Engineering Chemistry
ISSN: 2813-6535, Vol. 2, pp 3-11
doi:10.4028/p-fdqs03

Submitted: 2022-12-25
Revised: 2023-02-14
Accepted: 2023-02-16
Online: 2023-05-16

Synthesis and Optical Properties of Copper Terephthalate Metal Organic Frame Works

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Keywords: Metal organic frameworks, Copper metal, Bandgap energy, photoluminescence.

Abstract. Two new copper-based metal organic frame work (Cu-MOF21 and CU-MOF-22) was synthesized using bromo malonaldehyde and terephthalic an amino terephthalic acid. They synthesized CU-MOFs were characterized by FT-IR, UV-Visible spectroscopy. The XRD diffraction pattern indicated 2 θ at 17.3° and 26.8°. The Tauc's method was employed to calculate the band gap of Cu-MOFs and was found that Cu-MOFS-21 exhibited 3.14 eV and Cu-MOF-22 with average bandgap energy at 3.61 eV attributed to the ligand-metal charge transfer. The results indicate that both Cu-MOFs can be further modified by suitable dopants to enhance the conductivity and reduce the band gap energy.

Introduction

Metal based materials play very important role in various fields to achieve biologically active materials [1-10], energy storage materials [11-14], optical properties [15-20], photoluminescence [21-24], photocatalytic activity to study the dye degradation of hazardous dyes [25-27], sensors for various applications [28-30], high conducting electrical materials [31, 32], solar energy devices an efficient light source [33-36].

Metal organic frame works (MOFs) are molecules with small organic compounds that act as linkers and are connected to the various metal clusters through co-ordination bondage leading a diversified hybrid crystalline structure with varied physical and chemical properties [37]. In recent years, MOFs have been attracted research across the multidisciplinary subjects [38, 39], due to their diversified physical properties such as optical [40, 41], large surface area, high porosity, geometrically well-designed structures and versatility in chemical properties attained due to numerous functional groups of organic linkers [42, 43]. Various heteroatoms such as oxygen, nitrogen, sulfur have been used as organic ligands []. Carbonyl based fused compounds such as terephthalic acid, aminoterephthalic acid, benzenetricarboxylic acid, isonicotinic acid, diphenyl dicarboxylic acid, dihydroxy-1,4-benezenedicarboxylic acid, naphthalenedicarboxylic acid, 1,3,5- benzenetriphosphoric acid, 1,5-naphthalenedisulfonic acid, tetrakis (4-cyanophenyl)methane, adenine, histidine, tartaric acid, azciaic acid, glutaric acid, oxalic acid, fumaric acid, lactic acid, formic acid, gallic acid, 4,4',4''- (S)-trazine-2,4,6-triyl-tribenzoic acid, 4,4'4'' benzene-1,3,5-triyl-tribenzoic acid, benzene-1,2,4,5 tetracarboxylic acid, meso-terakis(4-carboxylicphenyl)porphyrin acid, nitrogen containing such as bipyridine, hexamethylene tetramine, imidazole, 2-methylimidazole, pyridine-4-carboxylic acid, meso-tetrakis(4-carboxylicphenyl)porphyrin acid etc. [44, 45]. To enhance their porosity property, tunable surface area, many metals are incorporated into the organic linkers through coordination bonds [46]. The metals interact with the linker (organic molecules containing more of conjugated π system or lone pair of electrons) either through π- π interactions or through weak Van der walls force or through charge transfer mechanism [47]. These mechanisms help to increase the conductivity properties of MOFs by potentially enhancing the applications of MOFS as energy storage materials, electrochemical system and electrocatalyst. Metals such as copper, nickel, silver, cobalt, zinc, scandium, cadmium, aluminum, strontium, chromium, vanadium, cerium, titanium, zirconium, etc. were widely used metals in enhancing the conductivity and porosity of the many composite and metal organic frame works. Among which copper-based MOFs as an efficient hybrid porous substance that can be used for various applications [48, 49], and among many p-type semiconductors, Cupric oxide is the most prominent metal oxide having high conductivity with a narrow band gap of 1.2 eV resulting in a to a grate choice of material for solar dye sensitized applications, supercapacitors, electrochemical catalysis applications etc. The copper-based MOFs have been synthesized by various methods to obtain varied morphology and electrocatalytic properties. Few of the methods that are used are precipitation method, hydro/solvothermal methods [50]. The optimal molar ration of ligand to metal is one of the essential factors to obtain the highly purified Cu-MOFs. Temperature also plays a crucial role during the synthesis to obtain maximum porosity, highly geometrically structured MOFs. Copper metal organic frame works doped with melamine are reported to have antibacterial property [51]. Thus, copper-based metal organic frame works are synthesized by numerous methods having wide applications in various fields of science. In the present work, two copper-based metal organic frame work were synthesized by chemical methods using bromo monoaldehyde as organic linker by solvothermal method. The synthesized Cu-MOFs are characterized by FT-IR, UV- Visible absorption spectra and XRD. The bandgap energy was calculated using Tauc plots and photoluminescence properties were carried out for both MOFs.

Experimental

Materials: All the chemicals and reagents have been purchased from Avra Synthesis Private Ltd, Sigma Aldrich, India and utilized without further modifications. 1,1,3,3-tetramethoxy propane 99% (Sigma Aldrich), Terephthalic acid 98%, 2-Amino-terephthalic acid 98% (Avra Synthesis Private Ltd), Cu (NO3)2.3H2O 98% (Sigma-Aldrich Ltd).

Preparation of 2-bromomalonaldehyde: Starting material 2-bromomalonaldehyde was prepared using the procedure given in literature. To a 100 ml of aqueous solution of 1, 1, 3, 3-tetramethoxypropane (100g, 0.12M), concentrated HCl (4.3mL) was added and stirred until it forms homogeneous solution, wherein temperature of the reaction mixture was maintained below 35 °C and later bromine (0.15M) solution was added drop wise slowly and stirring was continued for another 30 minutes. Then, reaction mixture was concentrated under vacuum maintaining temperature below 50 °C until thick slurry was obtained, and further washed using 200 mL cold water, 100 ml of cold dichloromethane and dried in vacuum. Yield: 65%, MP: 148 °C (Lit: 148 °C).

Preparation of Cu-MOF, compound 1: Followed the solvothermal method to synthesis these compounds. An equimolar amount of terephthalic acid (0.724g, 0.004 mol) and Cu (NO3)2.3H2O (1.053g, 0.004 mol) were taken in 50 ml DMF and kept at 1200C in hot air oven for 48 hours. Nano polymer MOFs was kept for stirring by adding one equivalent of 2-bromomalonaldehyde and continued for stirring for another two hours. Then centrifuged the reaction mixture (7000 rpm for 5 min) by repeatedly washing the compound formed with DMF, then with ethanol and dried the composite in vacuum oven at 150° C.

Preparation of Cu-MOF, compound 2: Synthesized via the solvothermal method where in same molar concentration of 2-amino terephthalic acid (0.724 g, 0.004 mol) and Cu (NO3)2.3H2O (1.053 g, 0.004 mol) in 50 mL of DMF and placed in hot air oven at 120 °C for 48 hrs. Nano polymer MOFs was kept for stirring by adding one equivalent of 2-bromomalonaldehyde and continued for stirring for another two hours. Further, it was centrifuged (7000 rpm for 5 min) with repeated washing in DMF, then with ethanol and the composite was dried. Figure 1. depicts the synthesis of Cu-MOFs.

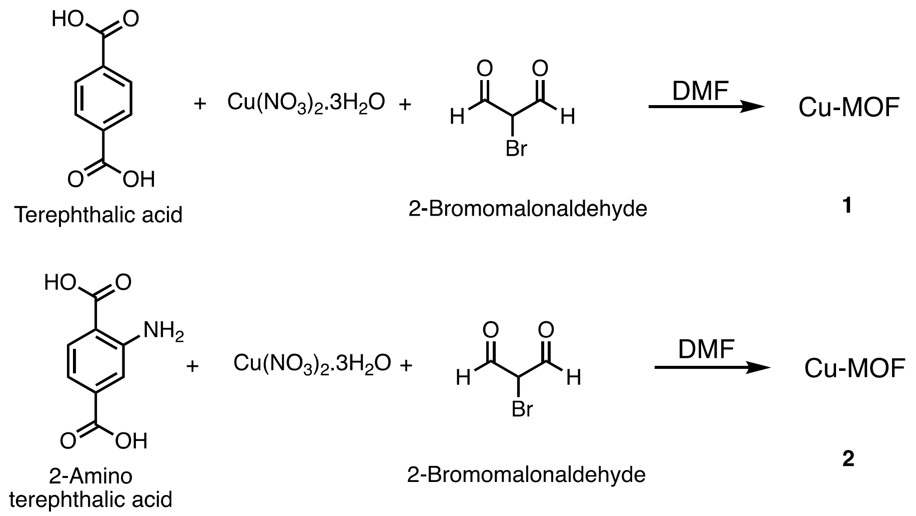

Figure 1. Preparation of Cu-MOFs

Result and Discussion

UV-Visible Spectral studies:

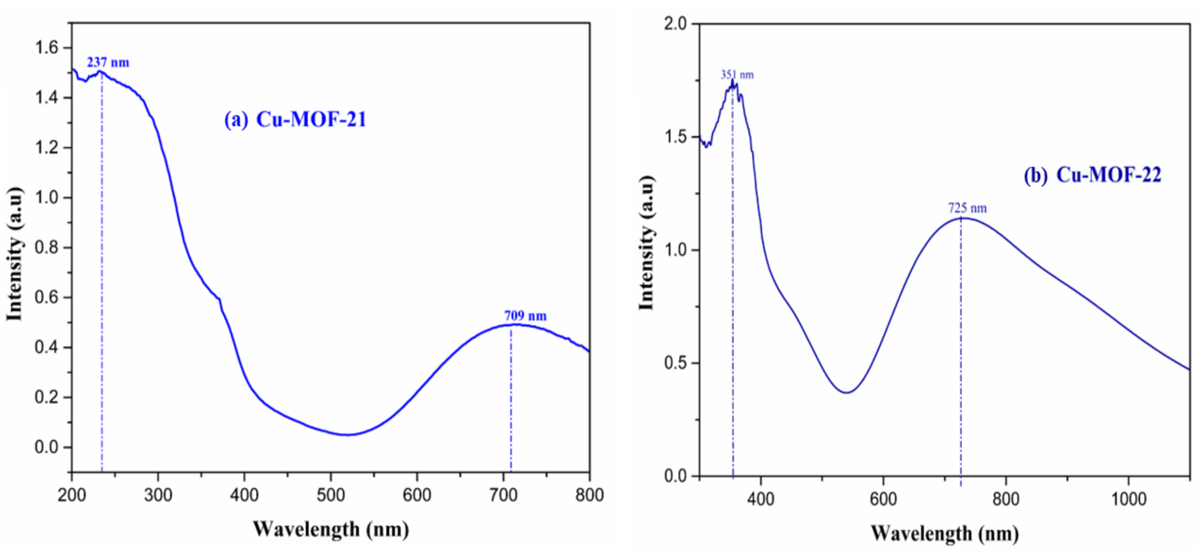

Figure 2. UV-Visible spectrum of (a) Cu-MOF-21 and (b) Cu- MOF-22

The UV-visible spectra of the synthesized Cu-MOFs are shown in the fig No.2. The Cu-MOF-21 showed two absorption bands at 237 nm and 709 nm whereas Cu-MOF-22 showed absorption bands at 351nm and 725 nm. Both the absorption bands recorded may corresponds to the n-π* and π-π* excitation attributing the interaction between the oxygen of the organic framework and apparently due to the optical transition of organic ligands to that of copper metal charge transfer [52].

FT-IR Spectral Analysis: The FT-IR spectrum of both CUMOFs were indicated in the Fig No 3. The presence of the O-H stretching band at 3353 cm^{-1} were observed in the CU-MOF-1. Also, the band at 3024 cm^{-1} corresponds to Ar C-H str, 2890 cm^{-1} at Ald C-H stretching due to fermi resonance and band at 1041 cm^{-1} corresponding to C-O stretching.

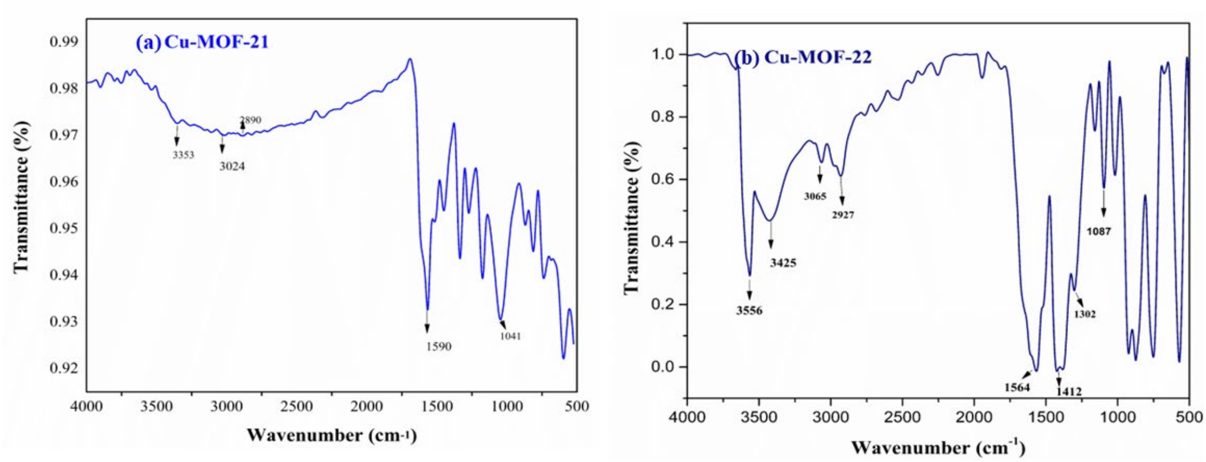

Figure 3. FT-IR spectrum of (a) Cu-MOF-21 and (b) Cu- MOF-22.

A sharp peak at 1590 cm^{-1} indicating the alkene like stretching due to the enolic form of aldehyde upon condensation of bromonaldehyde to the pterphthalic acid. In the FT-IR of Cu-MOF-22 band at 3556 cm^{-1} and 3425 cm^{-1} corresponding to O-H stretching and N-H stretching due to the presence of hydroxyl groups of carboxylic acid and amine group and both the bands have lower frequency which might be due to the coordination bond between the organic ligand and the metal. Similarly, the alkene C=C str was observed at 1564 cm^{-1}, N-H bending at 1412 cm^{-} and 1087 cm^{-} corresponding to O-H stretching frequencies.

XRD diffraction studies: The phase purity and crystallinity of synthesized Cu-MOFs were identified with the XRD diffraction studies as indicated in Figure 4. The A sharp arrow headed tripods with amorphous type of nature of peaks were observed due to the presence of pure copper with a cubic face centered structure. In both the Cu-MOFs the peaks values i,e 2θ at 17.3°, 26.8°, attributed to planes at (020), (040)respectively.. Many other peaks were observed between 2θ at 10° to 50°. Most of the peaks match with the literature reported as JCPDS card no. 89-2838 and 04-0836 for copper [53]. The particle size calculation was done with respect to FESEM as less intense peaks were obtained in the XRD pattern. The crystallite size of Cu-MOFs were calculated using Scherrer equation.

$$D = \frac{k\lambda}{\beta \mathrm{Cos}\theta} \quad \text{------------ (1)}$$

where K is crystallite shape constant (0.94), β is full width at half maximum, λ is wavelength of X-ray Cu-Kα radiation (1.5406 Å) and θ is glancing angle. The crystalline particle sizes were observed between 200-250 nm.

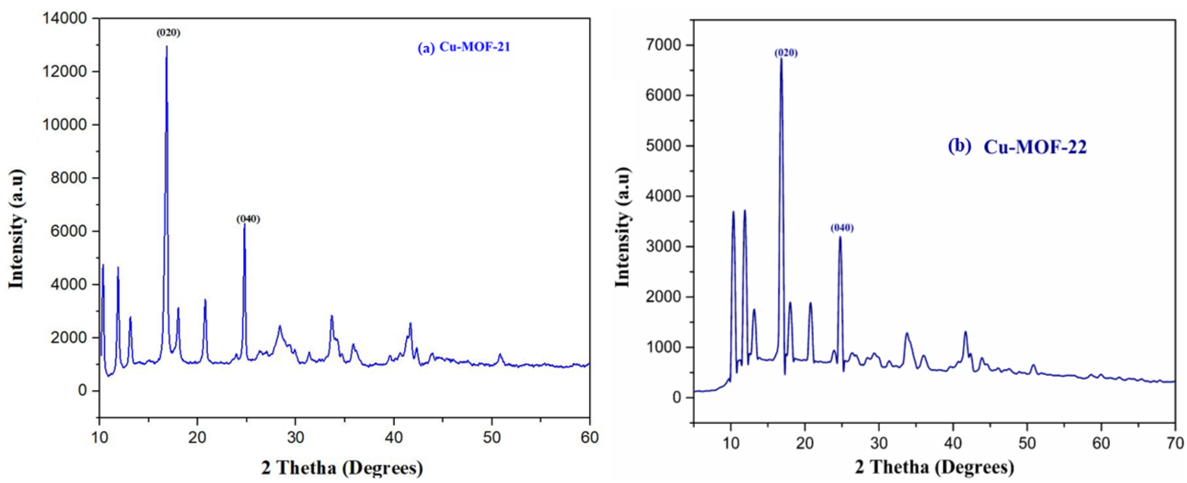

Figure 4. XRD patterns of (a) Cu-MOF-21 and (b) Cu- MOF-22

Photoluminescence features of Cu-MOFs

The PL emission characteristic of Cu-MOFs were shown in Fig. No. 5 strong intense peak was observed at 505 nm was observed for both Cu-MOF-21 and Cu-MOF-22 attributing to the excitation of copper metals ions along with the ligands. A small peak was observed at 756 nm and 757 nm for Cu-MOF-21 and Cu-MOF-22 respectively. However, the presence of amine group didn't have any significant effect on the luminescent intensity of the Cu-MOF-22 with that of Cu-MOF-21.

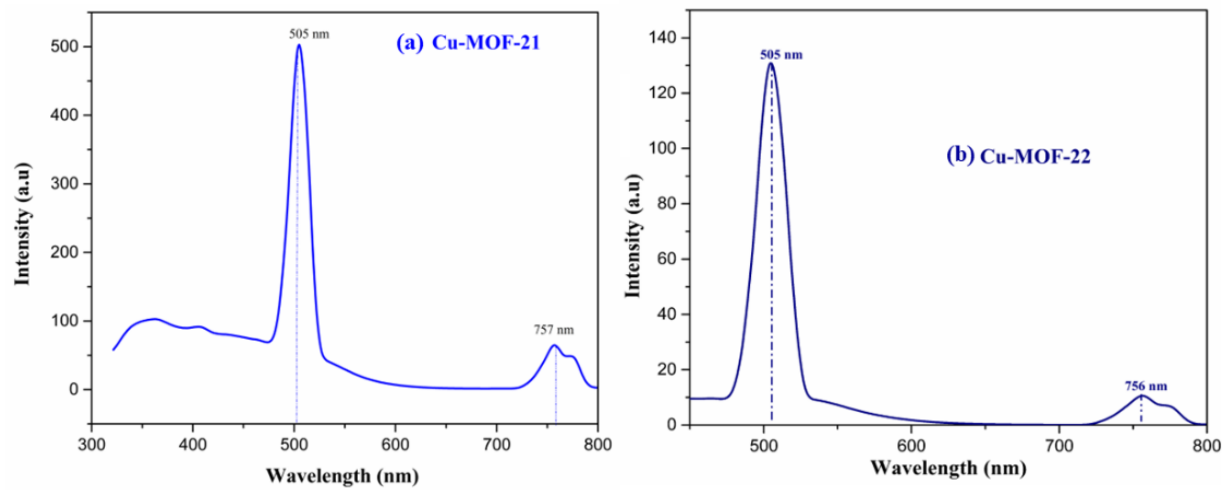

Figure 5. Photoluminescence of (a) Cu-MOF-21 and (b) Cu- MOF-22.

Optical properties.

Band gap analysis: The UV-Visible absorption spectra was carried out using 210 plus UV-Visible absorption Spectrometer. Bandgap energy was calculated by using Tauc's method and equation 2. Graphically the bandgap energy was obtained by extrapolating the tangential line intersecting x axis at hυ = Eg as shown in Fig. From the graph, the bandgap energy for 3.14ev.
The three-bandgap energy for Cu-MOF-2 was at 4.94, 3.04 and 2.85 eV. The average of the three bandgap energies will be 3.61 eV. The increase in bandgap energy in Cu-MOF-2 which may be due to the lattice distortion hybridization. Fig No. 6 indicates the bandgap energy of Cu-MOFs.

$$(\alpha h\nu) = A(h\gamma - E_g)^{1/2} \text{ ----------- (2)}$$

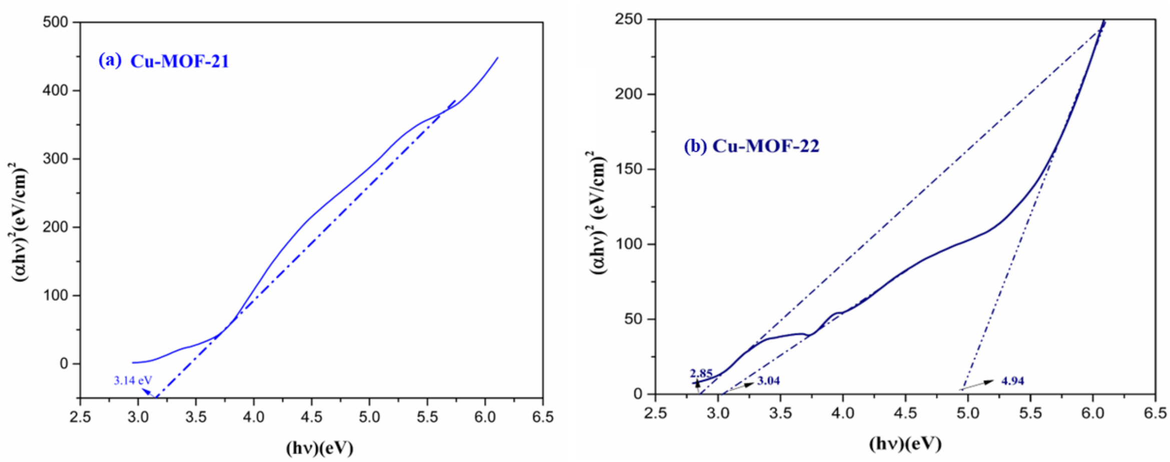

Figure 6. The energy band gaps of (a) Cu-MOF-21 and (b) Cu- MOF-22

Conclusion

This work reports the synthesis of two new Cu-MOFs using bromo monaldehyde as organic linker and copper metal as conducting material. The synthesized CU-MOFs were characterized by FT-IR, XRD and UV-Visible spectrometer. The photoluminescence spectrum inferred the maximum excitation of both the Cu-MOFS at 516 nm. The synthesized Cu-MOF -21 showed 3.14 eV band gap energy whereas the band gap energy for Cu-MOF-22 was at 3.61 eV. Though the obtained band gap energy is more, the bandgap energy can be addressed by adding suitable dopants and this work is in progress by our team to enhance the optical property of the Cu-MOFs.

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