indicates successful incorporation of iron into the silica structure.

Similarly, $FeSO_4$-doped MCM-41 also shows spectral changes, although slight differences in peak intensity and position were observed compared to $FeCl_3$ doping. These differences may be attributed to the different oxidation states of iron, where $FeSO_4$ provides Fe^{2+} ions while $FeCl_3$ provides Fe^{3+} ions. Overall, the FTIR results confirm the successful modification of the silica framework by iron incorporation.

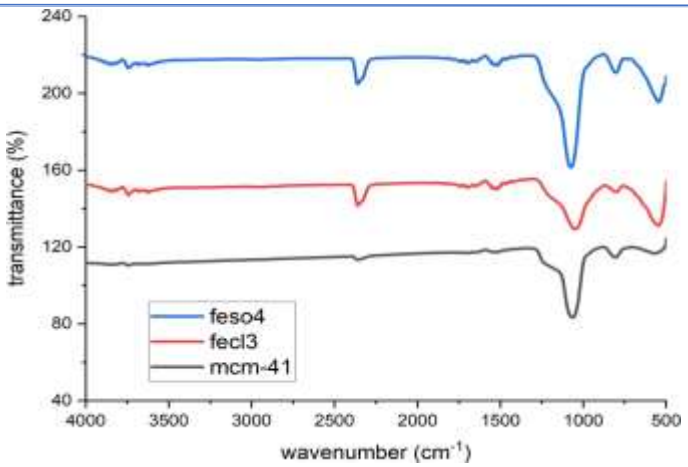

Fig 1: FTIR Characterization Results

3.3. XRD Analysis

XRD analysis was conducted to examine the structural properties and crystallinity of the synthesized materials. The XRD pattern of pure MCM-41 shows a broad diffraction peak in the low-angle region (approximately 2θ = 20–30°), indicating the amorphous nature of silica while maintaining mesoporous structural ordering.

For $FeSO_4$-doped MCM-41, several sharp peaks appear in the range of 2θ = 30–55°. These peaks suggest the formation of crystalline iron oxide phases such as Fe_2O_3 or Fe_3O_4 within or on the surface of the mesoporous structure. This indicates that Fe species derived from $FeSO_4$ partially crystallize rather than being fully dispersed in the silica framework[14][15].

In contrast, the XRD pattern of $FeCl_3$-doped MCM-41 exhibits broader peaks with fewer sharp reflections. This suggests that Fe species derived from $FeCl_3$ are more uniformly dispersed within the silica matrix and do not form large crystalline phases. Therefore, $FeCl_3$ tends to produce a more homogeneous doping within the MCM-41 structure compared to $FeSO_4$.These results demonstrate that the choice of iron precursor significantly influences the structural characteristics of the synthesized Fe-MCM-41 catalyst.

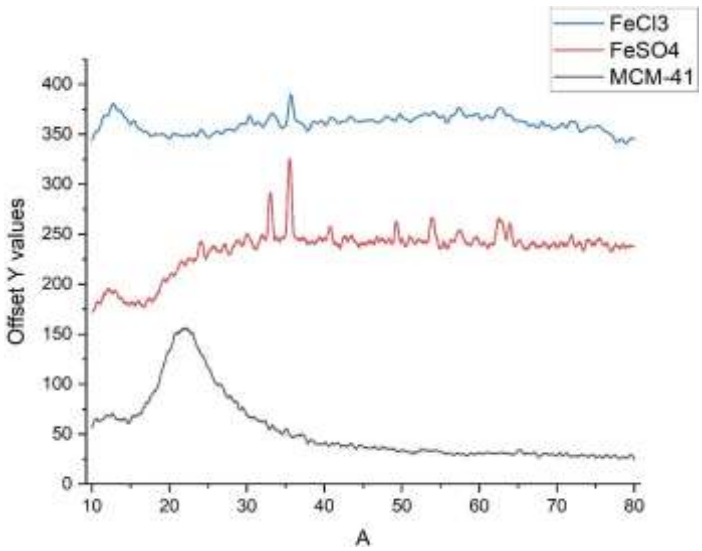

Fig. 2: XRD Characterization Results

4. Conclusion

In this study, Fe-doped MCM-41 was successfully synthesized using the one-pot synthesis method and characterized using FTIR and XRD techniques. FTIR analysis confirmed the presence of characteristic silica vibrations and indicated successful incorporation of iron species into the silica framework through the formation of Fe–O and Si–O–Fe bonds. XRD analysis revealed that pure MCM-41 exhibits an amorphous silica structure, while $FeSO_4$ doping leads to the formation of crystalline iron oxide phases. In contrast, $FeCl_3$ doping results in a more homogeneous dispersion of iron species within the mesoporous structure. Overall, the results indicate that $FeCl_3$ is more effective in maintaining the structural stability of MCM-41, while $FeSO_4$ tends to form additional crystalline phases.

5. Acknowledgment

The author would like to express sincere gratitude to the Laboratory of Basic Chemistry at Universitas Singaperbangsa Karawang for providing research facilities. The author also thanks the supervisor and laboratory staff for their guidance and support during the research process.

6. Abbreviations

MCM-41	Mobil Composition of Matter No. 41
CTAB	Cetyltrimethylammonium Bromide
TEOS	Tetraethyl Orthosilicate
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-Ray Diffraction
$FeCL^3$	Iron (III) Chloride
$FeSO^4$	Iron (II) Sulfate

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