

A comparative study of the morphologies and photocatalytic performances of bis[5-(2-pyridyltetrazolato)]diaquazinc(II) framework synthesized by hydrothermal and microwave methods

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The metal organic framework compound, bis[5-(2-pyridyltetrazolato)]diaquazinc(II) [(2-PTZ)₂Zn(H₂O)₂], has been synthesized by the reaction of 2-cyanopyridine, Zn(NO₃)₂·4H₂O and NaN₃ via both microwave assisted and conventional hydrothermal processes. The product obtained by the microwave process shows needle-like morphology, while that obtained by the conventional process shows rhombohedral morphology. The MOFs are characterised by scanning electronic microscopy, BET surface area analysis, room temperature photoluminescence and UV–vis spectroscopy. IR spectroscopy and single-crystal X-ray crystallographic studies of the MOF obtained by the microwave process have also been carried out. The average crystallite sizes of the two products, as determined by powder X-ray diffraction are found to be 72 nm and 26 nm respectively. Band gaps of the MOFs calculated by using the Tauc plots are 2.8 and 3.1 eV respectively. Their photocatalytic behavior using methylene blue under UV-vis irradiation has been investigated.

Keywords: Metal-organic framework, Microwave synthesis, Hydrothermal synthesis, Photocatalysis, Zinc

Tetrazole metal-organic frameworks (MOFs) are a relatively new class of crystalline coordination polymers, which have the potential for innumerable of applications such as gas storage, chemical separation, heterogeneous catalysis and also as optical, electronic and magnetic materials^{1–6}. Due to their fascinating structures and several possible modes of metal-ligand coordination tunable through the variation of building blocks and reaction conditions⁷, the interest in MOFs is increasing day by day. These are classically synthesized by hydro/solvothermal methods based on mass transfer techniques^{8–11}. However, these methods require very long crystallization duration that may be up to several days with the consumption of a lot of heat energy. Therefore, it is essential to improve the conventional synthesis protocols and develop facile, techno-economically viable and more efficient alternative techniques for large industrial-scale production. Microwave assisted (MW) synthesis has many advantages such as fast crystallization, cost efficiency and low waste production. After interacting with matter microwaves can be reflected, passed or absorbed by the material. Polar molecules have molecular dipole moments which interact with the high frequency electromagnetic radiation. This

interaction causes the molecules to vibrate and rotate which, in turn, raises the temperature of the polar solution¹². It is noteworthy that through homogeneous heating by MW, size and shape of the obtained crystals can also be controlled because it creates uniform seeding, and hence these crystals are empowered with better properties than those produced by conventional solvo/hydrothermal heating¹³.

Recently, some efforts have also been devoted to employ MOFs as photocatalytic materials particularly for the degradation of the organic pollutants¹⁴. The high physiological activity and low toxicity of tetrazole makes it possible to regard their metal complexes as substances of versatile biochemical and biological destination. Besides focused work on metal oxide photocatalysts^{15–17}, many efforts were made to develop other novel efficient photocatalysts under UV/visible/UV-visible irradiations. In the past decade, tetrazole MOFs have attracted intense interest due to their structural diversity and intriguing topologies¹⁸. Some MOFs constructed by transition metals, e.g. Zn(II), Cu(I)/Cu(II), Cd(II), Co(II)/Co(III), and Fe(II)/Fe(III) have been examined as photocatalysts to degrade organic pollutants under UV, visible or UV-vis light^{19–25}. MOF-5 was the first

to be used as a photocatalyst. Natarajan and co-workers²⁶ used a series of MOFs [$\text{Co}_2(4,4'\text{-bpy})(4,4'\text{-obb})_2$], [$\text{Ni}_2(4,4'\text{-bpy})(4,4'\text{-obb})_2 \cdot \text{H}_2\text{O}$], and [$\text{Zn}_2(4,4'\text{-bpy})(4,4'\text{-obb})_2$] with band gap values of 3.11, 3.89 and 4.02 eV, respectively, to degrade different organic dyes, such as orange G, rhodamine B, remazol brilliant blue R, and methylene blue. Later on it was also found that the introduction of different inorganic oxidants (as electron acceptors), such as H_2O_2 could greatly promote the photocatalytic properties of MIL-53(Fe)²⁷. Recently, Wen *et al.*²⁰ synthesized two isomeric 2D Cu(II) coordinated polymers [$\text{Cu}(3\text{-PTZ})$], and studied their photocatalysis for degradation of the organic dye. In addition, a few reports of MOFs having rod/needle type morphologies with high porosity and interesting properties have also appeared recently²⁸.

Herein we report a rapid and facile synthesis of a porous coordination polymer, [$(2\text{-PTZ})_2\text{Zn}(\text{H}_2\text{O})_2$], with needle-like morphology (MW-MOF-NE) by the microwave irradiation method. A comparison is also made with respect to the conventional hydrothermal method which generated rhombohedral morphology (CH-MOF-RH) of the same compound. In both cases the *in situ* [2+3] cycloaddition reaction of the nitrile and the azide yielded the products. The effects of heating methods on the MOF crystal morphology have been carefully examined. Zn-MOF compounds have been characterized by X-ray diffraction, SEM, BET, UV-visible and photoluminescence measurements. The performance of both types of morphologies on photocatalytic degradations of methylene blue has also been compared.

Materials and Methods

$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was procured from Merck India Ltd. 2-Cyanopyridine and sodium azide were purchased from Sigma-Aldrich. All the starting reagents used in this work were used without further purification. The size and morphology of the crystals were determined by RIGAKU Smartlab X-ray diffractometer using $\text{Cu K}\alpha$ (X-ray wavelength (Å) for $\text{K}\alpha = 1.54059$ Å) and scanning electron microscope Zeiss EVO MA 15. Fourier transform infrared (FTIR) spectrum was recorded on a Jasco FT-IR 5300 spectrophotometer (as KBr pellet) within 4000–400 cm^{-1} region. BET analysis was done by surface area analyser-Smart Sorb 93. Photoluminescence spectra were recorded on an Ocean optics system within the range of 285–750 nm. MW-MOF-NE needles were synthesized under microwave irradiation

using modified Samsung microwave oven, (model No. GE83HDT, 2450 MHz, 850 W) as the heating source. Single crystal data for crystal structure analysis were collected on an Oxford Diffraction Xcalibur3 diffractometer using $\text{MoK}\alpha$ radiation.

Synthesis

For synthesis of powder sample of compound MW-MOF-NE, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (298 mg, 1 mmol), 2-cyanopyridine (208 mg, 2 mmol) and sodium azide (195 mg, 3 mmol) were dissolved by stirring in 7 mL of milli-Q water and 3 mL of DMF ($\text{pH} \sim 7.5$) and transferred into a 20 mL pressurized glass tube to be irradiated in the microwave oven at 120 °C for 10 min. For growing single crystals suitable for single-crystal X-ray diffraction, the as-synthesized MW-MOF-NE powdered sample was recrystallized using liquid NH_3 as solvent. For conventional oven synthesis of the MOF (CH-MOF-RH) the above reactants (in the same batch composition) were transferred into a 20 mL Teflon-lined stainless steel reactor and crystallized at 120 °C for 48 h, under autogenous pressure and then brought to the ambient temperature. In both the methods, the product powders were separated by means of centrifugation at 3000 rpm for 10 min. and then dispersed in fresh 1:1 solution of ethanol/water followed by separation via centrifugation. The processes were repeated two more times. Subsequently, the isolated powder samples were dried in an electrical oven at 100 °C for 6 h.

Structure determination and refinement

A single crystal suitable for X-ray crystallography was mounted on a nylon loop. Complete single crystal X-ray data of (1) were collected at 123(2) K with $\text{Mo K}\alpha$ radiation ($k = 0.71073$ Å). The structure was solved by direct methods with SIR97³³ and refined through full-matrix least-squares techniques on F^2 by the SHELXL-97 program³⁴. All non-hydrogen atoms were refined anisotropically. The C-bound hydrogen atoms were located geometrically while the O-bound hydrogen atoms were refined freely. Pertinent crystal data and structural refinement for (1) are summarized in Table 1.

Band gap calculation

The UV-vis spectra of the samples show several distinct absorption peaks and the absorption edges caused by the optical transitions of the ligand-to-metal charge transfer (LMCT). The room temperature diffuse reflectance absorption spectra of MW-MOF-

Table 1 – Crystallographic data of (1)

Mol. Formula	C ₁₂ H ₁₂ N ₁₀ O ₂ Zn
<i>Mr</i> (g mol ⁻¹)	393.68
Crystal system	monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.0312(3), 12.8431(5), 7.2913(3)
α , β , γ (°)	90, 97.223(4), 90
<i>V</i> (Å ³), <i>Z</i>	746.10(5), 2
Calc. density (g cm ⁻³), μ (mm ⁻¹)	1.75239(12), 1.680
Abs correction	'multi-scan'
Transmission factor range	0.97519–1.00000
Refls. (measured), <i>R</i> _{int}	5255, 0.0292
Mean $\sigma(I)/I$	0.0317
θ range	4.24–28.27
Obs. Refls.	1553
Refls. in refinement	1842
<i>R</i> (<i>F</i> _{obs}), <i>R</i> _w (<i>F</i> ₂), <i>S</i>	0.0272, 0.0699, 1.046
Shift (errormax)	0.001
max electron density (e Å ⁻³)	0.319

NE and CH-MOF-RH photocatalysts, converted from reflectance to absorbance by Kubelka-Munk method (Eq. 1),

$$\frac{\alpha}{s} = \frac{(1-R)^2}{2R} \quad \dots (1)$$

where α is the absorption, *S* is the scattering coefficient, and *R* is the reflectance at a given energy. The absorption coefficient α is related to the incident photon energy by the Eq. (2),

$$= A(E - E_g)^n \quad \dots (2)$$

where *A* is a constant for the given material, *E* is the photon energy, *E_g* is the band gap and *n* is a constant of different values (e.g. 1/2, 3/2, 2 and 3) which eventually depends on the type of electronic transition (i.e. permitted/prohibited-direct or indirect transition). To obtain the precise value of the band gap, extrapolation of the linear portion of the plot to abscissa was taken in the $\alpha h\nu^{1/2}$ versus *hν* graph.

Results and Discussion

CH-MOF-RH was synthesized hydrothermally after treating Zn(NO₃)₂·6H₂O with NaN₃ and 2-cyanopyridine at 120 °C for 2 days while the microwave irradiation process required only 10 min to produce fully crystallized MW-MOF-NE sample. Single crystals (1) suitable for X-ray diffraction was obtained through recrystallization of the MW-MOF-NE sample.

Table 2 – Selected bond lengths (Å) and bond angles (°)

Bond lengths (Å)	
Zn1-O1	2.1719(14)
Zn1-N1	2.1401(15)
Zn1-N2	2.1144(14)
O1-H12	0.76(3)
N1-C5	1.350(2)
N2-N3	1.346(2)
Bond angles (deg.)	
N2-Zn1-N1i	101.46(6)
N2-Zn1-O1	90.40(5)
N1i-Zn1-O1	89.35(5)
C6-N5-Zn1	112.04(12)
N3-N2-Zn1	141.75(12)
C1-N1-Zn1	

Symmetry code *i* = 2-*x*, 1-*y*, 1-*z*.

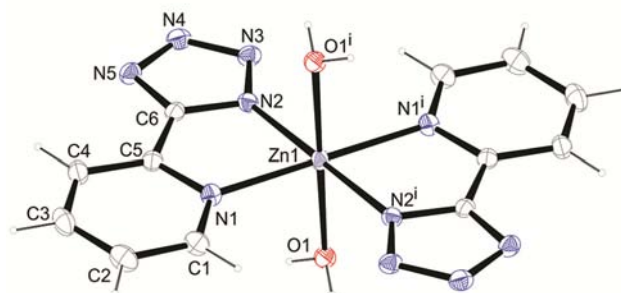


Fig. 1 – Coordination environment of the Zn(II) in complex (1).

Crystal structure of (1)

Compound (1) crystallizes in the monoclinic crystal system with the space group *P*2₁/*c* and *Z* = 2 (see Table 1). In the crystal structure (Fig. 1) the central Zn(II) is coordinated in a transoid distorted octahedral geometry which is completed by two water molecules, two nitrogen atoms from different tetrazolyl groups and two nitrogen atoms from pyridyl groups respectively, resulting in the formation of the centrosymmetric monomeric zinc complex.

The distance of Zn1-N2 (2.1145 Å) is shorter than that of Zn1-N1 (2.1401 Å) with bond angles varying from 90.40° to 89.32° (Table 2). Each Zn(II) mononuclear unit exhibits both proton donors (water molecules) and acceptors (uncoordinated N atoms on the tetrazole rings) and can therefore act as a good building unit for hydrogen bonded networks. As shown in Fig. 2a, the classic hydrogen bonds between uncoordinated N atom of tetrazolyl groups and coordinated water molecules (O1-H11...N5 2.776 Å, O1-H12...N4 2.833 Å) link neighboring mononuclear [Zn(2-PTZ)₂(H₂O)₂] units resulting in an infinite

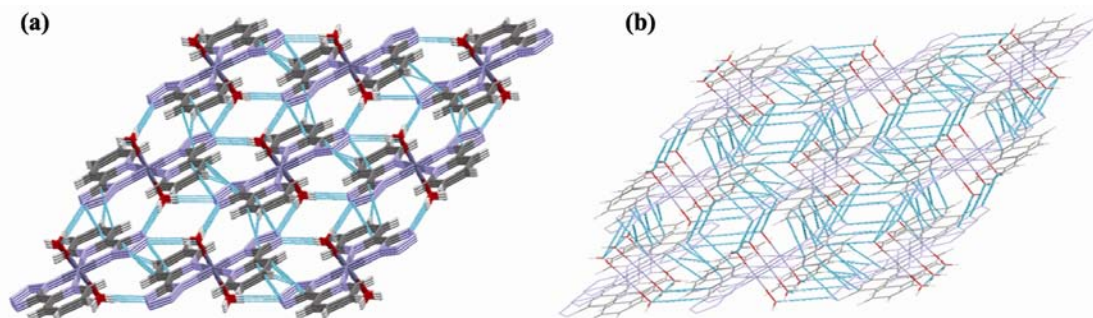


Fig. 2 – (a) 2D network of (1), parallel to the [011] plane, and, (b) 3D network of (1) along c-axis.

hydrogen bonded layer running parallel to [011]. Furthermore, the crystal structure also contains intermolecular (C2-H2—O1 3.302 Å) hydrogen bonding interactions between the 2-PTZ ligands and the coordinated water molecules that construct a 3D net structure (Fig. 2b). The data are slightly different compared to a previously reported structure²⁹.

Characterization

In IR spectrum of sample (1), a peak at 3400–3500 cm^{-1} suggests the presence of coordinated water molecules (Supplementary Data, Fig. S1). The absence of absorptions around 2300 cm^{-1} and $\sim 2100 \text{ cm}^{-1}$ indicate the elimination of cyano and azide groups, while the peaks in the range 1657–1447 cm^{-1} indicate that the [2+3] cycloaddition reaction between cyano group and azide anion has taken place.

In order to study the size and morphology of the MOF compounds, XRD and SEM data were obtained. The structure of $\text{Zn}(\text{2-PTZ})_2 \cdot 2\text{H}_2\text{O}$ was identified by a careful comparison of the experimental X-ray powder diffraction (PXRD) pattern of both morphologies with the pattern simulated from the single crystal structure of $\text{Zn}(\text{2-PTZ})_2 \cdot 2\text{H}_2\text{O}$ (1). From PXRD patterns (Fig. 3), the crystallites produced by the microwave irradiation method have the same structure as the hydrothermally synthesized ones. The average crystallite size of the samples was determined according to the Debby-Scherer equation: $D_{\text{XRD}} = 0.9\lambda/\beta \cos\theta_B$, where D_{XRD} is the average crystallite size (nm), λ is the X-ray wavelength (Cu K α , 1.542 Å), β is the full-width at half-maximum intensity (in radian), and θ_B the half of diffraction peak angle ($2\theta_B$). The average crystallite sizes of CH-MOF-RH and MW-MOF-NE were calculated to be 72 nm and 26 nm, respectively.

The scanning electron microscopy (SEM) images of the synthesized products are presented in

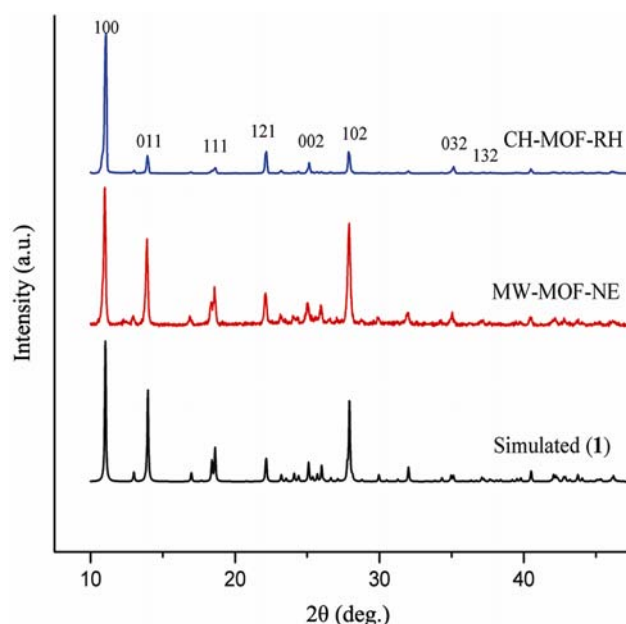


Fig. 3 – Comparison of PXRD patterns of samples with microwave irradiation synthesis and hydrothermal synthesis.

Fig. 4 (a, b). The CH-MOF-RH sample shows widely uneven rhombohedral pieces while MW-MOF-NE is dispersed as needles. Magnified images (Supplementary Data, Fig. S2) reveal rough surface for MW-MOF-NE (Supplementary Data, Fig. S2(b)) and relatively smooth surface for CH-MOF-RH (Supplementary Data, Fig. S2(a)).

Using Brunauer–Emmett–Teller nitrogen-adsorption technique, the specific surface area of rhombohedral and needle-like MOF compounds were found to be 4.8 m^2/g and 67 m^2/g respectively. Gas adsorption studies revealed that needle-like powders had higher nitrogen gas uptake and hence higher surface area. Lesser amount of adsorption in sample CH-MOF-RH may be due to higher agglomeration.

Consequently, the photoluminescent properties of both the samples have been investigated at room

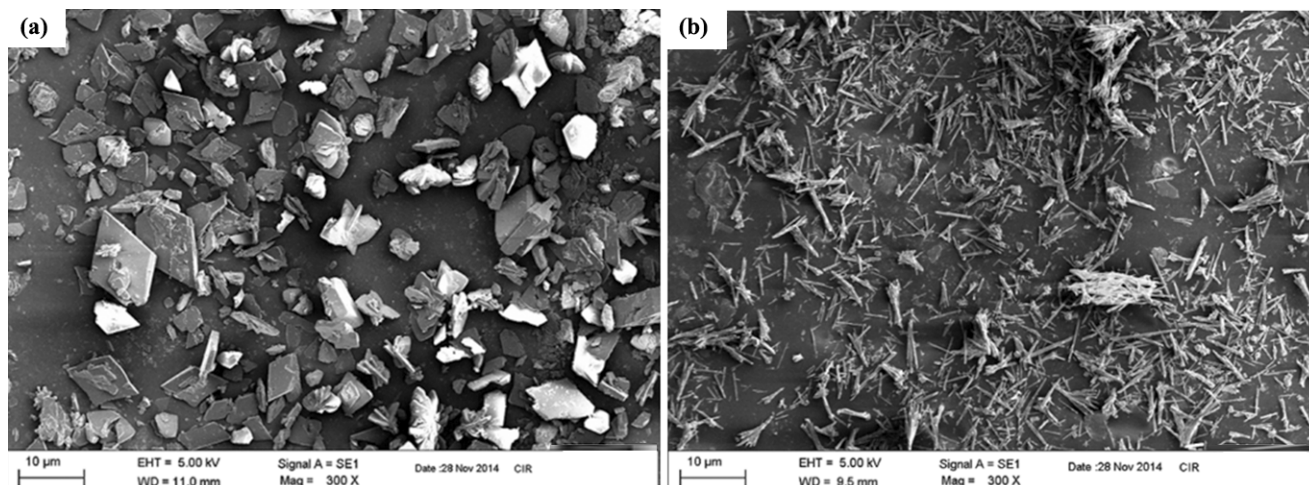


Fig. 4 – SEM images of synthesized Zn-MOF. [(a) CH-MOF-RH with rhombohedral morphology, (b) MW-MOF-NE with needle-like morphology].

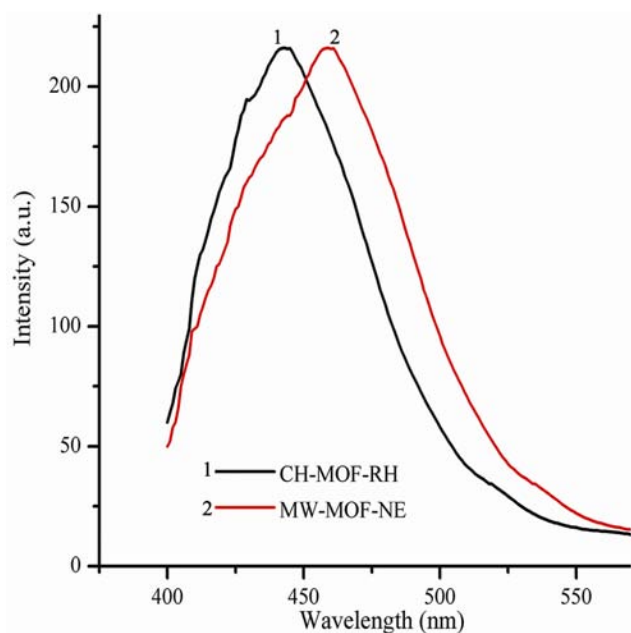


Fig. 5 – The emission spectra of Zn-MOF powders at room temperature. [1, CH-MOF-RH; 2, MW-MOF-NE].

temperature (Fig. 5). Upon irradiation of ultraviolet light at 360 nm, the emission maximum wavelengths for CH-MOF-RH and MW-MOF-NE are 443 and 460 nm, respectively. This emission of CH-MOF-RH and MW-MOF-NE may be attributable to the $5s_{Zn} \rightarrow \pi_{LZ}$ metal-to-ligand charge transfer (MLCT) transition. The UV-vis spectrum (Fig. 6) of the samples show several distinct absorption peaks and the absorption edge is around ~ 430 nm and 390 nm for MW-MOF-NE and CH-MOF-RH, respectively, which is probably caused by the optical transitions of the ligand-to-metal charge transfer (LMCT).

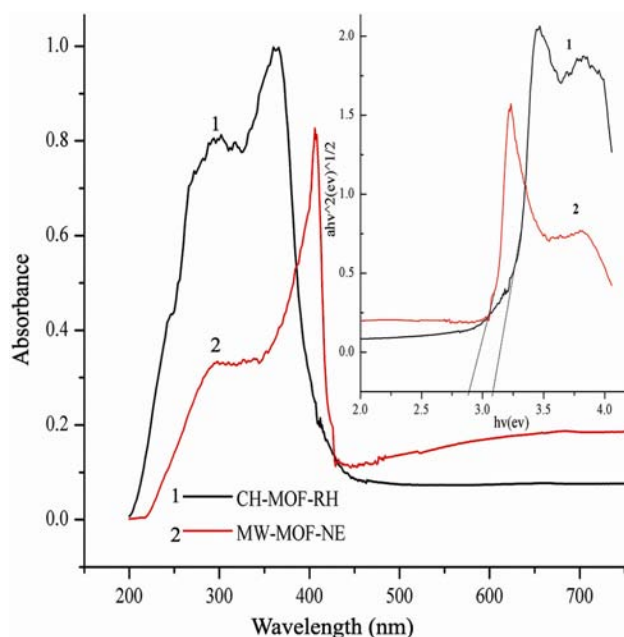


Fig. 6 – UV-vis diffuse reflectance spectra. [1, CH-MOF-RH; 2, MW-MOF-NE. Inset shows the Tauc plot of Zn-MOF samples].

The values of the band gap obtained from the corresponding transitions are 2.8 and 3.1 eV for MW-MOF-NE and CH-MOF-RH, respectively. Although both the morphologies are isostructural difference in particle size, morphology and surface area between the two may lead to distinct band gap size, which may give rise to the difference in their photocatalytic activities. This is already known for oxide nanoparticles³⁰⁻³². Clearly, the band gaps of CH-MOF-RH and MW-MOF-NE follow the order: CH-MOF-RH > MW-MOF-NE while the degradation rates of MB follow the reverse order.

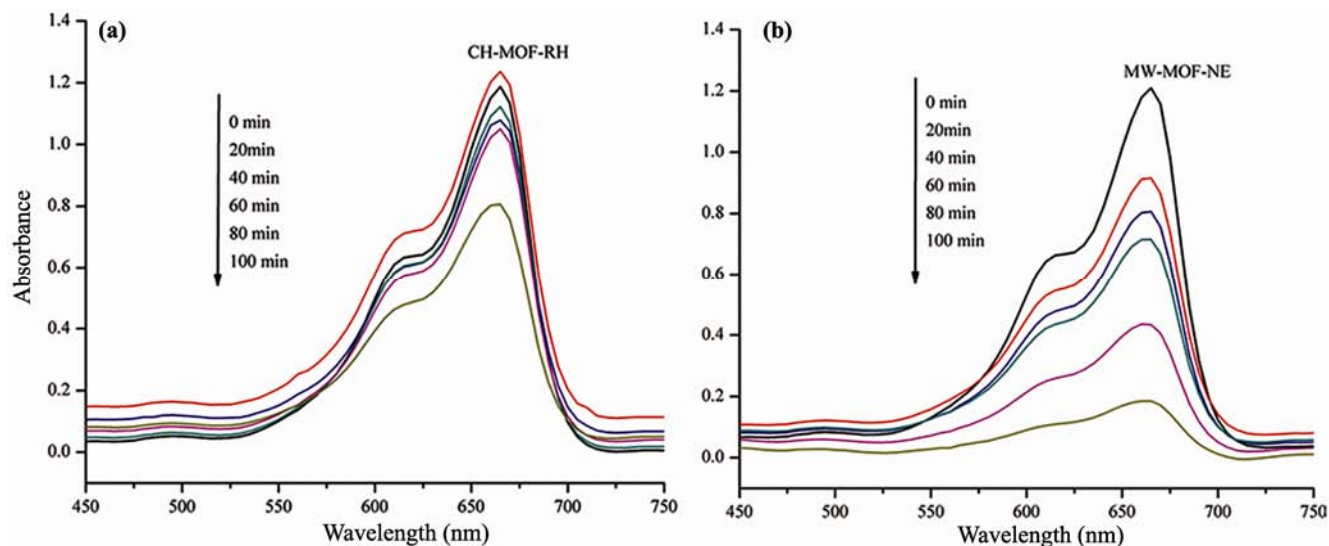


Fig. 7 – MB dye adsorption over (a) CH-MOF-RH photocatalyst, and, (b) over MW-MOF-NE photocatalyst at varying times.

Photocatalytic property

The photocatalytic activities of the MOFs were studied over the degradation of organic dye methylene blue (MB) and it was found that the MW-MOF-NE showed a higher photodegradation for MB than CH-MOF-RH under the same conditions (Fig. 7). Experimental details are given as supplementary data. The characteristic absorption of MB at about 650 nm was selected to monitor the adsorption and photocatalytic degradation processes. Figure 8 shows a comparison of the photocatalytic activities of the two morphologies. Interestingly presence of a small amount of H_2O_2 increases the activity of the catalyst (Supplementary Data, Scheme S1). The photocatalytic activity of each sample was gradually enhanced with the time increasing from 0 to 40 min. It is obvious that the needle-like photocatalyst exhibits much higher activity. It is notable that MW-MOF-NE and CH-MOF-RH respectively show 14% and 2% degradation of organic dyes without H_2O_2 . When the UV-vis light exposure time was increased to 100 min, degradation of MB reaches 99% in the case of MW-MOF-NE. In contrast, CH-MOF-RH requires a little longer time (100 min) for degradation of the same dye and reaches up to 43%. This shows that the needle-like photocatalyst possessed higher activity than the rhomboidal form. It is clear that difference in their average particle size, morphology and surface area as well as slight structural difference leads to difference in their band gaps, which may be the reason for the difference in their photocatalytic activity. The UV light photocatalytic activity of the

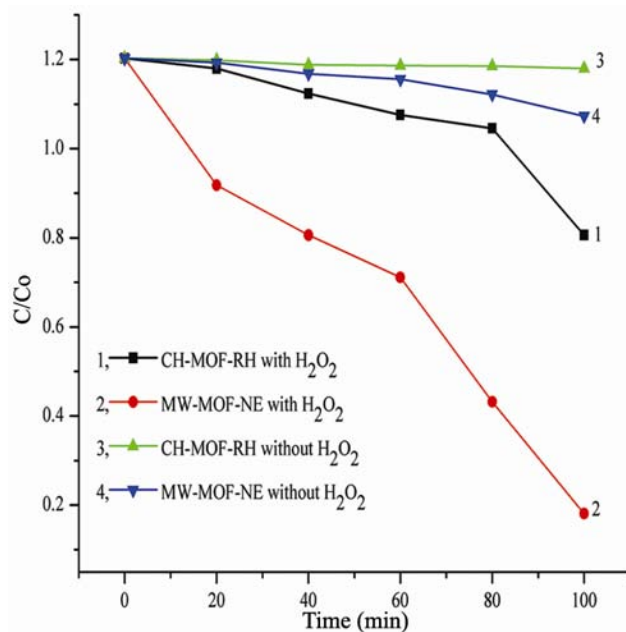


Fig. 8 – Photocatalytic decomposition of MB solution under UV light with Zn-MOF. [1, CH-MOF-RH with H_2O_2 ; 2, MW-MOF-NE with H_2O_2 ; 3, CH-MOF-RH without H_2O_2 ; 4, MW-MOF-NE without H_2O_2].

MW-MOF-NE was also measured by varying the operating parameters, and was found to be closely dependent on the catalyst dosage (Supplementary Data, Fig. S3).

In order to investigate the stability of MW-MOF-NE as UV-visible light photocatalysts, we repeated the photocatalytic degradation of MB three times. It is interesting that similar photocatalytic efficiencies were retained and the PXRD patterns of MW-MOF-NE at

the end of each repeated bleaching experiment were almost identical to that of the as-prepared sample.

Conclusions

In summary, through *in-situ* “click” reactions in different heating methods, two morphologies for zinc(II) were successfully synthesized. The results reveal distinct crystallite size, surface area, photoluminescence, and photocatalytic properties presented by the two morphologies of a MOF with very similar structural features. Considering the high efficiency and high stability on photocatalytic degradation of organic dyes, the needle-like morphology may be a good candidate for photocatalytic applications. The present morphological design approach provides an ideal platform for the fabrication of MOF based materials with high activity, stability and photocatalytic recyclability for sustainable energy and environmental remediation.

Supplementary Data

CCDC 1033231 (1) contains the supplementary crystallographic data for this crystal. This data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Other supplementary data associated with this article, i.e., Figs S1-S3, and, Scheme S1, are available in the electronic form at [http://www.niscair.res.in/jinfo/ijca/IJCA_54A\(07\)851-857_SupplData.pdf](http://www.niscair.res.in/jinfo/ijca/IJCA_54A(07)851-857_SupplData.pdf).

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