

HYDROTHERMAL SYNTHESIS OF Cu-BTC MOFs WITH HIGH REMOVAL EFFICIENCY FOR Pb (II) and Cd (II) FROM SPIKED WATER SAMPLES

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ABSTRACT

In this research article, a hydrothermal approach was adopted to synthesise the Cu-BTC metal–organic frameworks and SEM, FTIR, BET and XRD analyses was utilised to characterise them. The adsorption ability of the same was examined towards both metal ions (cadmium and lead) in water solutions under varying experimental parameters, viz. MOF adsorbent dose, temperature, pH of the medium, time duration, etc. The experimental results revealed that the synthesised MOFs exhibited rapid adsorption kinetics along with high removal efficiency of 88% for cadmium and 98% for lead. The adsorption isotherm results showed the best agreement with the Langmuir plot, with a maximum uptake capacity reaching 202 mg/g for both Cd(II) and Pb(II) ions. In addition, kinetic analysis showed that the process follows PSO behaviour. These results can be used as a basis for further research in relation to the regeneration of adsorbents and wastewater treatment on an industrial scale. The research aligns with SDG 6: emphasising clean water and improved sanitation.

Keywords: Hydrothermal Synthesis, Cu-BTC MOFs, Water Treatment, Heavy Metal Adsorption, Porous Material.

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INTRODUCTION

Industrial sectors such as mining and metallurgical processing, electroplating and metal finishing, battery fabrication, electronics manufacturing, textile dyeing units, and chemical production facilities represent significant sources of toxic metal ions released into aquatic systems.¹⁻⁵ The presence of these metal ions in aquatic systems causes severe environmental and public health hazards by virtue of their high toxicity, persistence, and non-biodegradable nature. Prolonged contact with metal ions, including cadmium and lead, can lead to critical health issues, including neurological disorders, kidney, liver, and heart impairment, and interruption of biological functions. Various treatment procedures, viz., membrane filtration, bioremediation, photocatalysis, electrochemical systems, etc., are considered to remediate heavy metals from wastewater. Nevertheless, these methods often face limitations including high operational costs, partial removal efficiency, secondary pollution, and complicated operational procedures.⁶⁻¹⁰ Among the available strategies, the adsorption technique has emerged as one of the most capable methods due to its straightforwardness, excellent performance and operational simplicity. Metal–organic frameworks (MOFs) have recently gained notable interest as advanced adsorbent materials due to their large surface area, controllable porosity, and large range of coordination sites capable of efficient interaction with metal ions.¹¹⁻¹³ In particular, Cu-BTC (HKUST-1) MOFs have shown great potential for environmental remediation; most studies have primarily addressed the uptake of individual metal ions or different MOF systems,¹⁴⁻¹⁷ highlighting the need for further investigation of Cu-BTC MOFs for simultaneous heavy metal remediation under optimised conditions. This study will focus on synthesising and characterising Cu-BTC MOFs from hydrothermal reactions and assessing their capacity for Pb(II) and Cd(II) ion uptake. Such a study will sustainably contribute to water purification, and this relates to SDG-6.

EXPERIMENTAL

Materials and Methods

Chemicals meeting analytical specifications were employed throughout the study. 1,3,5-benzenetricarboxylic acid (BTC), cadmium(II) chloride (CdCl₂), copper(II) nitrate trihydrate

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(Cu(NO₃)₂·3H₂O), N,N-dimethylformamide (DMF), lead(II) nitrate (Pb(NO₃)₂), and ethanol were procured from Sigma-Aldrich.

Synthesis of Copper-Based MOFs

A copper-based MOF was synthesised via a hydrothermal approach. Initially, 1.0 g of BTC was dissolved in a 50 mL mixture of (1:1v/v) DMF/ethanol. In a separate vessel, 2.0 g of copper(II) nitrate trihydrate was added to 50 mL of the above mixture (1:1, v/v). Both precursor solutions were stirred for 15 minutes to ensure complete dissolution. Subsequently, the solution was slowly transferred into the metal salt solution, and the same was kept under stirring for 30 minutes to facilitate metal-ligand coordination. Finally, the resulting mixture was heated at 120 °C in an autoclave for 20 hrs. After reaction completion, the autoclave was permitted to cool down, and finally the blue crystalline product was separated via centrifugation. The product was thoroughly rinsed repeatedly with deionised water followed by ethanol. The obtained solid (Cu-BTC MOFs) was oven-dried at 50 °C overnight and subsequently used for further experiments.

Characterization Techniques

XRD analysis was performed using a SmartLab 3 kW Rigaku diffractometer (GJU, Hisar, Haryana, India) to evaluate the crystalline nature and structural features of the synthesised MOF. The surface morphology and functional groups were characterised by SEM and FTIR spectroscopy at the SAIF Laboratory, Chandigarh. Textural properties were determined through nitrogen adsorption-desorption studies after activating the sample at 393 K for 6 hours. The pore volume and accessible surface area were evaluated by applying the BET model.

Batch Mode Adsorption Studies

To investigate the metal uptake capability of Cu-BTC MOF, the batch mode was opted. The stock solutions for the selected metal ions (1000 mg/L) were made in DI water and further adjusted to the required concentrations. The solutions' pH was carefully adjusted using 0.1 M dilute NaOH or HCl. In a typical experiment, key factors such as the metal solution pH (1-7), MOF adsorbent dose (2-12 mg), metal ion concentration (15-45 ppm), temperature (20-45° C), and 30-450 min of interaction time was changed throughout the studies. After completion of adsorption, the MOF was filtered from the solution via a 0.45 µm membrane. The remaining ion concentration of metals in the filtrate was subsequently quantified using AAS. The equilibrium adsorption capacities for Pb(II) and Cd(II) ions were determined and represented as q_e (mg g⁻¹) according to Eq. (1).

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

Where C₀ represents the starting metal ion concentration and C_e equilibrium residual concentration. The Cu-BTC MOFs amount and solution volume were denoted by M (g) and V (L), respectively.

Furthermore, the eq. (2) was used to get the removal efficiency (%)

$$\text{Removal \%} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

RESULTS AND DISCUSSION

Morphological, Structural Analysis of Cu-BTC MOFs

The PXRD pattern presented in Fig.-1(a) exhibits characteristic diffraction peaks of the Cu-BTC sample appearing at different 2θ values: 6.6°, 9.6°, 11.8°, 14.7°, 16.9°, 17.4°, 20.5° and 24.9°. The obtained diffraction pattern closely matches the reported Cu-BTC MOF structure, confirming successful synthesis. The strong reflection at 2θ=11.8° indicates excellent crystallinity.¹⁸⁻¹⁹ The presence of higher crystallinity increases framework stability and maintains the accessibility of active binding sites, which helps in the efficient uptake of Pb(II) and Cd(II). The obtained FTIR spectrum is displayed in Fig.-1(b). The band observed at 750 cm⁻¹ corresponds to Cu-O stretching vibrations. Further, the band from 850 to 1150 cm⁻¹ corresponds to C-O and O-C=O stretching vibrations for both free and coordinated carboxylate groups.²⁰ The symmetric and asymmetric stretching peaks at 1380, 1470, and 1630 cm⁻¹ of carboxylate groups confirm coordination of the BTC ligand with copper ions.²¹ Around 3350 cm⁻¹, the broad absorption indicated the existence of water molecules and hydroxyl functionalities.

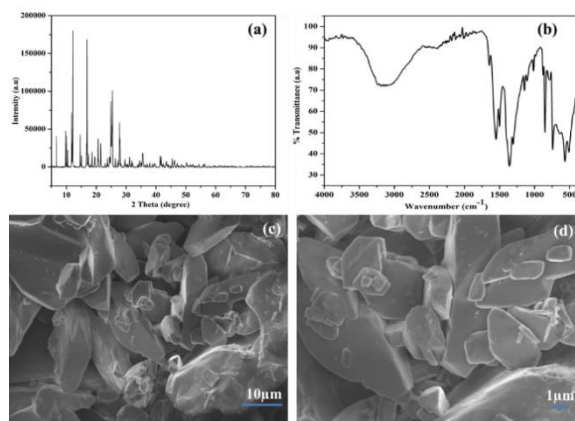


Fig.-1: (a) XRD Characterisation, (b) FTIR Spectrum, and (c-d) SEM Micrographs at different Magnifications of Synthesised Cu-BTC MOFs

The SEM pictures show that the Cu-BTC crystals possess smooth facets and sharp edges in a uniformly well-defined octahedral shape, indicating good crystallinity as displayed in Fig.-1(c-d). The particle sizes are comparatively consistent and appear to be evenly distributed, although slight aggregation is observed, which is typical for MOF materials due to interparticle interactions. The well-developed octahedral shape suggests controlled crystal growth during synthesis and is characteristic of Cu-BTC MOFs reported in the literature.²² The SEM observations clearly indicate the effective development of structurally well-defined Cu-BTC crystals with consistent morphology. Fig.-2(a-b) illustrates the N₂ sorption isotherms, which exhibit a porous framework. The nitrogen uptake in all samples increases significantly at low relative pressures (P/P_0), suggesting that microporosity is the predominant pore structure in the Cu-BTC MOFs. Pores less than 2 nm in width are defined as micropores and provide confined adsorption spaces that strengthen interactions between the Cu-BTC framework and N₂ molecules, allowing significant adsorption even at low pressures. Using the DFT model, the pore size distribution was analysed, indicating an average pore diameter of 1.62 nm. The BET technique was applied to estimate the specific surface area, giving a value of 600.20 m²/g. Additionally, the pore volume was estimated to be 0.33687 cm³/g based on the adsorption analysis. The synergy between high surface area and porosity of these Cu-BTC MOFs will greatly contribute to their excellent adsorption properties in this study, which shows the capability of these synthesised Cu-BTC MOFs as good adsorbents for treating water samples.

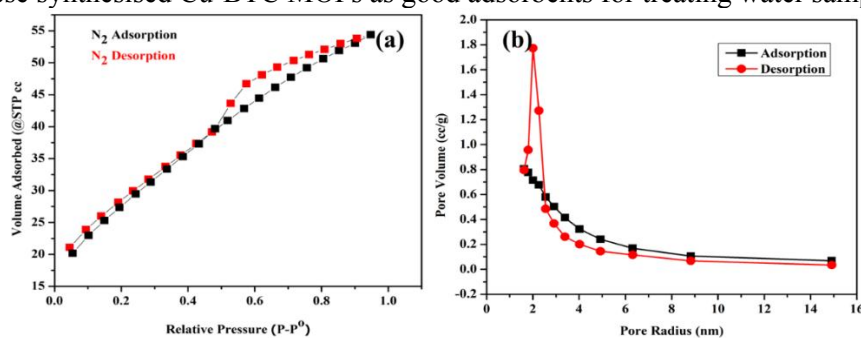


Fig.-2: (a) N₂ Sorption Isotherms and (b) Associated Pore Diameter Distribution of Cu-BTC MOFs

Impact of Different Parameters on Batch Studies

Impact of Solutions pH

The measured pH of the reaction medium is crucial during adsorption by controlling both the adsorbent's surface charge characteristics and the aqueous-phase metal ion speciation. As a result, a thorough investigation was conducted into how pH affected the Cu-BTC MOF's ability to adsorb both Cd (II) and Pb (II) ions. Fig.-3(a) illustrates that an upward trend in metal ion adsorption is observed as the pH rises from 1.0, reaching the highest values for Pb(II) and Cd(II) ions at pH 5.0 and 6.0, then exhibiting a slight

decline at higher pH values. From the results, pH 5.0 and 6.0 was identified as the optimal conditions and was subsequently selected for Pb (II) and Cd (II) species uptake studies. The enhanced concentration of hydronium ions at lower pH 2.0 causes significant surface protonation because they fiercely compete with metal ions at the surface-accessible binding sites of Cu-BTC MOFs.²³ This protonation shifts the surface charge from negative to positive, causing electrostatic repulsion of metal ions, leading to reduced adsorption efficiency. These findings demonstrate that controlling the solution pH is essential for maximising the adsorption efficiency of Cu-BTC MOFs.

Impact of MOF Adsorption Dose

Removal efficiencies of both Cd(II) and Pb(II) at variable adsorbent dosages are displayed in Fig.-3(b). The uptake ability was initially improved by increasing the adsorbent dosage because a larger dosage offers more active sites for metal ion binding. Accordingly, both metal ions showed progressively higher uptake ability as the adsorbent dosage increased and reached an optimum at 8 mg for the 25 mg/L metal ion concentration. This behaviour can be explained by more active centres accessible for adsorption. However, excess adsorbent loading resulted in reduced removal efficiency with time. At a fixed metal ion concentration, excess adsorbent rapidly captures the available ions in solution, and additional adsorbent does not contribute to further uptake. From the results, an adsorbent dosage of 8 mg per 25 mg/L was selected for subsequent experiments. Additionally, Pb (II) ions consistently showed higher removal efficiency than Cd (II) ions, which may be explained by the stronger interaction of Cu-BTC MOFs toward Pb (II) ions. This difference is associated with the distinct electronegativity and hydrated ionic radius of Cd (II) and Pb (II) species, which influence their affinity towards the adsorbent.

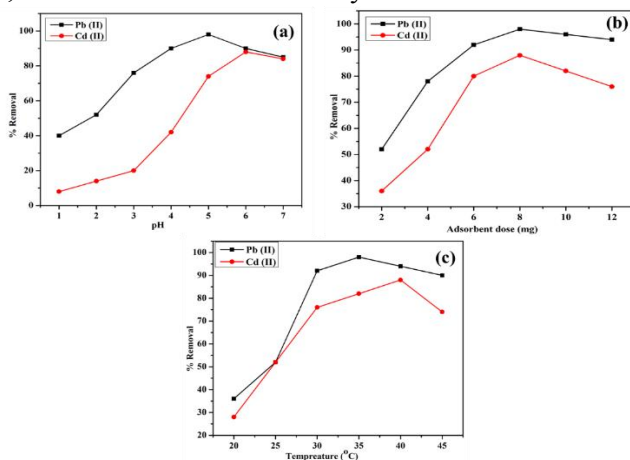


Fig.-3: Influence of (a) Solution pH, (b) MOF Dosage, and (c) Temperature on the Percentage Uptake of Cd(II) and Pb(II) Metal Ions using Cu-BTC MOFs

Influence of Temperature

Temperature effects on the uptake ability of Cu-BTC MOFs toward both metal ions were assessed under identical experimental conditions between 20 and 45 °C. Fig.-3(c) reveals that the adsorption capacities for the two metal ions increase with increasing temperature up to an optimum value, then a minor decrease slightly at higher temperatures. A substantial reduction in uptake capacity was detected when the temperature exceeded 40° C for Cd (II) and 35° C for Pb (II) ions. The obtained results shows 40° C and 35° C were the ideal temperatures for the adsorption ability of metal ions, and it also shows the good thermodynamic properties of the adsorbent.

Influence of Contact Time

Figure-4(a) illustrates how contact time affects both Cd (II) and Pb (II) ion uptake capacity employing Cu-BTC MOFs between 30 and 400 mins. Due to the numerous accessible adsorption sites on the surface of Cu-BTC, there is a noticeable rise in removal performance within the initial 100 min. The first phase of adsorption is primarily governed by rapid association of metal ions with accessible surface-active sites of the adsorbent. After 100 min, the adsorption rate gradually slows down, indicating progressive occupation of active sites and increased diffusion resistance. Equilibrium is reached after approximately

350-400 min, and afterwards the removal efficiency shows the negligible improvement. The increased adsorption efficiency of Pb (II) relative to Cd (II) is primarily associated with its stronger affinity for the functional groups embedded within the Cu-BTC MOFs, resulting in more effective coordination bonding. The short equilibrium time of the material reflects that adsorption is a quick process, hence making the material suitable for the use in water purification due to the quick removal of contaminants.

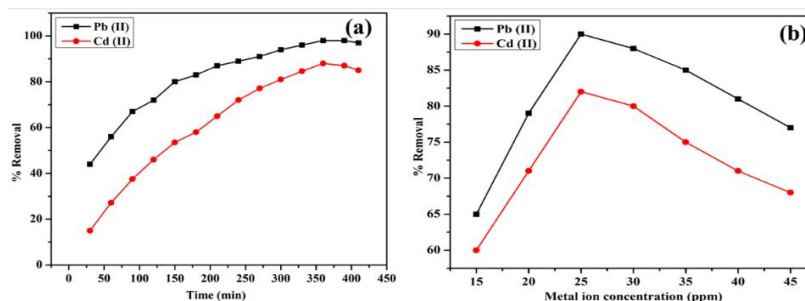


Fig.-4: Influence of (a) Contact Duration and (b) Starting Metal Ion Concentration on the Removal Percentage of Cd(II) and Pb(II) ions by Cu-BTC MOFs

Role of Initial Metal Ion Level

The metal ion uptake potential of the MOF was evaluated by changing the initial metal ion concentration in the range of 25–45 mg/L while maintaining a constant solution volume (100 mL), adjusted pH at optimised conditions, temperature (35° C for Pb (II) and 40° C for Cd (II)) and an 8 mg dosage of adsorbent. As shown in Fig.-4(b), from 15 to 25 mg/L, the concentration rose gradually, increasing the removal effectiveness until it reached 88% for Cadmium ions and 98% for lead ions. Cu-BTC MOF demonstrated the best removal efficiency for Pb (II) ions among the two metal ions, followed by Cd (II) ions at all concentrations.²⁴ The disparity in adsorption behaviour can be attributed to differences in metal–ligand affinity and ion-exchange interactions with MOF functional groups. Owing to the exhaustion of all existing active spots/sites, the metal uptake effectiveness decreased at increasing doses.

Study of Adsorption Isotherm

Analysing adsorption isotherm offers valuable insight into the interaction mechanisms occurring at the adsorbent-metal ion interface. Further, it also assists in evaluating adsorption performance under equilibrium conditions.²⁵ The adsorption equilibrium data for both metal ion removals were determined using Cu-BTC MOF at the optimum operating conditions as given in Fig.-5(a and b).

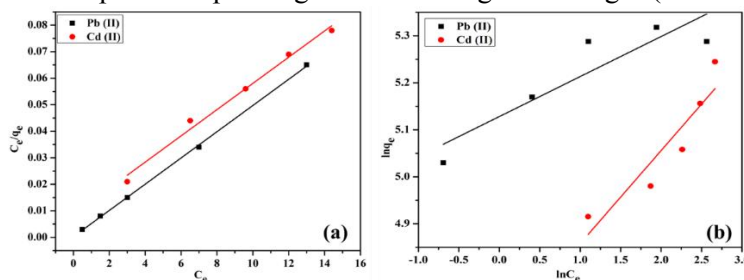


Fig.-5: Adsorption Isotherm Analysis for Metal Ions on Cu-BTC MOFs: (a) Langmuir and (b) Freundlich Approaches

Based on the Langmuir model, adsorption proceeds through a single adsorbed layer formed over a uniform surface with equivalent binding energies and no lateral interactions, while the Freundlich model incorporates surface heterogeneity and permits multilayer adsorption. The mathematical expression for the Langmuir adsorption isotherm is:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{bq_{max}} \quad (3)$$

In this model, the Langmuir adsorption constant is represented by b (L/mg), and the highest monolayer adsorption capacity as q_{max} (mg/g). Fig.-5(a) illustrates that a straight-line relationship is acquired for both

metal ions from plots of C_e/q_e versus C_e , confirming that the adsorption properties obey the Langmuir model. According to Eq. (4), the Freundlich adsorption relation is:

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_F \quad (4)$$

Where n denotes the adsorption intensity parameter, while the Freundlich constant is denoted by K_F (mg/g). Favourable adsorption is observed when the parameter $1/n$ is observed to be between 0.1-1. The isotherm parameters summarised in Table-1 demonstrate that the adsorption characteristics of both metal ions are more dominantly fitted by the Langmuir plot. According to this result, the adsorption process occurs as a single-layer coverage of metal ions on energetically equivalent adsorption sites. This suggests that the active sites on the Cu-BTC MOF surface possess relatively uniform adsorption energies, with negligible interactions between the adsorbed metal ions.

Table-1: Parameters of the Langmuir and Freundlich Models Describing Cd(II) and Pb(II) Adsorption onto Cu-BTC MOFs

S. No.	Metal ions	Langmuir adsorption constant			Freundlich adsorption constant		
		$q_{\max}$ (mg/g)	b (L/mg)	R^2	K_f (mg/g)	n	R^2
1.	Cd (II)	202	0.5763	0.98621	4.66007	5.05	0.81626
2.	Pb (II)	202	0.0018	0.99939	5.12817	11.7	0.76878

In comparison, the Freundlich adsorption plot exhibited comparatively weaker agreement with the observed findings, as reflected by a lower R^2 value. Although the calculated Freundlich parameters (K_f and n) indicate favourable adsorption characteristics, the relatively lower goodness of fit implies that this model does not adequately represent the adsorption mechanism in the present system.²⁶

Kinetic Study

Suitable kinetic models were employed to analyse the adsorption kinetics and elucidate the underlying mechanism. The behaviour of both ions on Cu-BTC MOFs was evaluated using PFO and PSO kinetic plots. PFO model is written as:

$$\ln(q_e - q_t) = \ln(q_e) - \frac{K_1}{2.303} t \quad (5)$$

PSO model equation is given by Eq. (6):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_t} \quad (6)$$

In these models, the PFO and PSO rate constants are denoted by k_1 and k_2 (min^{-1}). Figure-6(a and b) indicate that the PFO and PSO rate constants were derived from a linear fit of $\ln(q_e - q_t)$ against time and t/q_t versus time, using initial Cd(II) and Pb(II) concentrations in the range of 15-45 ppm. The adsorption behaviour of both metal ions on Cu-BTC MOFs was subsequently examined using PFO and PSO models to evaluate their suitability in describing the experimental data.

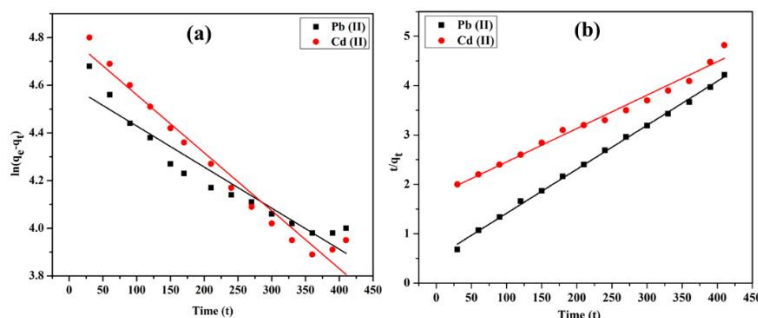


Fig.-6: Kinetic Modelling of Cd(II) and Pb(II) Adsorption on Cu-BTC MOFs using (a) PFO and (b) PSO Models

As evident from Table-2, the superior R^2 values exhibited by the PSO model for both Cd(II) and Pb(II) suggest that this model better captures the adsorption kinetics on Cu-BTC MOFs compared to the PFO model, supporting the conclusion that chemical adsorption is the principal mechanism.

The remarkable adsorption capacity of the prepared Cu-BTC MOFs shows their practical application as effective materials in treating spiked water samples. This study contributes to sustainable environmental remediation, meeting the objective of SDG-6.

Table-2: Kinetic Model Fitting Parameters for Cd(II) and Pb(II) Adsorption onto Cu-BTC MOFs with (a) PFO and (b) PSO Models

S. No.	Metal ions	PFO			PSO		
		K_1 (min ⁻¹)	R^2	q_e (mg/g)	K_2 (min ⁻¹)	R^2	q_e (mg/g)
1.	Cd (II)	0.00242	0.96128	121.8	2.57×10^{-5}	0.98258	147.7
2.	Pb (II)	0.00172	0.90932	99.5	1.52×10^{-5}	0.99812	112.1

CONCLUSION

The findings of this work demonstrate the effective synthesis of Cu-BTC MOFs through a hydrothermal approach and confirmed their crystalline nature, well-developed porosity, and large specific surface area through physicochemical characterisation. The synthesised Cu-BTC MOFs exhibited removal efficiencies of about 88% for Cd(II) and 98% for Pb(II) under optimised conditions. Within 300 minutes, the equilibrium was reached, suggesting a consistent uptake process and supporting the suitability of this material for wastewater treatment applications. Langmuir isotherm provided the best fit to the equilibrium data, suggesting monolayer adsorption on uniform binding sites and relatively high adsorption capacities. In addition, the kinetic analysis revealed PSO behaviour, implying that chemical adsorption governs the process. The overall results obtained show that Cu-BTC MOFs have good potential as adsorbents for water purification. Future studies on the regeneration of the adsorbent and its performance in wastewater are suggested to assess the feasibility of their application. The presented work is an effort toward sustainable technology for water purification in the context of SDG 6.

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CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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