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Eco-Friendly Synthesis of Cu/Ni-MOF Adsorbent from Waste PET Bottles for Wastewater Purification

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Abstract:

In this study, terephthalic acid (TPA) was synthesized from waste polyethylene terephthalate (PET) bottles via alkaline hydrolysis. The recovered TPA was then utilized as an organic linker for the hydrothermal synthesis of copper- and nickel-based metal–organic frameworks (Cu-MOF and Ni-MOF). The synthesized TPA and MOFs were characterized using Fourier-transform infrared (FT-IR) and ultraviolet–visible (UV–Vis) spectroscopy. The degradation performance of the prepared MOFs was evaluated by monitoring the removal of Congo red dye in aqueous solution using UV–Vis spectroscopy. Both Cu-MOF and Ni-MOF demonstrated effective dye degradation, highlighting their potential for wastewater treatment applications. This study presents a sustainable and cost-effective approach for recycling plastic bottle waste into value-added MOF materials, advancing green chemistry principles.

Keywords: Waste PET; Alkaline hydrolysis; Solvothermal synthesis; Cu-MOF; Ni-MOF; Congo red.

Introduction:

Introduction The accumulation of plastic waste, particularly polyethylene terephthalate (PET), poses a significant environmental challenge due to its widespread use in packaging and its resistance to degradation. Improper disposal of PET contributes substantially to ecological pollution. Chemical depolymerization of PET to recover terephthalic acid (TPA) is a promising approach, as TPA is a valuable monomer that can be reutilized as an organic linker for synthesizing metal–organic frameworks (MOFs) [1-5]. MOFs are porous crystalline materials formed through the coordination of metal ions or clusters with organic ligands. Their high surface areas, adjustable porosity, and chemical versatility make them attractive for applications such as gas storage, catalysis, and water purification [6-8]. Converting waste PET into MOFs using TPA as a linker provides a solution for plastic waste management while enabling the fabrication of functional materials for environmental remediation [9-11]. Azo dyes such as Congo red are extensively used in textile industries and are toxic and persistent contaminants in aquatic environments. MOFs derived from waste PET have been investigated for dye removal due to their high reactivity, tunable structure, and catalytic potential [12-14]. Despite previous research, the use of TPA recovered directly from waste PET for synthesizing copper- and nickel-based MOFs and evaluating their dye degradation efficiency has been less explored. This study addresses this gap by demonstrating a sustainable method to convert PET waste into functional Cu-MOF and Ni-MOF materials.

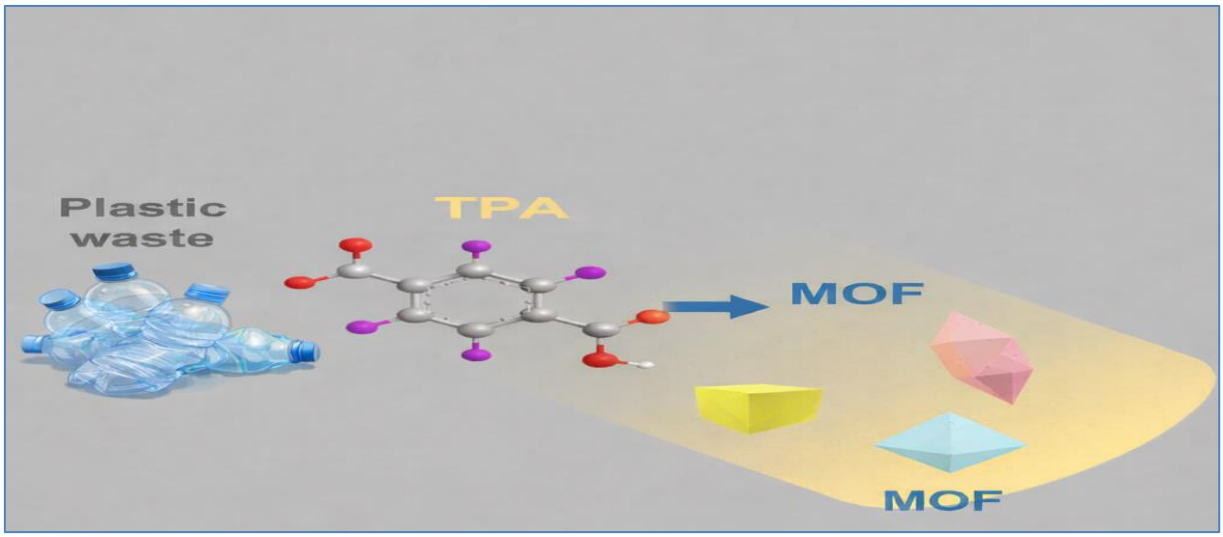

Fig. 1. Schematic Representation of MOF Synthesis from Plastic Waste via Terephthalic Acid (TPA)

Materials and methods:

Materials:

Copper (II) acetate monohydrate ($Cu(CO_2CH_3)_2 \cdot H_2O$), Nickel (II) acetate tetrahydrate ($Ni(OCOCH_3)_2 \cdot 4H_2O$), Ethanol, N-dimethylformamide (DMF), Distilled water, and Congo red. All the chemicals and solvents employed in this research are of analytical grade.

Synthesis of terephthalate acid (TPA):

Hydrothermal Synthesis:

Terephthalic acid (TPA) was synthesized by hydrothermal depolymerization of post-consumer polyethylene terephthalate (PET). PET flakes (6 g, ~2–5 mm) were introduced into a Teflon-lined stainless-steel autoclave containing either distilled water (55 mL) or a mixture of distilled water (50 mL) and ethylene glycol (5 mL). For base-assisted hydrolysis, NaOH (10–12 g) or Na_2CO_3 (~3.5 g) was added; control reactions were performed without base. The autoclave was sealed and heated at 225 °C for 12 h (no base) or at 200–210 °C for 12 h (with base), then cooled naturally to ambient temperature. The reaction mixture was filtered to remove unreacted solids, and in base-assisted runs, the filtrate was acidified with dilute HCl to pH ≈ 2 and left to stand for 30–60 min to precipitate TPA. The precipitate was collected by filtration, washed sequentially with distilled water (×3) and ethanol, and dried under vacuum at 80 °C for 4–6 h.

Following is the mechanism of preparation of TPA from plastic waste

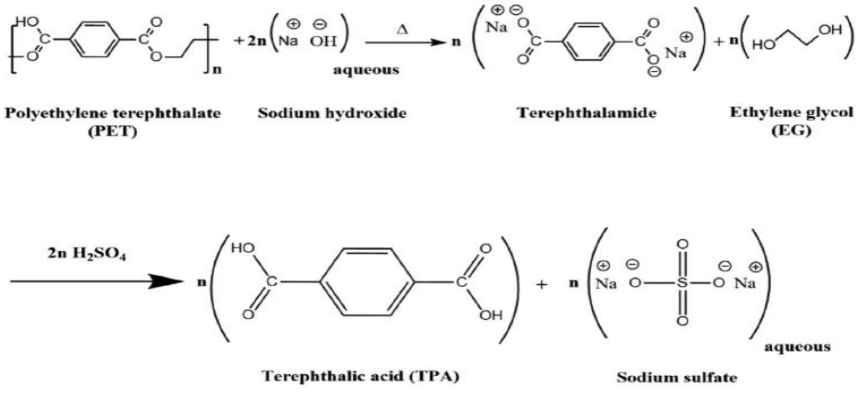

Fig. 2. Alkaline hydrolysis (saponification) of polyethylene terephthalate (PET) to terephthalic acid (TPA).

Synthesis of copper metal-organic framework Cu-MOF:

Copper-based metal–organic frameworks (Cu-MOFs) were synthesized via a solvothermal approach. Copper (II) acetate monohydrate (5.96 mmol) and terephthalic acid (TPA, 4.0 mmol) were dispersed in a solvent mixture of distilled water and dimethylformamide (DMF, total volume 20 mL). The homogeneous solution was transferred into a 100 mL polytetrafluoroethylene-lined stainless-steel autoclave and heated at 160 °C for 12 h. Upon completion, the autoclave was cooled naturally to room temperature. The resulting colloidal suspension was centrifuged and washed three times with ethanol and distilled water. This washing step was repeated to ensure removal of residual solvents and unreacted precursors. The purified product was dried in an oven at 80 °C for 8 h to yield Cu-MOFs, which were subsequently stored for further characterization. The synthesis route illustrates the conversion of plastic-derived TPA into Cu-MOF structures. (6,12)

Synthesis of nickel- metal organic framework Ni-MOF:

Nickel-based metal–organic frameworks (Ni-MOFs) were prepared via a solvothermal method. Nickel (II) acetate tetrahydrate (4.01 mmol) and terephthalic acid (TPA, 4.0 mmol) were dispersed in a mixed solvent of distilled water and dimethylformamide (DMF, 20 mL total). The solution was transferred into a 100 mL polytetrafluoroethylene-lined stainless-steel autoclave and heated at 160 °C for 12 h. After the reaction, the autoclave was cooled naturally to room temperature, and the resulting colloidal suspension was centrifuged. The precipitate was washed three times with ethanol and distilled water, followed by repeated rinsing to ensure removal of residual impurities. The purified product was dried in an oven at 80 °C for 8 h, yielding Ni-MOF crystals, which were stored for subsequent characterization. The synthesis route highlights the conversion of plastic-derived TPA into Ni-MOF structures. (6,12)

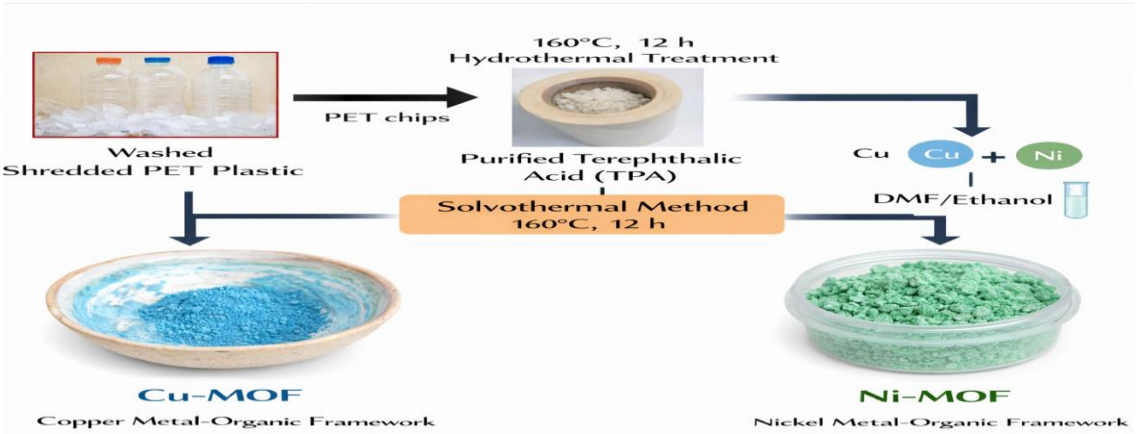

Fig. 3. Schematic Representation of Terephthalic Acid (TPA) Synthesis from PET Waste and Subsequent Cu-MOF and Ni-MOF

Characterization of TPA, Cu-MOF and Ni-MOF :

The functional groups of TPA, Cu-MOF, and Ni-MOF were analysed using Fourier-transform infrared (FTIR) spectroscopy. The FTIR spectra of TPA displayed strong absorption bands corresponding to carboxylate groups, confirming successful depolymerization of PET

into terephthalic acid. In the spectra of Cu-MOF and Ni-MOF, the characteristic ester peaks of PET disappeared, while new bands associated with coordinated carboxylates were observed. These shifts in stretching vibrations verified the effective binding of TPA ligands to Cu^{2+} and Ni^{2+} centers, indicating the formation of stable MOF frameworks. The optical properties of the synthesized MOFs were investigated using UV–Vis spectroscopy. Cu-MOF exhibited a sharp absorbance peak in the 200–250 nm region, attributed to π–π* transitions of the aromatic linker, followed by a gradual decrease in absorbance at higher wavelengths. Ni-MOF showed distinct absorption features in the 400–500 nm range, consistent with d–d transitions of Ni^{2+} ions. These spectral features confirmed the presence of metal–ligand charge transfer interactions and highlighted differences in electronic structures between Cu- and Ni-based MOFs. Photoluminescence (PL) studies further supported the optical characterization. Cu-MOF displayed moderate emission quenching, suggesting efficient electron transfer between the ligand and Cu^{2+} centers. In contrast, Ni-MOF exhibited broader emission bands, which may be attributed to structural defects and ligand field effects. The PL behavior of both MOFs indicates potential utility in photocatalysis and sensing applications, as their emission properties are strongly influenced by metal–ligand interactions. (9)

Result and Discussion:

Physio-chemical characterization

Structural Properties:

The infrared (IR) spectrum of terephthalic acid (TPA) confirms the presence of key functional groups characteristic of its molecular structure. A broad absorption band in the region of 3000–3500 nm corresponds to the O–H stretching vibration of the carboxylic acid group, while a sharp peak near 1700 nm indicates the C=O stretching vibration of the carbonyl group. Additional peaks below 1600 nm are attributed to aromatic C=C stretching and C–H bending modes, consistent with the benzene ring backbone of TPA. These spectral features validate the successful synthesis or isolation of TPA and serve as a benchmark for purity assessment, particularly in workflows involving recycled PET-derived precursors. **[Fig- 5(a)]**

The FT-IR spectrum of the Cu-TPA MOF shows key absorption peaks that confirm the successful formation of the metal-organic framework through coordination between copper ions and terephthalic acid (TPA) linkers: The ester carbonyl peak around 1720 cm^{-1}, present in PET, disappears or is greatly reduced, indicating PET breakdown and TPA formation. Strong bands near 1600 cm^{-1} and 1400 cm^{-1} correspond to asymmetric and symmetric stretching vibrations of carboxylate groups (COO^-), showing coordination of these groups to Cu^{2+} ions. Peaks between 1100 and 1300 cm^{-1} relate to C–O stretching vibrations. Broad bands below 800 cm^{-1} are attributed to metal–oxygen (Cu–O) vibrations, confirming metal coordination. Together, these features demonstrate the Cu-TPA MOF structure formed by coordination bonds between copper ions and the carboxylate groups of TPA, consistent with typical Cu-based MOF spectra. This confirms the successful synthesis of the Cu-MOF from PET-derived TPA. **[Fig- 5(b)]**

The infrared (IR) spectrum of Ni-MOF (Nickel Metal-Organic Framework) reveals key vibrational features that confirm the presence of both organic linkers and metal–ligand coordination. Prominent absorption bands between 1300 and 1700 cm^{-1} correspond to asymmetric and symmetric stretching vibrations of carboxylate groups, indicating successful coordination between nickel ions and the organic linker. Peaks near 600–800 cm^{-1} are typically

associated with Ni–O stretching, confirming metal–oxygen bonding within the framework. Additional bands in the fingerprint region (500–1500 cm^{-1}) reflect complex bending and stretching modes of aromatic rings and linker functionalities. **[Fig- 5(c)]**.

Optical Properties:

The UV-Vis absorbance spectrum of Cu-MOF (Copper Metal-Organic Frame work) provides insight into its electronic transitions and optical properties. The graph shows a strong absorbance peak between 200 and 250 nm, indicating intense $\pi \rightarrow \pi^*$ transitions typically associated with aromatic linkers in the MOF structure. This sharp peak suggests the presence of conjugated organic ligands coordinated to copper centers. As the wavelength increases beyond 250 nm, the absorbance gradually decreases, reflecting reduced electronic excitation in the visible range. The absence of significant absorbance above 300 nm implies that Cu-MOF has a wide optical bandgap and limited visible light absorption, which is relevant for applications in photocatalysis, sensing, or optoelectronic devices **[Fig- 6(a)].**

Nickel-based metal-organic frameworks (Ni-MOFs) synthesized using terephthalic acid (TPA) as the organic linker exhibit characteristic UV-Vis absorption features primarily in the ultraviolet region. The absorption spectra typically show strong absorbance peaks in the UV range (around 200–300 nm), which correspond to the π-π^* transitions of the aromatic rings in the TPA ligand and possible ligand-to-metal charge transfer (LMCT) bands involving Ni centers. These Ni-TPA MOFs generally have minimal absorbance in the visible region (>400 nm), indicating their optical band gaps lie in the UV range, which is consistent with their potential applications in photocatalysis and UV light-driven processes. Recent studies have demonstrated that the UV-Vis absorption profile of Ni-TPA MOFs can be influenced by synthesis conditions, particle size, and post-synthetic modifications, which can slightly shift the absorption edge or intensity. This UV absorption behavior complements the photoluminescence properties of terephthalic acid, providing a comprehensive understanding of the optical characteristics of Ni-MOFs based on TPA. **Fig-6(b).**

The photoluminescence profiles of Ni-MOF reveal distinct emission behaviours linked to their structural configurations. Ni-MOF exhibits a dominant emission peak cantered around 425 nm with an intensity approaching 8000 a.u., indicative of strong blue fluorescence likely arising from ligand-to-metal charge transfer (LMCT) transitions within a well-defined Ni coordination environment. In contrast, Ni-MOF shows its primary emission just below 413 nm, accompanied by a weaker secondary peak near 513 nm, suggesting the presence of multiple emissive centres or defect-related states. The broader spectral tail and dual-peak nature of Ni-MOF imply greater structural heterogeneity or energy transfer pathways absent in Ni-MOF. These differences underscore the influence of metal node architecture and ligand connectivity on the optical properties of nickel-based MOFs, with Ni-MOF demonstrating superior spectral purity and emission intensity suitable for optoelectronic applications. **[Fig- 4(c)].**

The photoluminescence spectrum of Cu-MOF demonstrates a strong and well-defined emission peak around 450 nm, with intensity reaching nearly 10,000 a.u., highlighting its superior optical activity compared to Ni-based MOFs and solvent controls. This intense blue emission is attributed to metal-cantered transitions and possible ligand-to-metal charge transfer processes within the copper coordination framework, which facilitate efficient radiative

recombination. The gradual decline in intensity at longer wavelengths suggests minor contributions from defect states or secondary emissive pathways, but the overall spectral purity remains high. Such behaviour underscores the potential of Cu-MOF as a promising candidate for optoelectronic applications, particularly in light-emitting devices and photonic sensors, where strong emission intensity and stability are critical performance parameters. **[Fig- 4(b).**

The photoluminescence behaviour of terephthalic acid (TPA) is characterized by its weak intrinsic emission under UV excitation, but it becomes highly fluorescent upon hydroxylation to form 2-hydroxyterephthalic acid. This transformation, typically induced by hydroxyl radicals generated in photocatalytic or oxidative processes, produces a strong blue emission cantered around 425 nm with intensity reaching nearly 3,000 a.u.. As a result, TPA is widely employed as a probe molecule in photocatalysis studies, where its conversion to the hydroxylated derivative serves as a sensitive and selective indicator of ·OH radical formation. The distinct PL response of TPA thus provides a reliable method for monitoring oxidative activity, offering valuable insights into the efficiency of advanced oxidation processes and the radical generation capabilities of functional materials. **[Fig- 4(a)]**

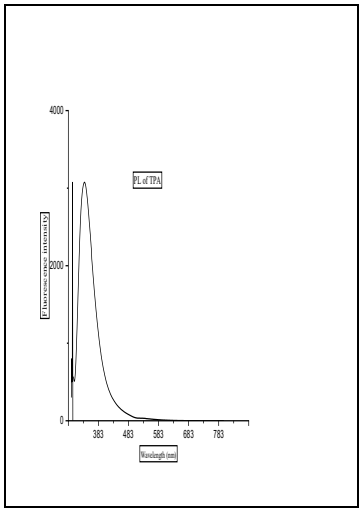

Fig. 4. (a) PL of TPA

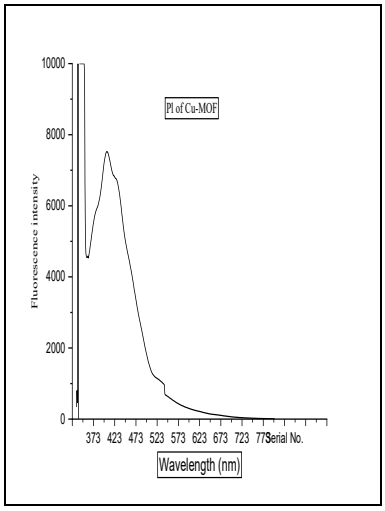

Fig. 4. (b) PL of Cu-MOF

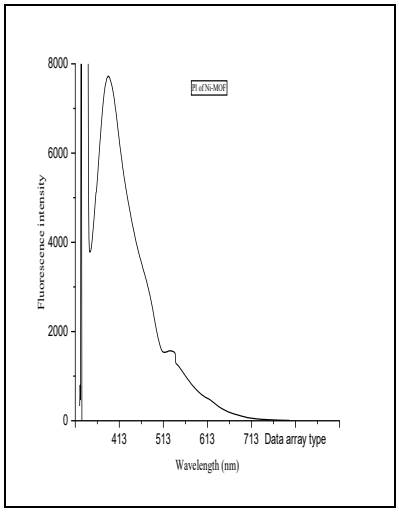

Fig. 4. (c) PL of Ni-MOF

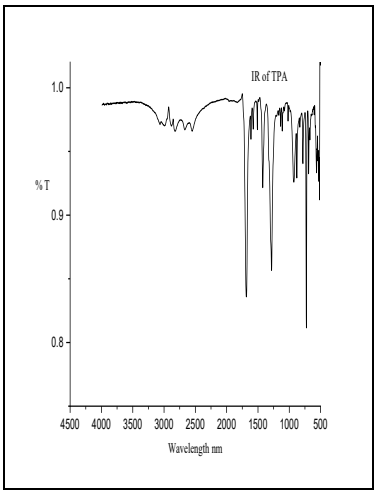

Fig. 5. (a) IR of TPA

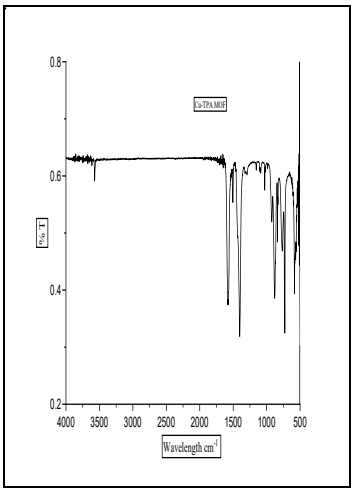

Fig. 5. (b) IR of Cu-MOF

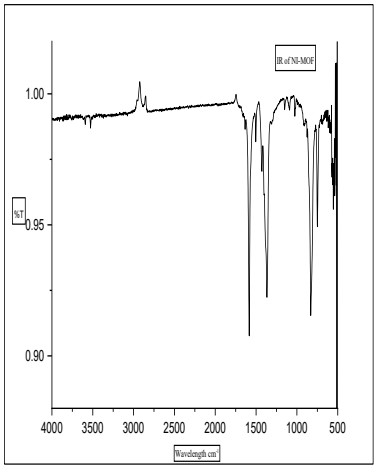

Fig. 5. (c) IR of Ni-MOF

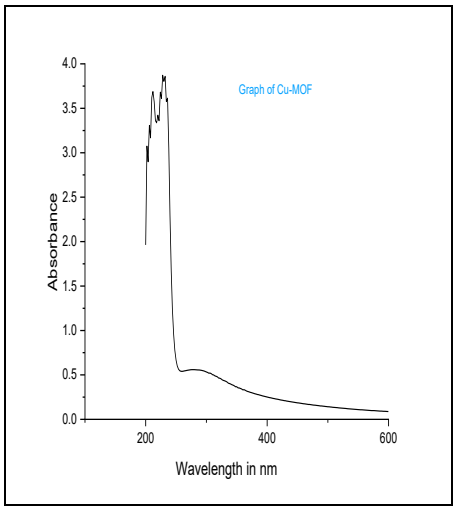

Fig. 6. (a) UV of Cu-MOF

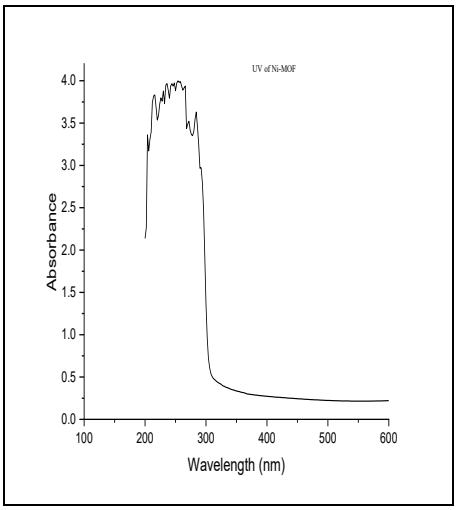

Fig. 6. (b) UV of Ni-MOF

Application:

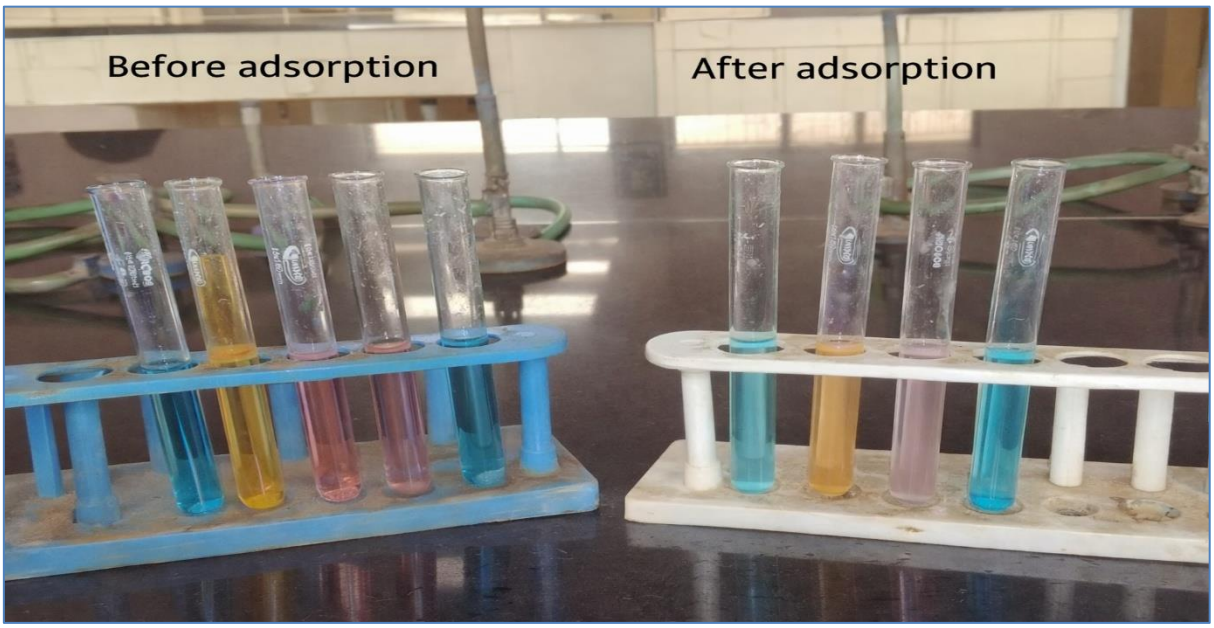

Figure:7 Dye degradation using synthesized MOF

The adsorption behaviour of Cu-MOF and Ni-MOF toward the dye was confirmed by the noticeable reduction in solution colour after treatment. The intense coloration observed before adsorption diminished significantly following contact with the MOFs, indicating effective dye removal from the aqueous phase. This adsorption efficiency can be attributed to the high surface area, porous framework, and availability of active metal sites (Cu^{2+}/Ni^{2+}), which promote electrostatic interactions, π–π stacking, and coordination with dye molecules. Differences in decolorization intensity suggest variation in adsorption efficiency between Cu-MOF and Ni-MOF, likely due to differences in pore structure and metal–ligand interactions. These results highlight the potential of Cu-MOF and Ni-MOF as efficient adsorbents for dye wastewater treatment.

Conclusions:

This study demonstrates a sustainable and low-cost approach for recycling post-consumer PET bottle waste into value-added functional materials. Through alkaline hydrolysis, terephthalic acid (TPA) was successfully recovered and employed as an organic linker for the hydrothermal synthesis of Cu-MOF and Ni-MOF. Spectroscopic characterization confirmed the formation of the desired frameworks, while dye degradation experiments revealed effective catalytic activity against Congo red in aqueous solution. The results highlight the dual benefit of this strategy: mitigating plastic waste accumulation and generating advanced materials for environmental remediation. Both Cu-MOF and Ni-MOF exhibited promising degradation performance, underscoring their potential as eco-friendly alternatives to conventional adsorbents and catalysts. Future work should focus on scaling up PET-to-MOF conversion, exploring adsorption kinetics and reusability, and extending applications to a broader range of pollutants. By integrating waste valorization with functional material design, this research aligns with the principles of green chemistry and circular economy, offering a pathway toward sustainable solutions for wastewater treatment.

Conflict of Interest:

There is no conflict to declare.

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