

Tailoring MOF-303 with CaCl₂ and Graphite: A Promising Material for Atmospheric Water Capture

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Abstract

Atmospheric water harvesting (AWH) is a promising approach to address water scarcity in arid and low-humidity regions. In this study, MOF-303 was synthesized hydrothermally and modified with CaCl₂ and graphite by wet impregnation. Among samples containing 20–40 wt% CaCl₂, MC30-G2 (30 wt% CaCl₂, 2 wt% graphite) showed the best performance, with an adsorption capacity of 402.53 mg g⁻¹ and an adsorption percentage of 40.25%, nearly 94.8% higher than MC30-G0 (206.60 mg g⁻¹). FTIR, PXRD, SEM-EDS, BET, and AAS confirmed successful incorporation of the modifiers while preserving the MOF-303 framework. The composite had a surface area of 823.58 m² g⁻¹. Cycling tests showed adsorption capacity decreased from 495.911 mg g⁻¹ to 361.371 mg g⁻¹ after five cycles, while desorption efficiency remained above 87.99%. These results indicate that MOF-303/CaCl₂/graphite is a promising adsorbent for atmospheric water capture.

Keywords: Atmospheric Water Harvesting (AWH), Metal-Organic Frameworks (MOFs), MOF-303, Calcium Chloride (CaCl₂), Graphite

INTRODUCTION

Atmospheric water harvesting (AWH) has emerged as a promising approach for obtaining water directly from humid air and has attracted growing attention as an alternative to conventional water collection methods such as rainwater harvesting, groundwater extraction, and desalination (H. Liu et al., 2023; Ertop et al., 2023). The performance of AWH systems largely depends on the adsorbent material, which should combine high water uptake, suitable pore architecture, and stable adsorption–desorption behavior (Karim et al., 2025). Among the various porous materials investigated for this purpose, metal–organic frameworks (MOFs) have received considerable interest owing to their high surface area, tunable porosity, and adjustable surface chemistry (Zeng et al., 2024). MOFs are one of the fastest-growing classes of porous materials, offering high surface area, adjustable porosity, and excellent structural stability (Palapessy et al., 2025). Structurally, they consist of metal ions or clusters with vacant coordination sites connected to organic ligands that act as electron-pair donors (Pambudi et al., 2021). These features make MOFs particularly attractive for

moisture capture, especially under low-humidity conditions.

MOF-303 has been reported as a promising candidate for water harvesting applications because of its aluminum-based framework and hydrophilic pore environment. It is also regarded as a promising material for atmospheric water harvesting due to its one-dimensional hydrophilic channels, strong water uptake under low-humidity conditions, and good structural stability over 150 adsorption–desorption cycles (Fathieh et al., 2018; Arjmandi et al., 2026). Nevertheless, the practical use of pristine MOF-303 is still constrained by its hydrolytic stability and by performance decline during repeated adsorption–desorption operation (Alkhatib et al., 2024). In this regard, post-synthetic modification offers a feasible strategy to tailor adsorption sites and further improve water uptake capacity.

Modification with hygroscopic and conductive additives may offer a feasible strategy for improving material performance. In this context, calcium chloride (CaCl₂) can enhance water uptake due to its strong affinity for water molecules. At the same time, graphite may contribute to improved heat transfer and support the overall functionality of the composite material.

This is consistent with previous findings showing that CaCl₂/MOF-303 composites can achieve substantially higher water adsorption capacities (around 300 mg/g) than pristine MOF-303, although the loading amount must be optimized to avoid pore blocking and diffusion resistance (An et al., 2023; Li et al., 2025), while graphite may contribute to improved heat transfer and support the overall functionality of the composite material. Similar studies on carbon-based conductive additives in MOF-303 composites showed that such materials can enhance thermal transport and accelerate water release during regeneration, thereby improving cyclic atmospheric water harvesting performance (Fathieh et al., 2018; Song et al., 2023; Z. An et al., 2026). In general, introducing additional components or modulators into MOFs can effectively tailor their structural characteristics and adsorption behavior (Pambudi et al., 2021). The hydrothermal method is chosen for MOF synthesis due to its ability to produce high-quality crystals with good stability. By using high temperatures in a controlled solvent, this method enables the formation of optimal MOF structures, which are essential for applications such as gas storage and water harvesting (Zheng et al., 2023; Ali et al., 2024).

Studies on MOF-303/CaCl₂/graphite composites for AWH applications are still limited, particularly with respect to gravimetric adsorption capacity and cycling stability under controlled relative humidity and temperature conditions. Therefore, this study investigates the synthesis and characterization of a MOF-303/CaCl₂/graphite composite using the hydrothermal method as a candidate material for water harvesting. The performance of the composite was evaluated quantitatively through gravimetric adsorption measurements and repeated adsorption-desorption tests over five cycles under controlled relative humidity and temperature conditions.

METHODOLOGY

Materials and Instrumentals

The primary chemicals used in this study were aluminum chloride hexahydrate (AlCl₃ · 6H₂O, 98%), 1H-pyrazole-3,5-dicarboxylic acid monohydrate (H₂PZDC, 97%), sodium hydroxide (NaOH, 97%), and Ketoprofen (KTP, ≥98%), ethanol (CH₃CH₂OH, 99.8%), extra-fine graphite powder, and calcium chloride (CaCl₂, 96.0%) were supplied by Sigma-Aldrich. All reagents and solvents were used as

received from commercial suppliers without further purification.

The instruments used in this study included an oven (Mettler UN30), an analytical balance (OHAUS), and a pH meter (Mettler Toledo Seven Compact). MOF-303 characterization was performed using Powder X-ray Diffraction (PXRD, Rigaku MiniFlex 300), Fourier Transform Infrared Spectroscopy (FTIR, Shimadzu IR Prestige-21), Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS, SU3500), and Atomic Absorption Spectroscopy (AAS, Shimadzu AA-7000).

Methods

MOF-303 was synthesized using a hydrothermal method (Zheng et al., 2023) with slight modifications. H₂PZDC · H₂O (0.871 g, 5 mmol) and NaOH (0.600 g, 15 mmol) were dissolved in 15 mL of deionized water and stirred for 30 min. In a separate vessel, AlCl₃ · 6H₂O (1.207 g, 5 mmol) was dissolved in 5 mL of deionized water. The two solutions were then combined and stirred for 45 min. The mixture was transferred to a Duran bottle and heated at 100 °C for 24 h. After the reaction, the bottle was left in the oven and allowed to cool naturally to room temperature. The product was collected by centrifugation, washed with deionized water and ethanol (3-4 h), and then dried at 80 °C for 6 h. MOF-303 was obtained as a white powder with an isolated yield of 1.32 g (55.66%).

Before composite preparation, several adsorbent compositions were prepared to evaluate the effect of CaCl₂ and graphite contents on adsorption performance. The investigated samples consisted of MC20-G0, MC30-G0, and MC40-G0 containing 20 wt%, 30 wt%, and 40 wt% CaCl₂ without graphite addition, respectively. In addition, graphite-containing samples designated as MC20-G2, MC30-G2, and MC40-G2 were also prepared with 2 wt% graphite.

The MOF-303/CaCl₂ composite adsorbent was prepared using the wet impregnation method according to H. An et al., 2023. MOF-303 was first dispersed evenly in 10 mL of aqueous solution and stirred for 30 minutes. Subsequently, a predetermined amount of CaCl₂ was added, and the mixture was stirred for 12 hours to facilitate the penetration of calcium and chloride ions into the pores. The excess salt solution was then removed by centrifugation at 6000 rpm for 10 minutes, followed by rapid washing with distilled water to eliminate residual CaCl₂ on the external surface of the MOF.

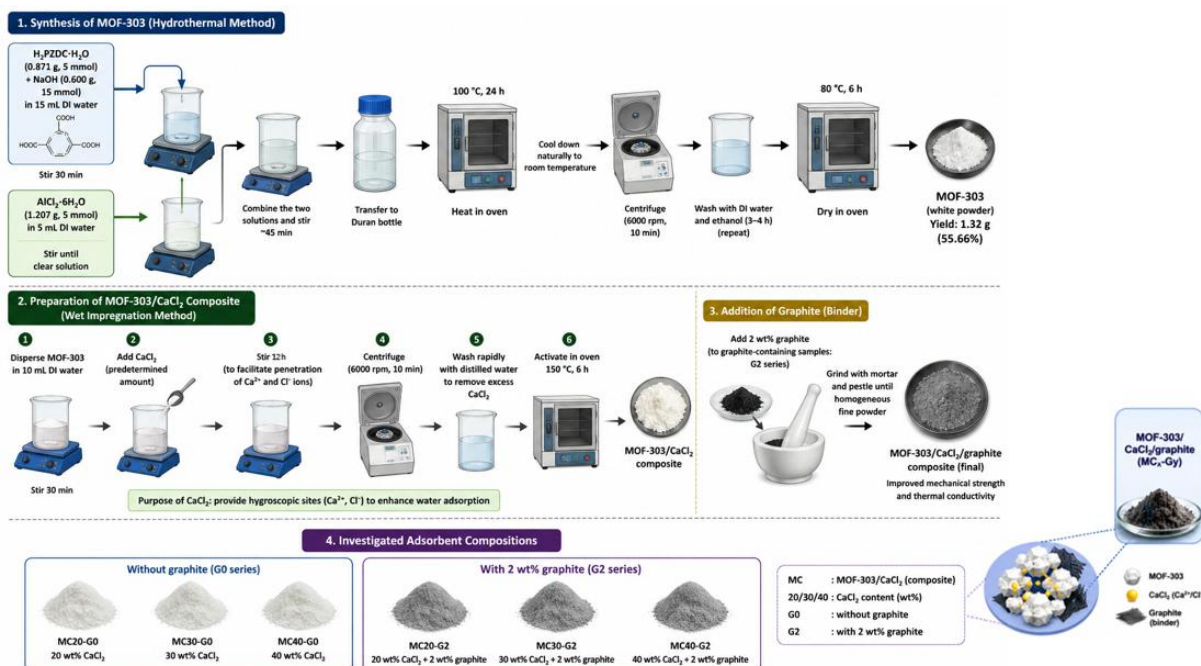


Figure 1. Synthesis and preparation of MOF-303/CaCl₂/graphite composites.

Finally, the MOF-303/CaCl₂ composite was activated at 150 °C for 6 hours in an oven. Graphite was added as a binder to improve the material's mechanical strength and thermal conductivity. The resulting mixture was then ground using a mortar and pestle until a homogeneous fine powder was obtained, as shown in Fig. 1. Further adsorption measurements were conducted.

RESULTS AND DISCUSSION

From the optimization results of several modified CaCl₂ and graphite compositions (Table 1), the sample containing 30 wt% CaCl₂ and 2 wt% graphite (MC30-G2) exhibited the highest adsorption performance, with an adsorption capacity of 402.53 mg/g and an adsorption percentage of 40.25%. This indicates that this composition provided the most effective balance between active adsorption sites and structural accessibility. Compared with the sample containing 30 wt% CaCl₂ without graphite (MC30-G0), the adsorption capacity increased from 206.60 mg/g to 402.53 mg/g, suggesting that graphite played an important role in enhancing the overall performance of the composite.

This improvement can be explained by the synergistic effect between MOF-303, CaCl₂, and graphite. MOF-303 is known for its hydrophilic channels, high structural stability, and suitability for water adsorption applications, according to Arjmandi et al. (2026). MOF-303 maintains good hydrolytic

stability and can undergo repeated adsorption–desorption cycles without significant structural degradation, making it a reliable framework for atmospheric water harvesting. In this study, the incorporation of CaCl₂ further increased the adsorption capacity because CaCl₂ is highly hygroscopic and can strongly interact with water molecules. As reported by Li et al. (2025), CaCl₂/MOF-303 composites benefit from a combination of physical adsorption inside the MOF pores, chemical hydration of CaCl₂, and interactions with the active sites of MOF-303. However, the amount of CaCl₂ must be optimized, since excessive loading may block the pores and reduce mass transfer. This explains why the 30 wt% CaCl₂ sample performed better than the 40 wt% CaCl₂ sample.

The addition of graphite also contributed to the improved performance. Graphite can enhance thermal conductivity, which helps accelerate heat transfer during adsorption and regeneration. Conductive carbon-based additives can improve desorption kinetics and support more efficient cyclic performance in MOF-based water harvesting materials (Abu El-Maaty et al., 2024; Z. An et al., 2026). In this work, the presence of 2 wt% graphite likely improved heat distribution within the composite, allowing water molecules to be adsorbed and released more effectively. As a result, MC30-G2 showed a much higher adsorption capacity than the samples without graphite.

Table 1. Optimization of CaCl₂ and Graphite Composition on the Adsorption Performance

Materials	MOF-303 (wt%)	CaCl ₂ (wt%)	Graphite (wt%)	Adsorption Capacities (mg/g)	Percent Adsorption (%)
MC20-G0	80	20	0	7.47	0.75
MC30-G0	70	30	0	206.60	20.66
MC40-G0	60	40	0	177.00	17.70
MC20-G2	78	20	2	187.47	18.75
MC30-G2	68	30	2	402.53	40.25
MC40-G2	58	40	2	280.67	28.07

Before modification, the FTIR spectrum of MOF-303 (Fig. 2) displayed the characteristic bands of the pyrazole-based framework. The peaks at 1001 cm⁻¹, 1478 cm⁻¹, and 1529 cm⁻¹ were assigned to =N–NH, C–C, and C=N vibrations, respectively, in agreement with previous reports on Li et al. (2025). These signals confirm the expected vibrational features of the pyrazole ligand. In addition, the bands at 1605.9 cm⁻¹ and 1396.2 cm⁻¹ are consistent with carboxylate-related vibrations, supporting the coordination between the organic linker and Al³⁺ centers. Wang et al. (2026) reported similar FTIR features as this study for MOF-303, with bands at 1080, 1310, and 1608 cm⁻¹ attributed to C–O, C–N, and C=O stretching vibrations.

After modification with CaCl₂, the carboxylate stretching band shifted from 1572 cm⁻¹ to 1556 cm⁻¹. This shift suggests a change in the local coordination environment and may indicate an interaction between Ca²⁺ species and oxygen-containing groups on the MOF surface, in line with previous observations of Ca-modified MOFs (Li et al., 2025; H. An et al., 2023). A slight change was also observed in the O–H stretching region (3370 cm⁻¹), which may reflect additional surface interactions after calcium treatment. In the graphite-modified sample, a weak band appeared around 1580 cm⁻¹, which is characteristic of the C=C stretch from the aromatic structure of graphite. While graphite itself is typically less active in the IR region, this band suggests that graphite is interacting with the MOF, possibly through π – π interactions or other surface phenomena. This is consistent with the previous work (Fathieh et al., 2018; Song et al., 2023), which demonstrated that the incorporation of carbon-based materials into MOFs resulted in the formation of new surface interactions and functional group modifications. This confirms that the graphite incorporation modifies the MOF-303 structure, likely enhancing its properties.

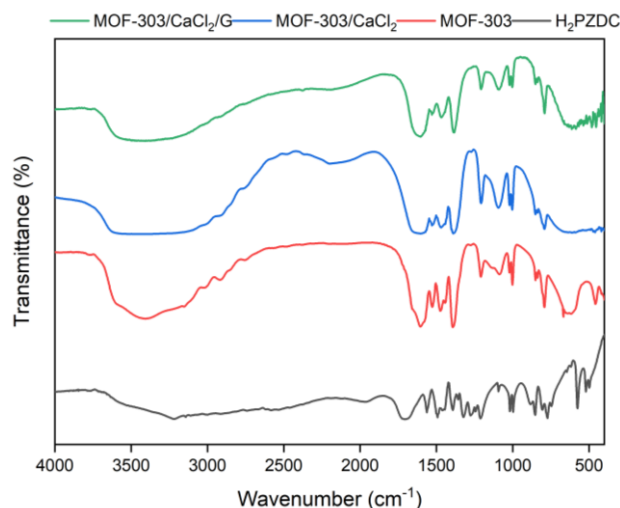


Figure 2. Infrared spectrum

These modifications enhance the material's potential for applications such as water harvesting, where the interactions between functional groups and the added materials can significantly improve adsorption and retention properties, as indicated in similar research on water harvesting technologies using MOFs (Song et al., 2023; Fathieh et al., 2018).

The PXRD patterns (Fig. 3) confirmed the crystalline structure of MOF-303, displaying sharp and well-defined peaks at 2θ values of 8.76°, 12.24°, 14.63°, and 17.58°, consistent with the simulated data from CCDC-2078719, and in accordance with several existing literatures (Hanikel et al., 2019; Chen et al., 2024). After activation and modification to form a composite (MOF-303/CaCl₂ and MOF-303/CaCl₂/G), the diffraction pattern remained stable, indicating that the structural integrity of the framework was maintained and remained intact despite the incorporation of external components.

However, several modifications in the diffraction pattern are notable. In the MOF-303/CaCl₂ sample, new peaks emerge at around $2\theta \approx 10.5^\circ$ and 16.4° , which are not present in the pristine MOF. These can be attributed to the presence of CaCl₂ or its hydrated form (CaCl₂ · 2H₂O), suggesting that Ca²⁺ ions have successfully interacted with or coordinated to the MOF structure. In the sample modified with graphite, particularly MOF-303/CaCl₂/graphite, an additional diffraction peak appears around $2\theta \approx 27.5^\circ$, corresponding to the (002) plane of graphitic carbon. This serves as clear evidence of successful graphite incorporation into the composite system.

A general decrease in peak intensity is also observed in the modified samples, especially after the addition of both CaCl₂ and graphite. This reduction in intensity may suggest a slight decrease in crystallinity,

which is commonly reported when external agents disrupt the uniform packing of MOF crystals or when guest molecules partially occupy the pores. Despite these changes, the consistent presence of the original MOF-303 peaks confirms that the fundamental structure is preserved, while the appearance of new peaks affirms the successful incorporation of modifying agents.

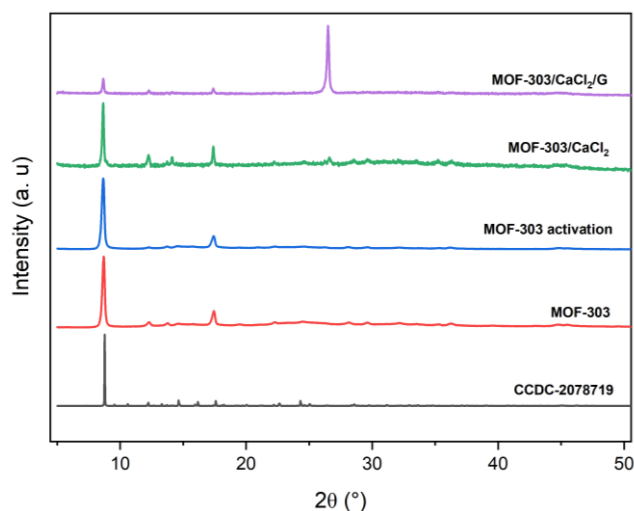


Figure 3. PXRD pattern

The PXRD patterns indicate that the MOF-303 framework remained intact after modification. This is consistent with previous reports on $\text{CaCl}_2/\text{MOF-303}$ composites and graphite-bonded MOF-303 (Song et al., 2023; H. An et al., 2023; Li et al., 2025), which showed that these additives can be incorporated without destroying the MOF-303 structure while improving practical performance.

The nitrogen adsorption–desorption isotherm of the MOF-303/ CaCl_2 /graphite composite (Fig. 4) shows a rapid uptake at low relative pressure, followed by a gradual increase and a sharp rise near $p/p_0 \approx 0.9$ – 1.0 , together with a clear hysteresis loop. This profile indicates that the material contains both microporous regions, where water molecules are initially adsorbed, and mesoporous or interparticle voids, where capillary condensation occurs at higher relative pressure. The BET analysis yielded a specific surface area of $823.58 \text{ m}^2 \text{ g}^{-1}$, a total pore volume of $1.1056 \text{ cm}^3 \text{ g}^{-1}$, and an average pore diameter of 2.6848 nm , confirming that the composite still retains a highly porous structure after modification. From a textural standpoint, this value is higher than that reported by Li et al. (2025) for $\text{CaCl}_2/\text{MOF-303}$, where the surface area decreased to 588 – $683 \text{ m}^2 \text{ g}^{-1}$ from $1276 \text{ m}^2 \text{ g}^{-1}$, suggesting that the addition of a small amount of graphite in the present work may help preserve the accessible pore structure while maintaining a large number of adsorption sites.

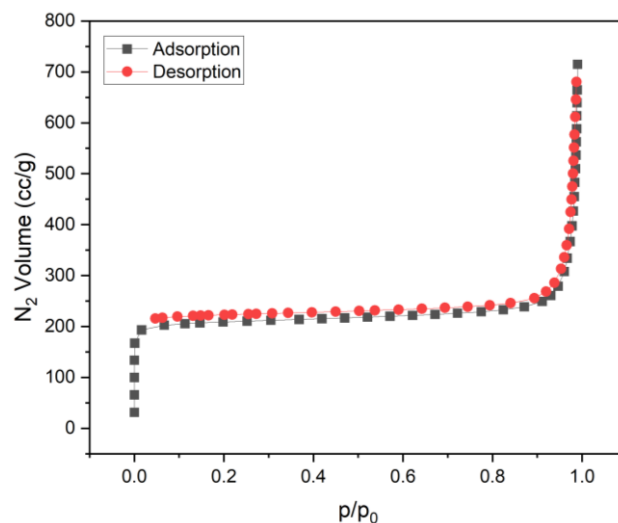


Figure 4. Nitrogen adsorption curves of MOF-303/ CaCl_2 /graphite composite

The SEM analysis of the MOF-303 samples (Fig. 3) reveals significant changes upon modification with CaCl_2 and graphite, highlighting their potential for atmospheric water capture. Initially, the unmodified MOF-303 (Fig. 3a) shows a well-formed porous structure with a rhombic shape. The uniform size distribution and moderate aggregation of the crystals suggest an organized crystalline framework that could facilitate water adsorption. The corresponding EDS spectrum (Fig. 3b) confirms the presence of aluminum (Al) and chloride (Cl), with aluminum making up 23.57 wt% and chloride 76.43 wt%, indicating that the material is a typical MOF structure composed of aluminum and organic linkers.

After modification with CaCl_2 (Fig. 3c), the SEM image demonstrates a slightly altered morphology with a more compact structure. The EDS spectrum (Fig. 3d) reveals the presence of calcium (Ca) along with aluminum and chloride, with calcium comprising 33.15 wt%. This modification introduces a potential improvement in the material's water adsorption capacity due to the ability of calcium to interact with water molecules through ionic bonds, enhancing moisture uptake. Additionally, the introduction of sodium (Na) is observed at a small fraction (0.64 wt%), which may result from residual NaCl in the synthesis process.

These modifications— CaCl_2 and graphite—alter the morphology and elemental composition of MOF-303, making it a promising candidate for atmospheric water capture (AWC) applications.

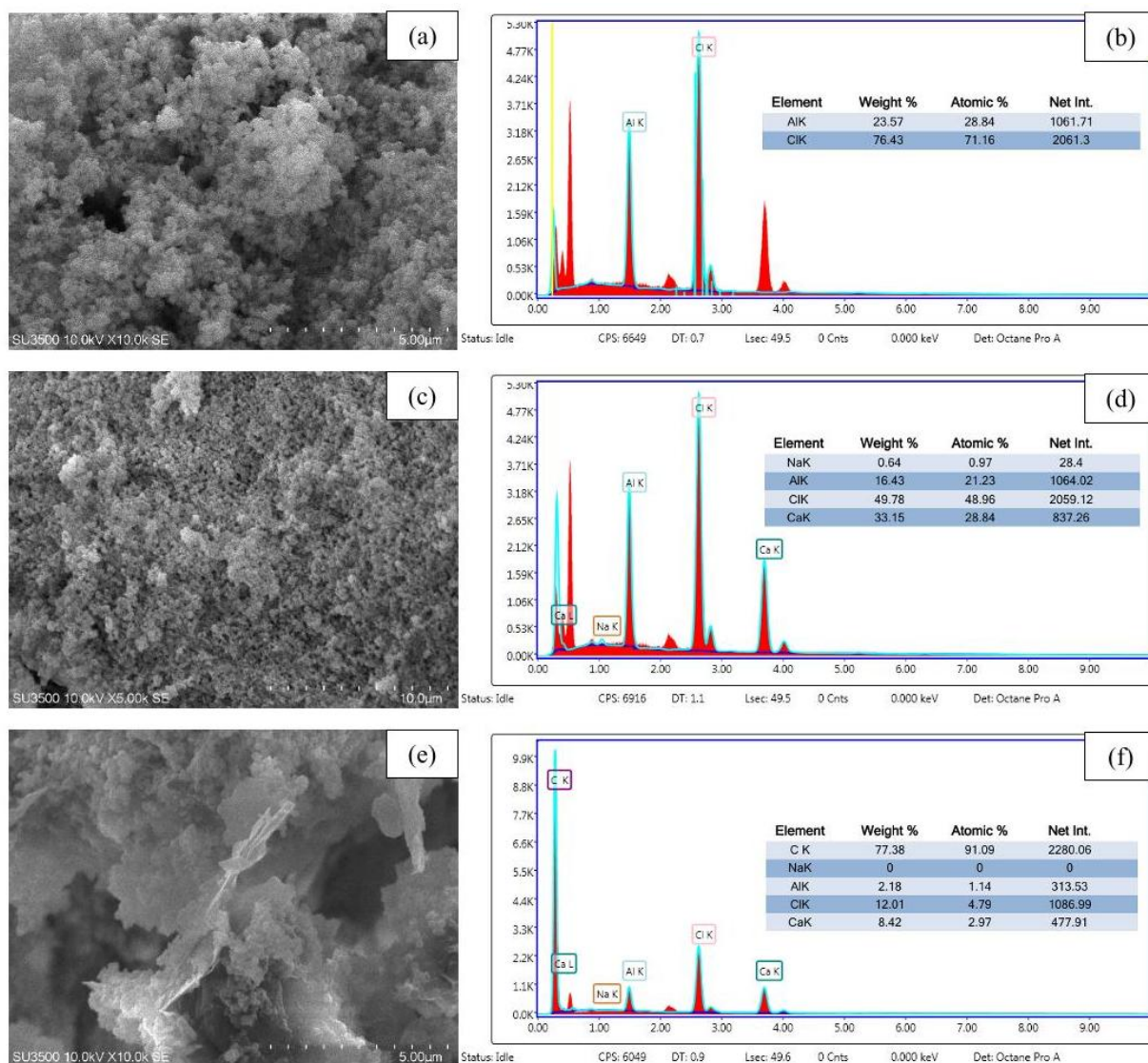


Figure 5. SEM-EDX analysis of (a), (b) MOF-303; (c), (d) MOF-303/CaCl₂; (e), (f) MOF-303/CaCl₂/G

The improved water adsorption capacity, combined with the structural stability provided by graphite and calcium, enhances the material's potential for water harvesting in arid environments. MOFs are well-known for their ability to adsorb large quantities of water from humid air, and the modification of MOF-303 further strengthens this characteristic, possibly improving efficiency and scalability for real-world applications.

The AAS analysis was performed to quantify the actual calcium content in the MOF-303 composite and to verify the incorporation of CaCl₂ into the material. The calcium content in the modified samples was found to be approximately 19.71%, 29.87%, 38.99%, 19.92%, 29.95%, and 38.68% for MC20-G0, MC30-

G0, MC40-G0, MC20-G2, MC30-G2, and MC40-G2, respectively, which is nearly consistent with the intended Ca loading in each sample. To support this quantification, the AAS measurement was calibrated using a standard curve that showed a strong linear relationship between calcium concentration and absorbance, with an R² value of 0.9936. The calibration equation, $y = 0.0053x - 0.0017$ (Fig. 6), was used as the basis for determining the calcium content in the composite.

The combined adsorption-desorption profile over five cycles indicates that the MOF-303/CaCl₂/graphite composite retained a relatively high water uptake capacity and good regenerability.

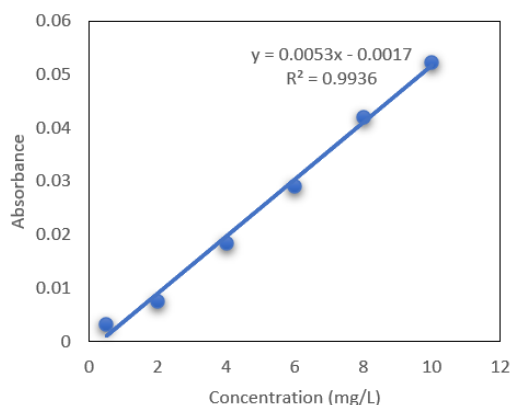


Figure 6. Calibration curve

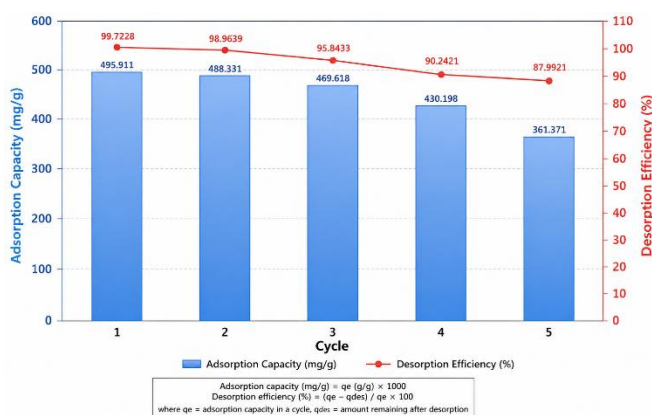


Figure 7. Adsorption Capacity and Desorption Efficiency Over 5 Cycles

However, a gradual decline was observed with repeated use. The adsorption capacity decreased from 495.911 mg g⁻¹ in cycle 1 to 361.371 mg g⁻¹ in cycle 5, corresponding to a reduction of approximately 27.1%. This decrease suggests that repeated adsorption-desorption treatment may have caused partial loss of active sites, incomplete regeneration, or minor structural changes in the composite. In parallel, the desorption efficiency remained high, decreasing only from 99.72% in cycle 1 to 87.99% in cycle 5. This result indicates that most of the adsorbed water could still be removed during the regeneration step, demonstrating that the material maintained good recyclability. Although the adsorption capacity gradually declined, the overall high desorption efficiency supports the potential of the composite as a reusable candidate for atmospheric water harvesting, particularly in preliminary application studies.

CONCLUSION

This study demonstrated that modifying MOF-303 with CaCl₂ and graphite can significantly improve its performance as an atmospheric water harvesting

material. The optimized composite, MC30-G2, showed the highest adsorption capacity of 402.53 mg g⁻¹ with an adsorption percentage of 40.25%, representing an improvement of nearly 94.8% compared with MC30-G0 (206.60 mg g⁻¹). The addition of graphite also contributed to better structural accessibility, as supported by the BET result of 823.58 m² g⁻¹ specific surface area, 1.1056 cm³ g⁻¹ total pore volume, and 2.6848 nm average pore diameter. AAS analysis confirmed the presence of calcium in the modified samples, with Ca loading values of 19.71%, 29.87%, 38.99%, 19.92%, 29.95%, and 38.68% for MC20-G0, MC30-G0, MC40-G0, MC20-G2, MC30-G2, and MC40-G2, respectively. In addition, the composite maintained good cycling performance, with adsorption capacity decreasing from 495.911 mg g⁻¹ to 361.371 mg g⁻¹ over five cycles, while desorption efficiency remained relatively high. Overall, the results confirm that CaCl₂ and graphite modification is an effective strategy to enhance the adsorption properties and practical potential of MOF-303 for atmospheric water harvesting.

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