



Synthesis and Structural Evaluation of Fe(III) Metal–Organic Frameworks formed from Terephthalic and Fumaric acids

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Abstract

Iron-based metal–organic frameworks (Fe-MOFs) were synthesized at room temperature using Fe³⁺ ions with diprotic carboxylate linkers; terephthalic acid (H₂BDC) and fumaric acid (H₂FUM). The synthesized MOFs were characterized by Thermogravimetric (TGA), Brunauer-Emmett- Teller (BET), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX). Fe-BDC achieved the highest yield (68.97%) and Fe-FUM (48.14%). The solubility and TGA analysis revealed that Fe-BDC framework demonstrated superior hydrolytic and thermal stability at 500°C compared to Fe-FUM MOF which showed partial hydrolysis and thermal stability at 450°C. FTIR spectra confirmed coordination between Fe ions and carboxylate groups, with characteristic bands at 1596–1577 cm⁻¹ (νC=O), 1394–1396 cm⁻¹ (νC–O), and 504–557 cm⁻¹ (νFe–O). XRD patterns displayed broad, low-intensity peaks at 2θ = 12.17°, 26.64°, 30.64°, and 33.56°, indicative of amorphous structures typical of room-temperature syntheses. SEM images revealed irregular and aggregated particle morphologies, while EDX confirmed the presence of Fe, C, and O, validating successful framework formation, forming frameworks with distinct elemental ratios influenced by linker type. Fe-BDC exhibited metal-rich composition, while Fe-FUM was more organic-rich.

Keywords: Synthesis, Metal-organic frameworks, Terephthalic acid, Fumaric acid, Fe-MOFs

Received: 27th February, 2026 Accepted: 12th May, 2026 Published Online: 24nd June 2026

Introduction

Porosity refers to the ability of a solid material to accommodate fluids within its internal voids. A material is considered porous when the cavities in its structure are sufficiently large to host guest molecules (Yilmaz *et al.*, 2012). Metal–organic frameworks (MOFs) represent a prominent class of crystalline porous materials composed of inorganic metal ions or clusters coordinated to polydentate organic ligands, commonly referred to as linkers. The metal centers act as connectors that bridge the organic linkers, generating extended three-dimensional structures with tunable pore structures. These linkages are mediated through metal–oxygen–carbon

bonds, forming secondary building units (SBUs) that define the framework’s geometry and topological characteristics (Rowsell and Yaghi, 2004). The intrinsic structural versatility of SBUs enables the rational design of MOFs with tailored porosity, surface functionality, and physicochemical properties (Yaghi *et al.*, 2003). Among the diverse dicarboxylic acid-based linkers explored, terephthalic acid (1,4-benzenedicarboxylic acid, H₂BDC) and fumaric acid (trans-butenedioic acid, H₂FUM) stand out as prototypical ligands that define structural diversity and functional tunability in MOFs (Furukawa *et al.*, 2013; Bara *et al.*, 2021). The aromatic and rigid nature of BDC favors the

formation of large-pore, highly ordered frameworks such as MOF-5 and MIL-53, whereas the aliphatic, unsaturated fumarate linker yields more compact and hydrophilic frameworks exemplified by MOF-801 (Ke *et al.*, 2018).

Metal ions from the first-row transition series, including Cr, Fe, Co, and Zn, are commonly utilized as coordination nodes in the assembly of metal–organic frameworks (MOFs) (Wu *et al.*, 2009; Ma, 2011). Among these transition metals, iron (^{56}Fe , group 8) stands out for its versatility, attributable to the easy interconversion between its Fe^{2+} and Fe^{3+} oxidation states. This redox flexibility promotes diverse coordination environments and framework topologies. Iron-based metal–organic frameworks (Fe-MOFs) represent a versatile subclass of coordination polymers in which $\text{Fe}^{2+}/\text{Fe}^{3+}$ centers are interconnected with organic linkers to generate highly ordered porous architectures. Owing to their large surface area, tunable chemical functionality, intrinsic redox activity, and environmental benignity, Fe-MOFs have attracted considerable scientific interest (Zhang *et al.*, 2021; Horcajada *et al.*, 2007). Prominent examples, including the MIL series (e.g., MIL-53, MIL-88, MIL-100, and MIL-101), display framework flexibility that enables reversible structural transformations in response to guest molecules, thereby enhancing their utility in adsorption, catalysis, and environmental remediation processes (Channab *et al.*, 2023).

The solvothermal method remains the most prevalent method for synthesizing MOFs, including Fe-MOFs; however, it typically necessitates elevated temperatures and pressures, extensive solvent consumption, and prolonged reaction durations, posing limitations for scalable production (Dhakshinamoorthy *et al.*, 2020; Taddei, 2019). Consequently, recent research has emphasized the development of sustainable and energy-efficient synthesis routes that operate under ambient conditions, employ benign solvents, minimize reaction times, and eliminate hazardous by-products or corrosive reagents, while reducing or avoiding laborious post-synthetic purification and activation

steps (Zhu *et al.*, 2023; Ke *et al.*, 2018). Room temperature MOFs synthesis provides several benefits, including less energy use, better repeatability, and easier synthesis processes (Dai *et al.*, 2021). For these reasons, Room temperature synthesis for Fe-MOFs was adopted.

Materials and Method

Chemicals and reagents

All materials were Analytical grade. $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, *N,N* Dimethylformamide (DMF), benzenedicarboxylic acid (H_2BDC), Fumaric acid ($\text{C}_4\text{H}_4\text{O}_4$) chloroform (CHCl_3), Methanol (MeOH , 99.9%) and ethanol (EtOH , 98%), Distilled water, Conc. HCl, Sodium hydroxide (NaOH)

Apparatus/Equipment

Beaker, Test tube, Spatula, Measuring Cylinder, Volumetric Flask, Conical Flask, Wash bottle, Funnel, Filter paper, Weighing Balance, Petri-dish, Water Bath. Stirring rod, Plastic dropper, Magnetic stirrer bar, Magnetic hot plate stirrer, oven, vacuum oven. The synthesized MOFs were analyzed for phase identification and crystallinity by FTIR, X-ray powder diffraction (XRD) and scan electron microscope (SEM).

Synthesis of Fe-Based Metal Organic Frameworks

The synthesis of Fe-MOFS was carried out according to the procedure reported by Rather *et al.* (2018), Kiros *et al.* (2017) and Sushil *et al.* (2022) with some modifications

Synthesis of Fe-FUM MOF

Iron(III) nitrate nanohydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 10.0 mmol; 4.04 g) was dissolved in 25 mL of ethanol, while fumaric acid (0.58 g) was separately dissolved in 10 mL of *N,N*-dimethylformamide (DMF). The iron salt solution was added dropwise to the linker solution under constant stirring and maintained for 20 h at room temperature, resulting in the formation of a brown precipitate. The solid product was separated by centrifugation and washed several times with DMF to remove unreacted precursors. To minimize residual DMF within the pores, the sample was further washed repeatedly with chloroform and then dried in an oven at 80 °C until a constant weight was obtained.

Synthesis of [Fe₂(BDC)]. H₂O

The Fe-BDC MOF was synthesized following a modified solvothermal approach. Terephthalic acid (H₂BDC; 1.26 g, 7.6 mmol) was dissolved in 23.72 g of DMF (solution 1). Separately, Fe(NO₃)₃·6H₂O (5.66 g, 14.0 mmol) was dissolved in 20 mL of ethanol (solution 2). Both solutions were stirred until completely clear, after which solution 1 was added dropwise to solution 2 under continuous stirring. The reaction mixture, having a Fe:H₂BDC:EtOH molar ratio of approximately 2:1:excess, was stirred at room temperature for 24 h to facilitate coordination and framework formation. The resulting solid was recovered by centrifugation at 4000 rpm, washed thoroughly with DMF to remove unreacted species, and soaked in chloroform to displace residual DMF. The product was then oven-dried to yield a brown amorphous Fe-BDC solid.

Characterizations of the Synthesized MOFs

The synthesized MOFs were comprehensively characterized to elucidate their physicochemical properties, bonding interactions, crystallinity, and surface morphology using solubility, TGA, BET, FTIR, XRD, and SEM-EDX analysis.

Infrared spectra Characterization

Fourier transform infrared spectroscopy (FT-IR) was used to identify the presence of some certain functional groups in the synthesized compounds and also confirm the identity of the compound through the collection of absorption frequencies. A Perkin Elmer 550s Fourier Transform Infrared Spectrometer, produced by Parker Company in the United States, was used. The composite material and pure potassium bromide were mixed and ground evenly in a ratio of 1:150. The mixture was then pressed into a card slot for testing. The test was conducted using pure potassium bromide tablets as the background. It involved 64 scans and covered a wavenumber range of 4000 cm⁻¹ to 500 cm⁻¹.

X-ray diffraction Characterization

The XD-3 powder X-ray diffractometer was used to test the crystalline structure of the MOFs material. The sample is placed on a glass slide with a specific groove to flatten its surface. The slit attachment is selected for

scanning testing, and the X-ray excitation source is Copper metal (Cu) target with K α , λ = 0.15418 nm. The working current and voltage are 40 mA and 40 kV, respectively, with a test step of 0.02 and 2 θ = 5~80°.

Scanning electron microscopy Characterization

The microstructure of the MOFs was observed and analysed using the JSM-6710F field emission scanning electron microscope, manufactured by a Japanese electronics company. Grind a small amount of the sample to be tested and apply it onto a conductive adhesive. Record the order of the sample arrangement, load the sample onto the sample stage, and insert it into the vacuum chamber for extraction until the vacuum level reaches 9.6 × 10⁻⁵ Pa. Then, activate the electron gun and adjust the focal length and astigmatism of the objective lens to observe the microstructure of the composite material.

Results and Discussion**Physical Characteristics**

The physical characteristics; colour, texture, yield, and solubility of Fe-based MOFs synthesised with terephthalic acid (BDC) and fumaric acid (FUM) are presented in Table 1. As shown in Table 1, the brown colour confirms the successful coordination of Fe²⁺ ions. The samples appeared amorphous, indicating limited crystallinity due to the mild synthesis conditions. Among the synthesized materials, Fe-BDC gave the highest yield (68.97%), while Fe-FUM yielded 48.14% as summarized in table 1. This superior yield for Fe-BDC is attributed to the rigid aromatic BDC linker, which promotes the formation of a more stable and extended framework. The solubility results presented in Table 1 further reveal that both MOFs are insoluble in ethanol and chloroform but soluble in DMSO, while Fe-FUM exhibits partial solubility in water. The greater hydrolytic stability of Fe-BDC can be attributed to stronger Fe–O coordination and the hydrophobic aromatic nature of the terephthalate linker, whereas the fumarate-based framework possesses relatively higher hydrophilicity.

Table 1: Physical Characteristics of the synthesized MOFs

MOFs	Colour	Texture	% Yield	Solubility				Average Size (nm)
				H ₂ O	EtOH	CHCl ₃	DMSO	
Fe-BDC	Brown	Amorphous	68.97	IS	PS	IS	SL	18
Fe-FUM	Brown	Amorphous	48.14	PS	PS	IS	SL	9

Key: BDC = Benzenedicarboxylic acid, FUM = Fumaric acid, IS = insoluble, PS = partial soluble, SL = soluble

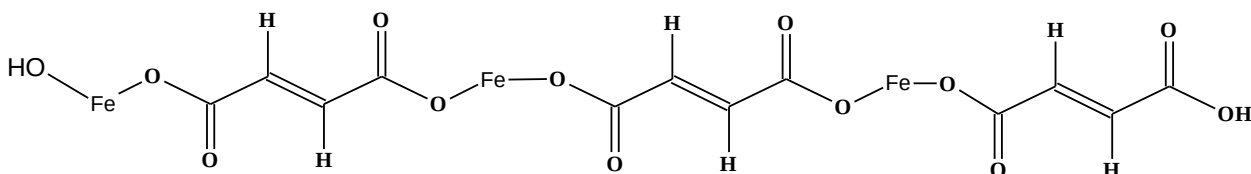
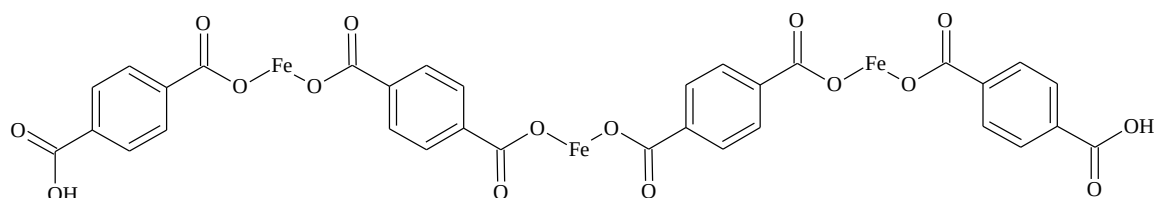
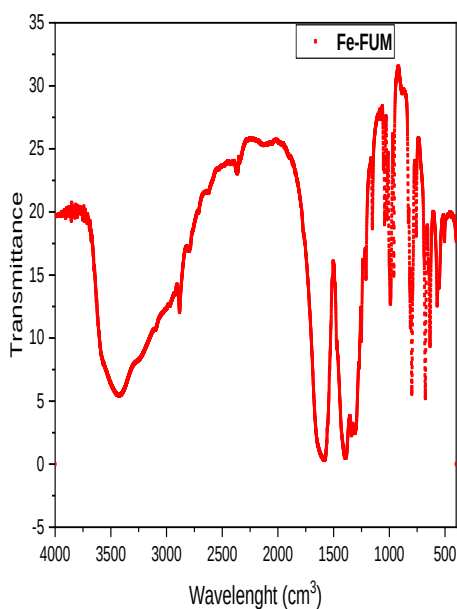

 Fig 1; Iron (III) fumurate; Fe₃O(FUM)₃

 Figure 2: Iron (III) terephthalate; Fe₃(BDC)₄


Figure 3: XRD spectrum Fe-FUM

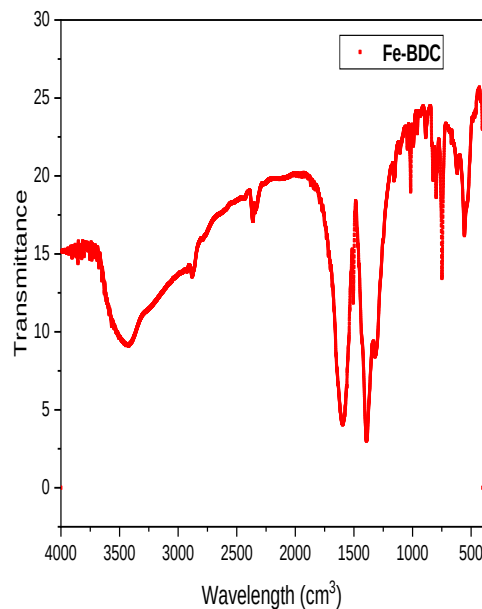


Figure 4: XRD spectrum Fe-BDC

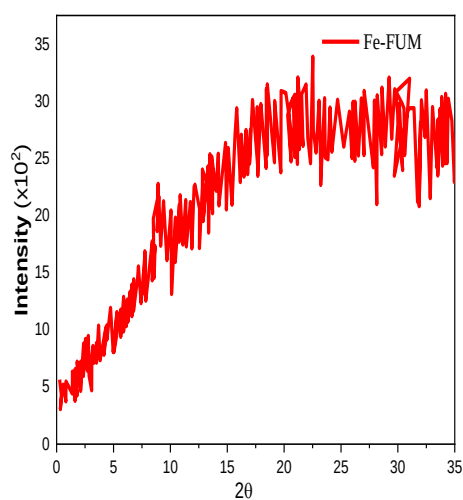


Figure 5: XRD pattern of Fe-FUM

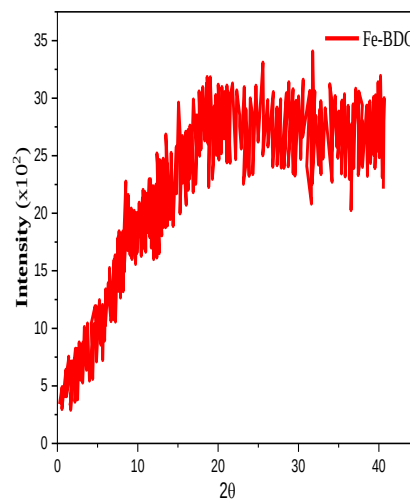


Figure 6: XRD pattern of Fe-BDC

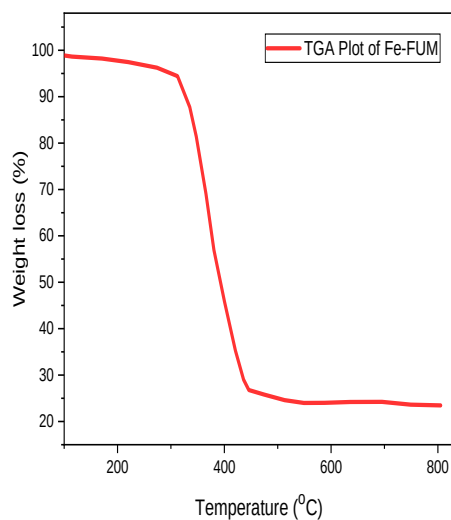


Figure 7: TGA plot of Fe-FUM

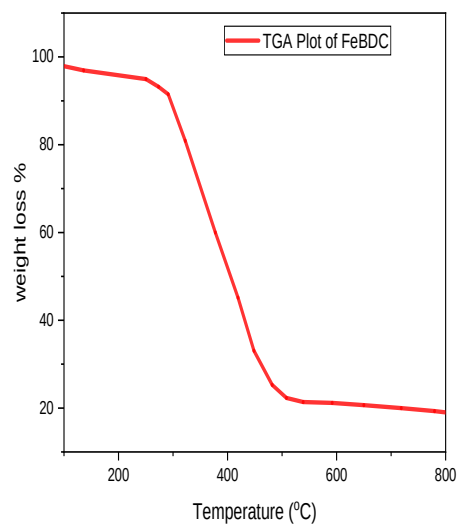


Figure 8: TGA Plot of Fe-BDC

Table 2: BET Result Analysis

S/N	MOFs sample	Pore diameter (nm)	Pore volume (cm ³ g ⁻¹)	Surface area (m ² g ⁻¹)
1	Fe-FUM	3.788	0.164	373.40
2	Fe -BDC	3.489	0.149	779.66

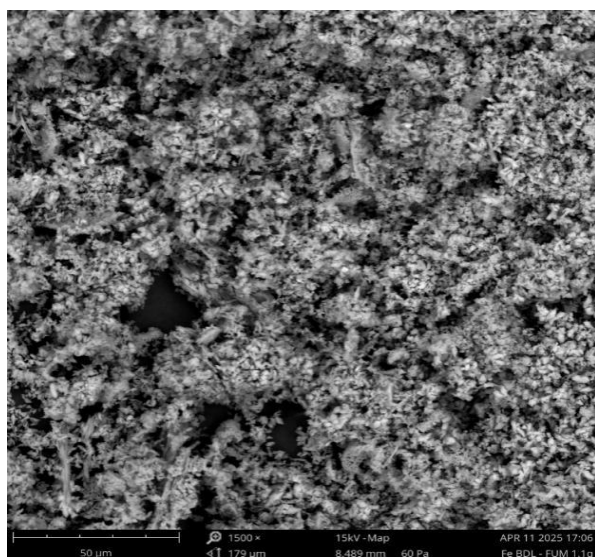


Figure: 9 SEM image of Fe-FUM

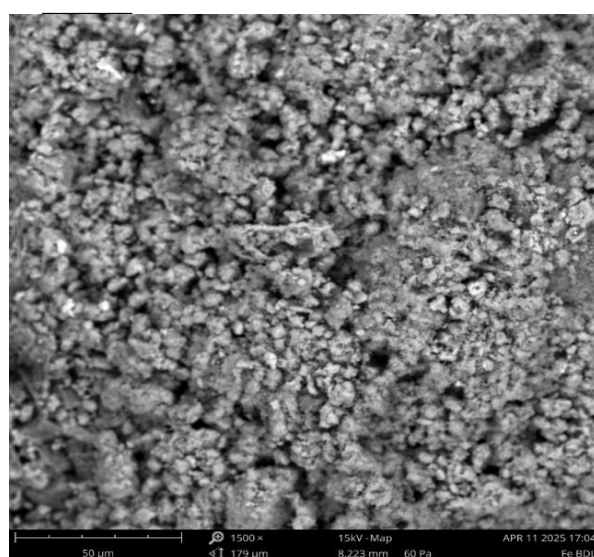


Figure 10: SEM image of Fe-BDC

Table 3: EDX analysis of synthesized Fe-MOFs

Sample	Elemental composition			
	Carbon	Oxygen	Iron	Total
Fe-FUM	59.40	10.84	11.18	99.29
Fe-BDC	24.94	12.46	34.50	99.87

Thermal Stability

Thermogravimetric analysis (TGA) was conducted to evaluate the thermal stability of Fe-based MOFs synthesized at room temperature over a temperature range of 30–850 °C at a heating rate of 10 °C min⁻¹. The thermogravimetric curves of Fe-FUM and Fe-BDC are presented in Fig 5 and 6 respectively. As illustrated in Figure 7 and 8, both Fe-BDC and Fe-FUM exhibited an initial weight loss of approximately 5%, attributed to the removal of physically adsorbed and trapped solvent molecules such as water, DMF, and chloroform from the pore channels. This desolvation step occurred around 350 °C for Fe-BDC and at a lower temperature of approximately 300 °C for Fe-FUM, suggesting weaker solvent–framework interactions and easier solvent diffusion in the fumarate-based structure. The second major weight-loss stage as shown in Figure 7 and 8 corresponded to the decomposition of the organic linkers. Fe-BDC showed a significant

mass loss of about 78% near 500 °C due to the breakdown of the terephthalate linker, while Fe-FUM exhibited a slightly lower weight loss of 75% around 450 °C. The lower decomposition temperature of Fe-FUM indicates reduced thermal stability of the fumarate linker compared to the aromatic terephthalate ligand, which benefits from greater resonance stabilization.

A significant difference was observed in the residual mass after decomposition at 800 °C. Fe-BDC retained approximately 13% residue, whereas Fe-FUM left about 30%, likely due to the formation of stable iron oxide species such as Fe₂O₃ or Fe₃O₄. The higher residue in Fe-FUM may also indicate a higher metal-to-linker ratio or partial carbonization during pyrolysis. These findings are consistent with previous reports by Kiros *et al.* (2017) and Sheta *et al.* (2022), which demonstrated superior thermal stability of terephthalate-based MOFs.

Brunauer-Emmett- Teller analysis

The textural properties (surface area, pore diameter, and pore volume) of the synthesized Fe-FUM and Fe-BDC are summarized in Table 2. Both materials exhibit mesoporous characteristics, since their pore diameters fall within the mesoporous range of 2–50 nm. Fe-FUM shows a slightly larger pore diameter (3.788 nm) and higher pore volume ($0.164 \text{ cm}^3 \text{ g}^{-1}$) than Fe-BDC (3.489 nm and $0.149 \text{ cm}^3 \text{ g}^{-1}$, respectively), indicating wider channels and greater internal void space in the fumarate-based framework. However, Fe-BDC possesses a significantly higher BET surface area ($779.66 \text{ m}^2 \text{ g}^{-1}$) compared to Fe-FUM ($373.40 \text{ m}^2 \text{ g}^{-1}$), suggesting a more extensive porous network and higher density of accessible active sites. This enhanced surface area is likely due to the rigid, aromatic benzene dicarboxylate linker, which promotes framework stability and favourable pore distribution.

The BET surface area of Fe-BDC compares favourably with recent reports: it exceeds those of Fe-BDC derived from recycled PET ($208.45 \text{ m}^2 \text{ g}^{-1}$) and jarosite ($63.72 \text{ m}^2 \text{ g}^{-1}$) (Alao *et al.*, 2025), but is lower than the exceptionally high value of $2240 \text{ m}^2 \text{ g}^{-1}$ reported for highly crystalline, solvothermally synthesised Fe-BDC (Knyazeva *et al.*, 2018). The observed pore diameters are consistent with mesoporous Fe-based MOFs ($\approx 3.4 \text{ nm}$) used in catalysis and environmental remediation (Zhang *et al.*, 2024). The mesoporous nature of both materials is advantageous for adsorption and catalytic applications, as it facilitates rapid diffusion of guest molecules.

Fourier-transform infrared Analysis

The FTIR spectra of the fumaric (FUM), terephthalic (BDC) linkers and their corresponding Fe-MOFs are presented in Figure 2 and 3 respectively. The free linkers display strong C=O stretching bands of carboxylic acids at $1679\text{--}1683 \text{ cm}^{-1}$, which disappear in the MOF spectra, indicating deprotonation and coordination through carboxylate groups. The Fe-MOFs exhibit characteristic asymmetric and symmetric COO^- stretching vibrations at $1577\text{--}1596 \text{ cm}^{-1}$ and $1393\text{--}1424 \text{ cm}^{-1}$, respectively,

confirming metal–carboxylate bond formation. The new absorption bands around $504\text{--}557 \text{ cm}^{-1}$ correspond to Fe–O vibrations, further verifying successful coordination. A broad band near $3415\text{--}3421 \text{ cm}^{-1}$ is attributed to surface or coordinated water molecules. These spectral features collectively confirm the formation of Fe-based MOFs and support the structural variations inferred from XRD and SEM analyses.

X-Ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) patterns of Fe-FUM and Fe-BDC as presented in Figure 5 and 6 reveal insights into their structural features. Both MOFs exhibit broad diffraction peaks rather than sharp reflections, indicating predominantly amorphous structures with low crystallinity. This behavior is typical of MOFs synthesized at room temperature, where limited energy input restricts long-range crystal growth. Fe-FUM displays a broad hump at $2\theta = 25.26^\circ$, corresponding to short-range ordering associated with Fe–fumarate coordination. The absence of sharp peaks confirms poor crystallinity, likely due to irregular metal–ligand geometry or framework disorder. This result was in agreement with commercially prepared Fe-BTC-RT.

Scanning Electron Microscopy (SEM) Analysis

The SEM micrographs as presented in Figure 9 and 10 show distinct morphological variations among the Fe-based MOFs. As shown in Fig 9, Fe-FUM exhibits irregularly shaped, loosely packed particles with a rough and porous surface, suggesting high surface area and heterogeneous nucleation. In contrast, Figure 10 reveal that Fe-BDC possess more compact, uniformly distributed granular particles, indicating improved crystallinity and stronger metal–ligand coordination.

Elemental Composition (EDX) Analysis

The elemental compositions of the synthesized MOFs are summarized in Table 3. As shown in Table 3, both MOFs contain carbon (C), oxygen (O), and iron (Fe), confirming successful synthesis of Fe-based metal–organic frameworks. The Fe-FUM contained the highest carbon content (69.40%)

and relatively low oxygen (0.84%) and iron (11.18%), reflecting the dominant influence of the fumarate linker and a high degree of organic content within the framework. Conversely, Fe-BDC exhibited a much higher iron concentration (34.50%) and balanced proportions of carbon (24.94%) and oxygen (12.46%), signifying a more metal-rich and densely coordinated structure. This composition aligns with the higher crystallinity and thermal stability typically observed in Fe–BDC frameworks (e.g., MIL-53(Fe) and MIL-88B(Fe)). These differences in atomic composition indicate that the type and combination of organic linkers strongly influence the coordination environment and metal-to-ligand ratio, which ultimately dictate the structural and functional behavior of the MOFs.

Conclusion

The present study successfully synthesized the Fe-FUM and Fe-BDC via simple room temperature method. TGA, BET, FTIR, SEM, XRD and EDX spectroscopic techniques were employed for the characterization. The findings indicate that Fe-BDC possesses superior textural properties in terms of surface area, while Fe-FUM exhibits slightly larger pore channels and pore volume. The XRD result reveals that MOFs FeBDC exhibit higher decomposition temperatures and thermal stability (~450 °C) than Fe-FUM (~450 °C), owing to stronger π – π conjugation and metal–carboxylate interactions. The enhanced stability of Fe-BDC is ascribed to its rigid aromatic framework.

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