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Synthesis and Optical–Electrochemical Characterization of Copper–Iron Bimetallic Metal–Organic Frameworks for Supercapacitor Applications

Ayesha Siddiqua*
Faculty of Sciences, The Superior University, Lahore, Pakistan

Daoud Sami
Faculty of Sciences, The Superior University, Lahore, Pakistan

Muhammad Nadeem Ahmad
Faculty of Sciences, The Superior University, Lahore, Pakistan

Kanwal Akhtar
Faculty of Sciences, The Superior University, Lahore, Pakistan
Department of Physics, Government College Women University, Faisalabad, Pakistan

Muhammad Khawar Abbas
Department of physics, Government College University, Faisalabad, Pakistan

ABSTRACT

MOFs had appeared as a new class of spongy materials having crystal structure. These are made up of clusters connected to organic connectors. The things like suitable pore morphology, chemical tuneability and very high surface area proved as a proof to made them attractive for multiple applications in present years as potential materials of electrode for supercapacitors. They owing to their excess of redox-active sites and efficient pathways for charge storage and transfer. These MOFs achieved very fundamental important. Due to their tuneable chemical structure high redox active sites and pros pity metal Organic Framework had been studies at large scale as emerging materials for energy storage. Their well-defined and well-structured frameworks permitted efficient ion transport and charge storage, became the reason to made them highly suitable candidates for supercapacitor electrodes. But MOFs also had restrictions on their direct usage in energy storage system practically because of the poor conductivity of electricity and finite structural stability of multiple pristine MOFs. To reduce these challenges', researchers had explored multiple alternative techniques. For example, metal doping, composite formation, and structural optimization. Iron-based MOFs had secured significant attention. it is due to environmental compatibility, multiple oxidation sites and low cost. This gave efficient redox reactions for charge storage. For reduction of cycling stability and reduced their rate capability, their intrinsic conductivity remains a forever limitation. In frequent studies, the introduction of a Fe MOFs framework into secondary metal had been shown to increase the overall electrochemical performance. copper had proven to be a particularly effective choice

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among various dopants. This is because of its excellent electrical conductivity and ability to produce stable bimetallic structures with iron. additional redox centres and improved electron mobility created due to presence of copper ions in the MOF lattice.

Keywords: Metal Organic Framework, Redox Active Sites, Supercapacitors, Electrochemical Performance, Cycling Stability, Low Cost, Energy Storage Devices

Introduction

MOFs had appeared as a new class of spongy materials having crystal structure. These are made up of clusters connected to organic connectors. Things like suitable pore morphology, chemical tuneability and very high surface area proved as a proof to made them attractive for multiple applications in present years as potential materials of electrode for supercapacitors. They owing to their excess of redox-active sites and efficient pathways for charge storage and transfer. These MOFs achieved very fundamental importance. (R. Zhao, Liang, Zou, & Xu, 2018). Due to their tuneable chemical structure high redox active sites and porosity metal Organic Framework had been studied at large scale as emerging materials for energy storage. Their well-defined and well-structured frameworks permitted efficient ion transport and charge storage, became the reason to made them highly suitable candidates for supercapacitor electrodes. This study determines the way out of production of MOFs. It had great concentration on hydrothermal ways in this regard. It exerted stress on the mechanism for the nourishment of crystals and nucleation. The basic concepts were used the breakdown of bonds and formation of bonds that needed energy. In the synthesis of MOFs these concepts were used because in MOFs there was also a bonding between linkers of iron and ions of metals.(J. Zhao, Yang, Li, & Zhou, 2023)

On other hand research also showed the other way to form MOFs, in this regard any device that could be a source of generation energy could be used for the development of required MOFs. It also described and revealed the sources of heat production. For example, microwaves, waves like X-rays could be obtained that don't have any material for their propagation hydrothermally engaged oven, electric potential and ultrasound in mechanical way. Usually, the techniques that were used for the creation of MOFs were not repeatedly tested, but they were untested before that. Slightly above the boiling point, in aqueous solution the synthesis of MOFs was accomplished by hydrothermal method. On the other hand, another method like solvothermal not just involves water but also few solvents, especially polar. These both synthesis methods were practiced in a closed reactor. Highly costly techniques and tools were required to treat secondary heavy metals solutions.(Zhang, Jiang, Lv, Peng, & Pang, 2023) These included the implementation of exchange of ionic resins or also considering constat bed reactors of carbon activated in packing. The improvement could be brought in the betterment of performance of metals removal. This could be practiced by using particles at nano level having absorbent-like properties, especially magnetic effect related properties. This allowed to acquire very large area of surface. Moreover, last liquid solid separation's simplification by using the magnetic effect could be helpful to prevent the bed packed columns' applications.(Zahoor et al., 2021) These studies showed that the production of Cu ferrites at nano level by using very suitable way of synthesis. This had properties

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like stability in water, acquired larger area of surface and formed material became magnetically practicable. These properties had led its implementations in removal from water different kinds of sorbent materials which were considered heavy metals. Poor conductivity of MOFs composites has always considered as most observable hindrance for improvement of the electrochemical work of various MOFs. The process of hybridization can be used to balance these MOFs with other functional materials. These materials performance in terms of electrical conductivity is considerable for example metals, polymers which are conductive and carbon materials. Among all of them, materials related to carbon , for instance carbon cloth (CC), graphene (GE), carbon fibres (CFs), carb, and carbon nanotubes (CNTs).(Wang et al., 2009) These are usually integrated with MOFs under the condition of solvothermal or reactions based on hydrothermal. In a general process to form composites of MOFs which contain carbon, ions of metals solvents and ligands, are combined with the carbon. In next step the MOF will considered situ development placed on surface of the substrate of carbon at temperature and in a particular duration of time. The process of hybridization of a metal substrate. For instance MOFs and nickel foam can also be obtained through like procedure.(Yewale et al., 2023) The method used for this purpose was hydrothermal way out. It included a method which was performed at changing pH. It Also operated at treatment that was done hydrothermally, this was completed approximately at 120c for almost one hour. It included hexane and water for washing purposes, used to dry and a process like sintering that was performed for almost one hour but changing temperature of almost 100celcius to 200 Celsius. The structural characteristics and behaviour of $CuFe_2O_4$.

Experimental protocol

Copper (11) Chloride Dehydrate, Iron (111) Nitrate 9-Hydrate, . Organic linker Terephthalic Acid, Emplura N,N- Dimethyl-Formamide, Triethylamine, ethanol were used as precursor in the preparation of copper iron based MOF.(Nayak, Nayak, Kittur, Nayak, & Joshi, 2024) To prepare a sample of Copper and Iron based MOFS 50 ml of distilled water taken in a beaker. Then 2.13g of copper (11) Chloride Dehydrate was added in distilled water. Placed that solution for stirring for 10 minutes. Same procedure was repeated for the solution of Iron (111) Nitrate 9-Hydrate of 5.05g in 50ml distilled water and stirred it for 15 minutes. After this mixed both prepared solution and stirred that solution for 30 minutes. Organic linker Terephthalic Acid of 1.25g added in 30ml of Emplura N,N- Dimethyl-Fomamide and kept to stirring purpose for 15 minutes. Then this linker was added to solution of Cu-Fe solution and kept solution on stirring. 10ml of Ethanol were added in above solution and placed for stirring almost 30 minutes. After this the pH of solution was checked that lied between 4 to 6. Then 10ml of Triethylamine in above solution. The addition of this was done with care because of its acidic and smelly nature. At this stage pH was analysed that lied between desirable range which was greater the 4 and less than 6. This solution was kept in autoclave and placed that for 12 hours (overnight) at 140C. Then kept until it cooled down. Then 4 centrifuges tubes were filled with solution till 40ml. placed that centrifuges tubes placed in centrifuge for 5 minutes. The layered (Crystals) inside tubes were collected and pH was checked with pH paper that lied between 4&6. Then 35ml of Ethanol were filled

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in tubes then again placed in centrifuge for 5 minutes. After keeping out from centrifuges placed in oven for drying purpose to remove solvent. After removal of solvent from MOFs crystals the grinding was done.

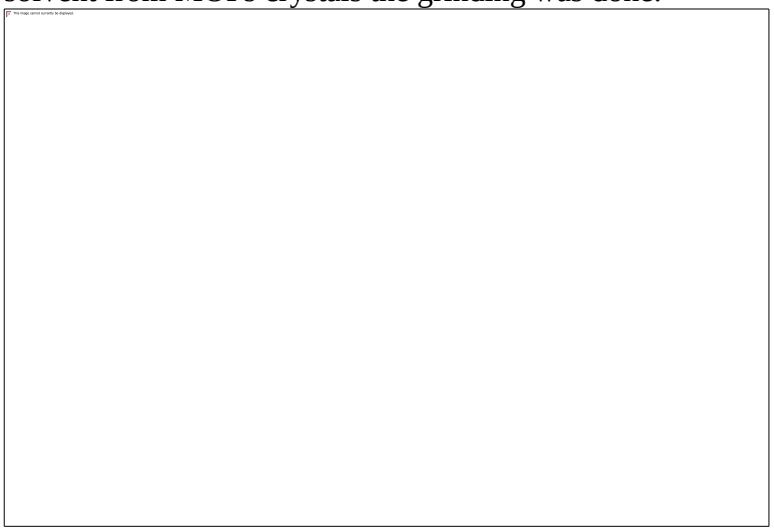


Figure 1: Set up and synthesis procedure for MOF

Hydrothermal synthesis is a ecofriendly, powerful, “bottom-up" chemical technique used for preparation of MOFs. It is used to form highly crystalline, without impurity, and often tailored shaped materials (particles at nano level, single crystals, in form of powders) from aqueous solutions at temperatures greater than 100°C and above 1 atm pressure within a tightly packed or closed vessel. (Muslim, Ali, & Ahmad, 2024). It mimics natural geological procedures to prepare materials. It is often tough task or impossible to produce through ancient or old solid-state methods. It also allow at lower-temperature formation, precise and accurate, high purity, homogeneous crystal-controlled growth.(Tabatabaee, Mohamadinasab, Ghaini, & Khavasi, 2010)

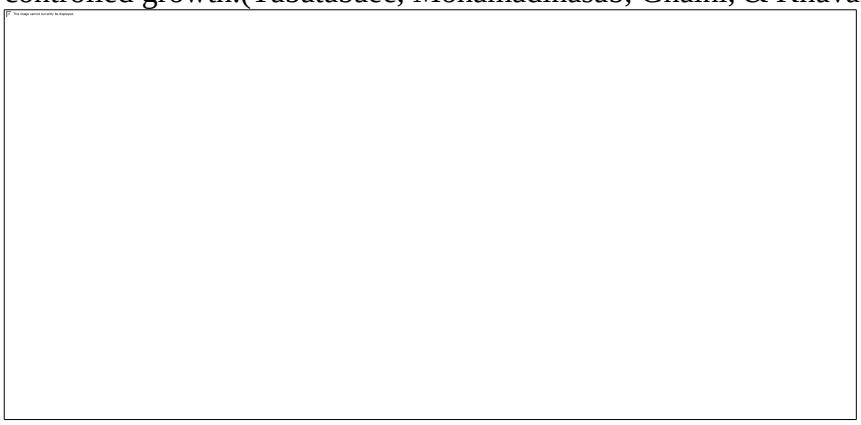


Figure 2: General Flow Chart for Synthesis of MOFs

Characterization

The characterization of prepared MOF was done with the help of analytical techniques. UV-Visible (UV-Vis) spectroscopy is an analytical procedure. It is used in measuring the absorption of light in the UV. It also tells the penetration of light in visible regions.

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It helps in facilitating qualitative identification. It tells quantitative amount determination of analytes which based on their chromophores. It depends on Beer-Lambert law and electronic transitions.(Tummala, Bobba, Satyanarayana, & Technologies, 2022)

Results and discussion

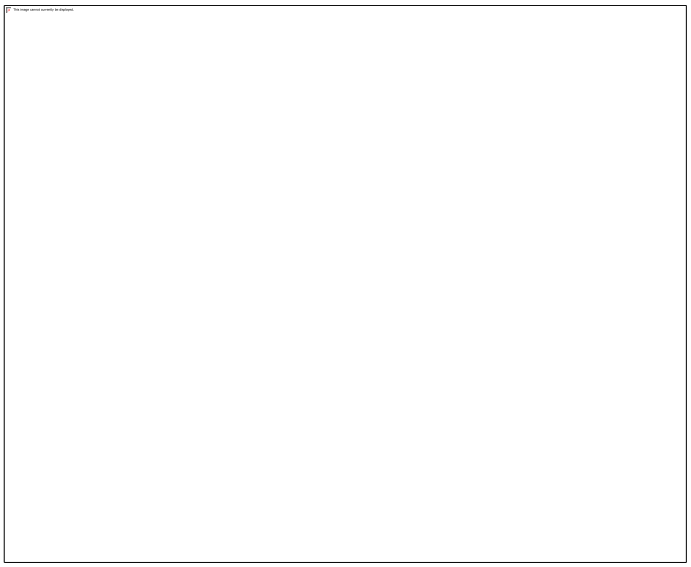


Figure 3: Absorption Spectra or Cu/Fe MOF

The absorption spectrum demonstrated a wide range of absorption in the 300–800 nm UV–visible range, indicating that the bimetallic MOF were successfully prepared.(Yewale et al., 2023) The surface plasmon resonance (SPR) of the material is thought to be represented by an initial absorption band observed in the 460–480 nm region. The second broad absorption maximum frequency is located about 610–630 nm, where it is evident that iron contributes and that Fe and Cu interact strongly. Perhaps because of some nanoparticle agglomeration, the comparatively broad peaks show a relatively large particle size dispersion. As the wavelength increases, the pigment's absorbance progressively increases, suggesting that the bimetallic coupling is causing them to absorb more light. Overall, the spectrum demonstrates that Cu/Fe MOFs were successfully synthesized and have stable optical properties.(Viezbicke, Patel, Davis, & Birnie III, 2015). Absorption spectrum of the prepared Cu/Fe MOF was figure out in the UV–Visible range (300–700 nm) to analyze the optical characteristics of the material. From the UV region to the visible region, as can be seen in the spectrum, absorbance is sluggishly enhancing, portraying good absorption of light over a wide range of wavelengths. Weak absorption is showcased at ~ 450–480 nm, possibly because of a ligand to metal charge transfer or it can be due to the electron transitions between the organic linker and the Cu/Fe metal centers. (Agale et al., 2023) There is a fall down at small extant in absorbance. at about 500 nm and a much greater enhancement occurs in absorption. in visible spectrum. The higher absorbance of the spectrum is in the wavelength range from ~600-650 nm, showing that the Cu/Fe MOF has a great ability to absorb visible light. The energy between Copper and Iron ions

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within the metal–organic framework increased the absorption as well as charge transfer and widens the optical response.(Attia, Mohamed, Barakat, Hassan, & Zoubi, 2022) Typically, the broad absorption band also verify the existence of a strong structure in which electronic interactions are extended.

Table 1: UV–Visible Absorption Characteristics of Cu/Fe MOF

Parameter	**Observation**	**Result**
Kind of Material	Cu/Fe MOF	Metal–Organic Framework
Range of Wavelength	300–700 nm	UV–Visible analysis
Initial absorption value	Minimum at 300–400 nm	Finite UV absorption
Absorption band	Approximately 450–480 nm	Possible electronic transition
Maximum absorbance	Approximately 600–650 nm	Greater visible-light absorption
Optical behaviour	Broader absorption band	Increased charge transfer between Cu and Fe
Application	Photocatalysis, batteries, supercapacitors, pollutant degradation, solar energy conversion	Appropriate for visible-light-driven procedure

The efficient visible-light absorption capacity of Cu/Fe MOF grants it the potential use as a photocatalytic degrader for organic contaminants, as a photocatalytic solar energy conversion material, and as a material used in various environmental remediation technologies.(Mosupi, Masukume, Weng, Musyoka, & Langmi, 2025) In the visible region, it is believed that greater enhancement of the optical response leads to greater creation of a number of electron–hole pairs in response to light irradiation, thus increasing photocatalytic efficiency. Overall, the UV–Visible spectrum showcased that the as- Cu/Fe MOF contain the suitable optical property for applications of supercapacitors (Mendhe & Panda, 2023).

Conclusions

Copper and iron-based MOFs could be proven significant materials for use in further development in field of super capacitors. These have great potentiometer for enrichment regarding storage of energy. The morphology shape structure composition and porosity can be balanced and further attention can be given to the accessible area of surface and active sites. Bimetallic MOFs of copper and iron can be useful for further improvement in electrical conductivity and ability of charge storage.

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