Synthetic Approaches, Classification, Properties and Application of Metal-Organic Frameworks: A Review

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Abstract: Metal-organic (MOF) is a group compound that consists of an ion or metal group coordinated by organic ligands to make many-dimensional structures. MOFs are subclassed by coordinated polymers with speciality porous structures. The pursuit of rapid development in the area of catalysis, solar energy, environmental remediation, wastewater treatment and other aspects of ecological and sustainable chemistry has prompted substantial research by material chemists, physicists, academics and other scientists for the development of porous material. A lot of energy had been invested in scientists, this course by research organizations and public sector agencies from antiquity, but the resulting solution was somewhat undesirable until two decades ago when Metal-Organic Frameworks (MOFs) appeared to offer a glimpse of a solution to those challenging issues. The adaptability, flexibility, uniqueness and usability of the metal-organic frameworks material, and its suitability to every facet of scientific and technological advancement has since triggered an incredible upsurge in studies aimed at uncovering more of the inherent influential properties of the materials, and its improvement for the betterment of material science and research world. This review is aimed at investigating some of the recent advancements/breakthroughs in metal-organic frameworks, synthetic methodologies and applications.

Keywords: Metal-organic frameworks (MOFs); Synthetic methods, Classification, Applications of MOFs

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1.0 Introduction

In the year 1965, a new group of materials known as metal-organic framework (MOF) was first time discovered. The basic molecular structure of MOFs is composed of metal nodes with organic linkers connected to form one, two and three-dimensional highly porous structures. Although MOFs were discovered in the previous century, during the initial days researchers devoted their interest to understanding the fundamental properties and structures of these groups of molecules. However, after realizing the highly porous structure, and excellent catalytic behaviour, a great scope of application of MOFs emerged (Batten, et al., 2013).

Metal-organic frameworks (MOFs) or porous coordination polymers (PCPs) are crystalline materials possessing highly ordered structures consisting of networks formed by single metal ions or metal clusters connected by multidentate organic groups acting as linkers (Stock and Biswas, 2012). MOFs are compounds in which all the components are linked by strong covalent bonds and have porous crystalline structures with high surface areas (Moghadam, et al., 2017).

The structure of MOFs is constituted by inorganic secondary building units (SBUs) and organic linker molecules between them. SBUs are generally metal ions, rare earth elements or lanthanide series while organic linkers are ionic or neutral organic molecules. This dual nature of MOFs allows great manipulation of their structure. By combining different SBUs with different linkers, it is possible to obtain various structures with different properties. In general, the porous structure of MOFs makes them a great candidate for gas storage, adsorption and separation applications. They have been at the centre of many types of research due to their unique properties (Bavykina *et al.*, 2020).

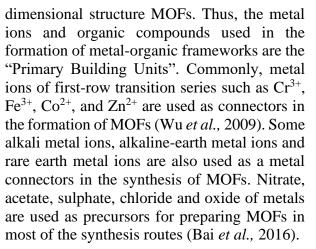
Metal-organic frameworks have a lot to offer to the technological improvements in our world. Their eccentric features have been heavily exploited by researchers for the improvement of energy, environmental, and bio-medicinal applications. And without a doubt, they will continue to influence many other fields in the science community.

Generally, MOFs are formed from two main components, organic linkers or connecting ligands and metal ions. MOFs are made by combining organic linkers and metal ions to form crystal and porous networks. This process is referred to as modular synthesis. Various methods have been developed to obtain the best results in the synthesis process of MOFs. In general, the MOF synthesis method is divided into conventional solvothermal methods and non-conventional methods (Sumida *et. al.*, 2012).

2.0 Structural Features of Metal-Organic Frameworks

2.1 Primary building units

The metal ions (connectors) connecting the organic polymers which are linkers are basic primary units that result in porous three-



2.2 Secondary building units (SBUs)

In MOFs, organic linkers are connected through Metal oxygen-carbon clusters, instead of metal ions alone. These metal-oxygencarbon clusters are referred to as "Secondary Building Units" (SBUs). SBUs have intrinsic geometric properties, which facilitate MOF's topology. In describing MOF structures, secondary building units (SBUs) designate clusters of metal (m) ions joined to more than one other metal via non-metals bonds, (e.g., M-O-M and M-O-C-O-M, oxo and carboxylate bonds, respectively) that from 3D periodic network (Yaghi *et al.*, 2003).

2.0 Classification of Metal-Organic Frameworks

Many synthesized MOFs, however, have relatively weak thermal and chemical stability compared to zeolites which restricts their use in large-scale industrial applications. It is possible to classify MOFs based on framework robustness (Stock & Biswas, 2012). Based on the porosity, MOFs are classified into four different categories and they are as follows:

3.1 First generation MOFs

These MOFs are the earliest examples of porous coordination polymers prepared by linking single metal ions to monodentate Ndonor polytopic pyridine-based ligands



(Gardner *et al.*, 1995). In these types of porous materials, the structure and topology of the obtained framework are controlled by the ligand geometry, These MOFs lack permanent porosity and rigidity (Marinho *et al.*, 2025). The material often collapses upon the removal of solvent molecules from the framework and it is very hard to predict their structure due to the lack of directionality. This behaviour makes these MOFs similar to charged MOFs containing counter anions in the pores. In the first-generation MOFs, the framework undergoes irreversible collapse once the guest molecules are removed (Liu *et al.*, 2014).

3.2 Second generation MOFs

first-generation, Compared to secondgeneration MOFs have stable and rigid frameworks characterized bv permanent porosity that remains even after the guest molecules are removed. They are similar to zeolites and neutral MOFs and built from rigid ligands containing carboxylate groups (Liu, et al, 2014). The incorporation of rigid carboxylate groups into MOF structure helped to lock the metal ions in place and resulted in frameworks with higher crystallinity, rigidity and stability.

3.3 Third generation MOFs

These frameworks are highly flexible and respond well to guest exchange or external stimuli. These MOFs are also known as breathing MOFs. In contrast to secondgeneration MOFs, which become stationary after the incorporation of metal ions, flexible MOFs show guest-responsive behaviour which is very important for the selective adsorption of guest molecules. Third-generation MOFs possess flexible and dynamic frameworks which can change their channels or pores reversibly in response to an external stimulus i.e. radiation, light, pressure etc (Furukawa, *et al.*, 2013).

3.4 Fourth generation MOFs



MOF materials modified post-synthesis fall under this category. These are the recently developed MOFs which retain their underlying topology and integrity after various chemical modifications post-synthesis. These MOFs contain various complex functionalities within the pore environment for more sophisticated applications (Cohen, 2012).

4.0 Basic methods of synthesis of metalorganic frameworks

The synthesis of MOFs has been attracting close attention throughout their rapid development. The porous compound MOFs are most recognized due to their specific functional and structural properties. The main objective of MOF synthesis is to establish appropriate synthesis conditions under which the desired compound can nucleate and grow (Stock & Biswas, 2012).

As highlighted in the introductory note of this review, there are several methodologies involved in the synthesis of Metal-Organic Frameworks like solvothermal and non-solvethermal synthesis, electrochemical synthesis, microwave synthesis, mechano-chemical synthesis, sonochemical synthesis (Soni *et al.*, 2020). However, different criteria should be taken into consideration, namely, choosing cheaper, safer, and/or biocompatible building units, decreasing energy input, using innocuous reaction media, easy activation and continuous manufacturing (Jiang & Yaghi, 2016).

4.1 Sonochemical Synthesis

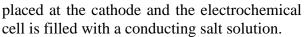
The impact of ultrasound on liquid and colloid systems is mainly caused by cavitation. This phenomenon alternative route to the solution synthesis of MOFs. This method uses frequencies which are higher than those detectable by the human ear. This is a rapid and environment-friendly method in which ultrasonic radiation is used for MOF synthesis that produces high-quality crystals in high yield and the longer the synthesis time is, the larger the crystals formed (Aslani, 2009).

This method is facile, efficient and inexpensive where ultrasound in the frequency range of 20 kHz-10 MHz (Akhbari et al., 2013) causes changes in pressure which generates cavities (small bubbles) in the solvent. Over time, these bubbles grow and collapse (cavitation). Thus, the formation and collapse of bubbles (cavitation) lead to local pressures and temperatures above 1000 atm and 5000 K within the liquid. The hot spots which evolve lead to superior heating and cooling rates which accelerate the formation of MOFs. One advantage of this method over solvothermal synthesis is the avoidance of long reaction times as well as high heating temperatures. (Gedanken, 2004).

4.2 Electrochemical method

Electrochemical synthesis is also an alternative way of synthesizing MOF materials with precise process control (Mueller *et al.*, 2006). This method is fast because of the absence of counter ions such as nitrates, perchlorate or chloride from metal salts, lower temperatures of reaction and extremely quick synthesis (Gangu *et al.*, 2016). The key idea of the electrochemical synthesis of MOFs is that metal ions are introduced not from a solution of the corresponding salt or through the formation of these ions during the reaction of a metal with acid, but as a result of the electrochemical process.

Electrochemical synthesis of MOF uses metal ions continuously supplied through anodic dissolution as a metal source instead of metal salts, which react with the dissolved linker molecules and a conducting salt in the reaction medium (Lee *et. al.*, 2013). Protic solvents are used to avoid the deposition of metal on the cathode, but H_2 is generated in this process (Mueller *et al.*, 2006). In this method ions are placed at the anode while the organic ligand is



It has been pointed out that the product obtained via the electrochemical route reveals inferior quality due to the incorporation of linker molecules and/or conducting salt in the pores during crystallization. The electrochemical route is too feasible to run a consistent procedure to acquire a higher solid content than the ordinary batch reaction (Bedia *et al.*, 2019).

4.3 Microwave (MW) Assisted Methods

The application of electromagnetic radiation such as microwaves in synthetic organic and inorganic chemistry is well known (Jhung et. al., 2005). In this method, energy for the reaction is provided in the form of microwave (MW) radiation (Lee et. al., 2013). The MW method relies on the interaction of electromagnetic radiation with polar molecules in a solvent or conducting ions in a solid (Hu et al., 2009). In contrast to solvothermal methods, where thermal energy is transferred from the source of heat to the solution through the reaction container, in microwave synthesis the interaction is directly with the reactants (Zhang, 2009). The direct interaction of radiation with polar solvents develops hot spots which result in more efficient and faster heating. Furthermore, in microwave-assisted MOF synthesis crystallization happens at the hot spots that form as a result of the direct heating of the solvent, as opposed to the wall of the reactor vessel associated with conventional heating methods. The precursors for the MOF synthesis are put in a microwave oven and heated, this results in rapid heating and a short time for the formation of the MOF ranging between seconds to a few minutes.

Consequently, microwave-assisted synthesis is much faster and results in a smaller particle size (Phan *et al.*, 2023). Other than time-saving benefits, microwave-assisted MOF synthesis also affords the synthesis of MOF crystals



under variable reaction conditions. However, microwave-assisted MOF synthesis does not give good crystals large enough to obtain good structural data. (Ni, 2006).

4.4 Mechanochemical Process

Some chemical reactions and also several physical phenomena can be conducted by the mechanical chemical transformation, which reveals the mechanical breakage of intramolecular bonds (Bai et al., 2016). Nowadays, mechanochemistry is applied in synthetic chemistry and also in multicomponent (binary and higher) reactions to establish co-crystals, inorganic solid-state chemistry, polymer science, and many others. The method uses mechanical forces, instead of using a solvent, at room temperature, to form coordination bonds by either manual grinding of the reagents or more often in automatic ball mills. In some cases, a small amount of solvent may be added to the solid reaction mixture and succeeded in obtaining one-dimensional, twodimensional, three-dimensional and coordination polymers (Friscic et al., 2012).

This method is highly adopted for several reasons. First, environmental issues; the reactions can be performed without organic solvent reactions at ambient temperature. Secondly, quantitative yields of products can be obtained in short durations of reaction (10-16 min) (Beyer and Clausen-Schaumann, 2005) and thirdly, as a side product water is produced in some cases of metal salts. Besides this, the reactions of low solubility of metal (Ganay and Pichon, 2007).

4.5 Solvo-thermal and Non-Solvothermal Synthesis

The term solvothermal implies the use of any solvent and is more general than the term hydrothermal used where the solvent is water (Stock & Biswas, 2012). Non-solvothermal synthesis occurs below the solvent boiling point in open flasks at atmospheric pressure, whereas solvothermal synthesis is carried out at the boiling temperature of the solvent or above this boiling point in special closed chemical reactors at elevated pressure caused by solvent vapour or produced by a pump (Cohen, 2012). Non-solvothermal synthesis does not require complex equipment and can be accomplished both at room temperature and on heating. A common scheme of such synthesis implies the choice of the salt (metal source), organic linker and solvent, as well as the adjustment of pH and temperature to provide the maximal yield of the target MOF. For a precipitate to form, the reagent concentrations should be selected in such a way that the nucleation conditions are achieved (Tanabe & Cohen, 2011). This is usually promoted by increasing temperature and evaporating solvent. In addition, the concentration gradient can be created by slow cooling of the solution, solvent layering or slow diffusion of one of the reactants (Safaei et al., 2019).

Solvothermal synthesis affords higher yields and better crystallinity of the product. Owing to the elevated pressure, the solvent can be heated above its boiling point (at a pressure of 1 atm), which enhances the solubility of the salts involved in the reaction and promotes the reactions. In addition, slow crystallization from a solution leads to the formation of regular large crystals with a high internal surface area (Safaei et al., 2019). Among the advantages of this procedure is the possibility of total control of synthesis conditions for a long period, which allows one to develop reproducible protocols. However, solvothermal synthesis requires special equipment (autoclaves or sealed containers that can withstand increased pressure); in addition, the duration of the process should be taken into account (the synthesis can continue for several weeks and even months).

4.6 Hydrothermal Synthesis



The hydrothermal synthesis technique has similarities with the solvothermal technique. The solvothermal synthesis technique uses non-aqueous solvents; otherwise, the hydrothermal synthesis technique uses a water solvent to dissolve the precursors or reactants (Mueller et al., 2006). The use of water solvent to dissolve precursors is considered more practical besides being a common solvent which is quite abundant if we compare it to non-aqueous solvents in the solvothermal synthesis method. The principle of the hydrothermal technique is not much different from the principle of the solvothermal technique, namely heating the reactants in a closed container using a water medium where this closed system allows the pressure and temperature to increase rapidly (Shekhah et al., 2009).

The parameters that are controlled to obtain a certain structure consist of pressure, temperature, solvent composition, reagent concentration, etc.

The synthesis of MOFs is determined by many factors related to the reaction time and temperature, the solvent used, the nature of the metal ions and the organic ligands, the size of the nodes and their structural characteristics, the presence of counter ions, and the kinetics of the crystallization, which should lead to nucleation and crystal growth. In most cases, the synthesis of the MOFs is performed in the liquid phase by mixing solutions of the ligand and the metal salt.

5.0 Applications of Metal-organic frameworks

Metal-organic frameworks are versatile compounds with unique properties. In MOFs, three properties are acting simultaneously, namely, the crystalline nature, porosity, and presence of strong metal–ligand interaction. A rare combination of these properties makes MOFs an exceptional class of materials. The



high surface area, small density, structural flexibility, and tunable pore functionality of MOFs make them useful in a broad range of intended applications, such as in gas storage and delivery, drug delivery, rechargeable batteries, supercapacitors, separation membranes, catalysis, sensing, etc. (Pettinari *et al*, 2017).

5.1 Sensors

A sensor is a device that responds to a physical or chemical stimulus (e.g. heat, light, sound, pressure, chemical vapour) and transmits a resulting impulse. The porous and tunable structure of MOFs and their composites have been exploited in chemical sensors (Park et al., 2017). Due to the insulating characteristics of MOFs, they show great potential in the preparation of electrochemical sensors supported by carbon, which extends their application to the detection of analytes in industrial different fields. including environmental and biomedical fields, among others (Alkhudhiri et al., 2012). MOFs are great detectors of pollutants due to their affinity for specific groups of organic molecules. Organic solvents, aromatic compounds, and heavy metal ions can also be detected using MOFs made from lanthanides (Kadhom & Deng, 2018).

5.2 Environmental Applications

The environmental applications of metal– organic structures have been widely explored in recent years, as the growing drive to minimize the impacts of chemical residues has become the focus of extensive research around the world. MOFs are used as efficient removers of heavy metals in fluids and aquatic environments (Du *et al.*, 2021). They have been used to remove harmful gases and pollutants, such as carbon dioxide, based on their adsorption capacity. (Ma *et al.*, 2019) Synthesized a MOF compound given its application as a biosensor of organophosphate pesticides, i.e., common pollutants in the agroindustry. These nanomaterials played a substantial role in the detection and removal of organic substances and solvents, organic dyes, antibiotics, volatile organic compounds and other contaminants of industrial effluents.

Another essential environmental application is the detection of ammonia levels as a result of bioaccumulation, which has drawn the attention of environmentalists. Depending on concentration ranges, this can cause serious problems in aquatic food chains. Thus, metalorganic structures are an excellent alternative for identifying levels of environmental pollutants and in the treatment of effluents (Ma *et al.*, 2019).

5.3 Magnetism and its Application

Metal-organic framework materials show magnetism when paramagnetic 3d transition metal nodes are used along with suitable diamagnetic organic linkers and MOFs having magnetic properties are named magnetic metalorganic frameworks (MMOFs). The MOFs consisting of first-row transition metals (V, Cr, Mn, Fe, Co, Ni and Cu) have contributed significantly to developing porous molecular magnets. Close-shell ligands such as oxo, cyano, azido bridges and polycarboxylic ligands, which give weak magnetic interaction, are good candidates for this purpose. (Kurmoo, 2009).

5.4 Gas storage and separation

MOFs have been extensively studied for applications in gas storage. For example, H_2 and CH₄ represent alternative energy resources for future vehicles, and their effective usage remains a challenge for the automotive industry. The capture of toxic industrial gases, such as NH₃ and H₂S, and volatile hydrocarbons, like benzene, as well as the removal of SO₂ and NOx from flue gas, are of great importance for environmental protection (Wu *et al.*, 2009). There are several methods for storing gas effectively but these require high-pressure tanks and multistage compressors. These methods are highly expensive for practical uses and there is a need for simpler and cheaper solutions. To overcome these issues and to find safer storage methods, several materials, like Zeolite or activated porous carbons, have been studied for gas storage (Farha et al., 2010). In this context, MOFs have provided an edge over other materials. The porous structure of MOFs makes them excellent candidates for gas storage and separation. The high surface area aids the adsorption of the gasses while high pore volume determines the amount of gas that can be stored in the structure. Practically any gas can be stored inside the MOF structure however; methane, H_2 and CO_2 are the most important gasses in terms of environmental issues (Wong-Foy et al., 2006).

5.5 Biomedicine applications

Another expanding area in science is biomedicinal applications. As technology improves the search for effective and targeted drug delivery systems has become more and more plausible. The unique structural diversity of MOFs makes them an excellent platform for biomedicinal applications. In contrast to conventional nanomedicines, MOFs show biodegradability and high loading capacity. Their high surface area, large pores, and chemical properties are suitable for novel drug delivery systems and controlled release of compounds. MOFs and their derived materials have been increasingly studied as drug carriers, bioimaging agents, and therapeutic agents due to their excellent physicochemical properties. Promising applications have been investigated on different drugs such as ibuprofen, procainamide, and anticancer treatments (Lu et al., 2018).

5.6 Catalytic Applications



MOFs have found a great niche in every facet of catalysis and catalytic reactions, ranging from photo-catalysis, electro-catalysis, redox catalysis, Bronsted acid catalysis, Lewis acid catalysis etc. In the spirit of candor, MOFs incorporation to catalyze has made catalysis a more interesting field of research and widened its scope of applicability. A lot of new MOFs are synthesized by green chemistry and they are and as well in the future will be useful in the biological aspects to retrieve some environmental issues with the highly efficient recyclable capability of catalyst (Yan et al., 2018).

Some new and novel synthesis techniques were also been done for the synthesis of symmetrical and unsymmetrical compounds/MOFs from stable halides and other components like ethyl potassium xanthogenate. Their catalytic yield for some compounds is greater than 80% which showed a profound increment in the MOF industry (Tabatabaeian *et al.*, 2017).

5.7 Waste Water Treatment

Without a doubt, water is the most essential substance on earth. Through industrial applications or just simple everyday usage, a great deal of wastewater that contains harmful chemicals is produced. Thus, it is very important to establish effective wastewater treatment methods. Their tunable porous structure allows the selective separation of organic and inorganic substances from water. All of the MOFs used in wastewater treatment are water-stable MOF structures. These MOFs can be divided into two different categories. In the first category, hard acids are combined with the hard base while in the second category soft acids are combined with soft acids to form strong bonds in the MOF structure. Waterstable MOFs show great performance in wastewater cleaning due to their excellent selectivity and permeability, tunable structure,

better compatibility and potential recycling after their lifetime =(Jian-Rong, 2012).

6.0 Conclusion

In this review article, we have discussed various synthetic methods of MOFs along with their applications. Different methods lead to the MOFs having different properties. MOFs introduced a huge number of applications including gas storage and separation, catalysis, magnetism, sensors, electrical energy storage systems etc. Several applications of MOFs have not been explored yet, With the increasing research in the field of Metal-Organic Frameworks for various scientific endeavours, there is an urgent need for a better understanding of most of the main synthetic routes towards its synthesis and directives on their applications.

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