



Morphological and Elemental Characterization of a Mn-Based Metal-Organic Framework Synthesized from Sodium tartrate and Manganese Chloride

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Annotatsiya

Metall-organik karkaslar (MOFlar) o'zining noyob strukturaviy xususiyatlari va kataliz, adsorbsiya hamda elektrokimyoviy tizimlardagi keng qo'llanilishi sababli katta qiziqish uyg'otayotgan g'ovak materiallar sinfiga kiradi. Ushbu ishda natriy-kaliy tartrat va marganets xloriddan sintez qilingan marganets asosidagi metall-organik karkasning morfologiyasi va element tarkibi skanerlovchi elektron mikroskopiya (SEM) hamda energiya-dispersiv rentgen spektroskopiyasi (EDS) usullari yordamida o'rganildi. SEM tahlili g'ovak nanohajmli zarrachalar, kristall domenlar va yirik aglomeratlardan tashkil topgan geterogen tuzilmalarning hosil bo'lganligini ko'rsatdi. Kuzatilgan morfologiya yuqori sirt maydoni va murakkab sirt arxitekturasining mavjudligini ko'rsatadi. EDS tahlili material tarkibida uglerod, kislorod, natriy, kaliy, xlor va marganets elementlari mavjudligini tasdiqladi. Miqdoriy element tahlili uglerod (37,80 massa %) va kislorod (36,94 massa %) asosiy tarkibiy qismlar ekanligini, marganets esa (9,51 massa %) asosiy metall komponent sifatida ishtirok etishini ko'rsatdi. Elementlar xaritalanishi marganetsning karkas tuzilmasi bo'ylab nisbatan bir tekis taqsimlanganligini namoyish etdi. Olingan natijalar marganets saqlovchi metall-organik karkasning muvaffaqiyatli hosil bo'lganligini hamda uning katalitik va elektrokimyoviy tizimlarda qo'llash istiqboliga ega ekanligini tasdiqlaydi.

Аннотация

Металлоорганические каркасы (MOF) представляют собой важный класс пористых материалов, привлекающих значительное внимание благодаря своим уникальным структурным свойствам и широкому спектру применений в катализе, адсорбции и электрохимических системах. В данной работе морфология и элементный состав металлоорганического каркаса на основе марганца, синтезированного из натрий-калиевого тартрата и хлорида марганца, были исследованы с использованием сканирующей электронной микроскопии (SEM) и энергодисперсионной рентгеновской спектроскопии (EDS). Анализ SEM показал формирование гетерогенных структур, состоящих из пористых наночастиц, кристаллических доменов и более крупных агломератов. Наблюдаемая морфология свидетельствует о наличии высокой удельной поверхности и сложной архитектуры поверхности. Анализ EDS подтвердил присутствие в материале углерода,



кислорода, натрия, калия, хлора и марганца. Количественный элементный анализ показал, что углерод (37,80 мас. %) и кислород (36,94 мас. %) являются преобладающими компонентами, тогда как марганец (9,51 мас. %) выступает в качестве основного металлического компонента. Элементное картирование продемонстрировало относительно равномерное распределение марганца по всей структуре каркаса. Полученные результаты подтверждают успешное формирование марганецсодержащего металлоорганического каркаса с потенциальными возможностями применения в каталитических и электрохимических системах.

Abstract

Metal-organic frameworks (MOFs) are an important class of porous materials that attract significant attention due to their unique structural properties and wide range of applications in catalysis, adsorption, and electrochemical systems. In this work, the morphology and elemental composition of a manganese-based metal-organic framework synthesized from Sodium tartrate and manganese chloride were investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). SEM analysis revealed the formation of heterogeneous structures consisting of porous nanosized particles, crystalline domains, and larger agglomerates. The observed morphology suggests the presence of a high surface area and complex surface architecture. EDS analysis confirmed the presence of carbon, oxygen, sodium, potassium, chlorine, and manganese in the material. Quantitative elemental analysis demonstrated that carbon (37.80 wt.%) and oxygen (36.94 wt.%) are the dominant constituents, while manganese (9.51 wt.%) acts as the principal metallic component. Elemental mapping showed a relatively homogeneous distribution of manganese throughout the framework structure. The obtained results confirm the successful formation of a manganese-containing metal-organic framework with potential applications in catalytic and electrochemical systems.

Keywords: Metal-organic framework, manganese chloride, Sodium tartrate, SEM, EDS, elemental mapping, manganese MOF.

Introduction

Metal-organic frameworks (MOFs) are crystalline porous materials formed through the coordination of metal ions with multifunctional organic ligands. Due to their exceptionally high surface area, tunable pore structures, and versatile chemical properties, MOFs have become one of the most extensively studied groups of advanced materials. These materials have found applications in gas storage, adsorption, catalysis, chemical sensing, environmental remediation, and energy conversion technologies. The properties of MOFs strongly depend on the nature of both the metal centers and the organic ligands used during synthesis. Transition metals are particularly attractive because of their ability to form stable coordination structures and provide catalytic activity. Among them, manganese is of special interest due to its environmental compatibility, low cost, abundance, and multiple oxidation states. Tartrate-containing ligands possess hydroxyl and carboxyl functional groups capable of coordinating with transition metal ions. Such ligands can facilitate the formation of extended coordination networks and contribute to the development of porous structures. Understanding the morphology and elemental composition of newly synthesized MOFs is essential for evaluating their structural properties and potential practical applications. Scanning electron microscopy (SEM) is commonly used to investigate surface morphology, particle size, and structural organization, whereas energy-dispersive X-ray spectroscopy (EDS) provides information about elemental composition and spatial distribution. Therefore, SEM and EDS analyses were



employed in this study to characterize a manganese-containing MOF obtained from Sodium tartrate and manganese chloride.

Synthesis of Mn-Based Metal-Organic Framework

A manganese-based metal-organic framework (Mn-MOF) was synthesized by a slow crystallization method using manganese chloride tetrahydrate and potassium tartrate as precursors. Briefly, 0.099g (0.5 mmol) of manganese chloride tetrahydrate ($\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$) was dissolved in 10 mL of distilled water. In a separate vessel, 0.113g (0.5 mmol) of potassium tartrate was dissolved in 10 ml of distilled water under continuous magnetic stirring at 50 °C until complete dissolution was achieved. The two clear solutions were subsequently mixed and stirred for several minutes to ensure homogeneous distribution of the reactants. The resulting reaction mixture was then transferred to a thermostatic chamber and maintained at 30 °C for 14 days to promote slow crystallization and framework formation. After completion of the crystallization process, the obtained crystals were collected and washed several times with distilled water and ethanol to remove unreacted precursors and soluble impurities. The purified crystals were dried in an oven at 60 °C until constant weight was reached.

The final crystalline product was obtained with a yield of approximately 80%.

Results and Discussion

SEM Analysis

The SEM images of the synthesized Mn-based MOF at different magnifications are presented in Figure 1. The micrographs reveal a heterogeneous morphology consisting of nanosized particles, crystalline domains, and larger agglomerated structures. The observed morphology indicates that the material is composed of structural units with different dimensions and shapes, resulting in a non-uniform surface architecture. At 3000^x magnification (5 μm scale), the material exhibits a porous morphology composed of fine particles distributed throughout the surface. The presence of pores and nanosized particles indicates the formation of a highly developed microstructure. The particles appear closely packed while maintaining visible voids between neighboring grains, which contributes to the porous nature of the material. Such morphology suggests that the crystallization process resulted in the formation of small interconnected particles rather than dense compact structures.

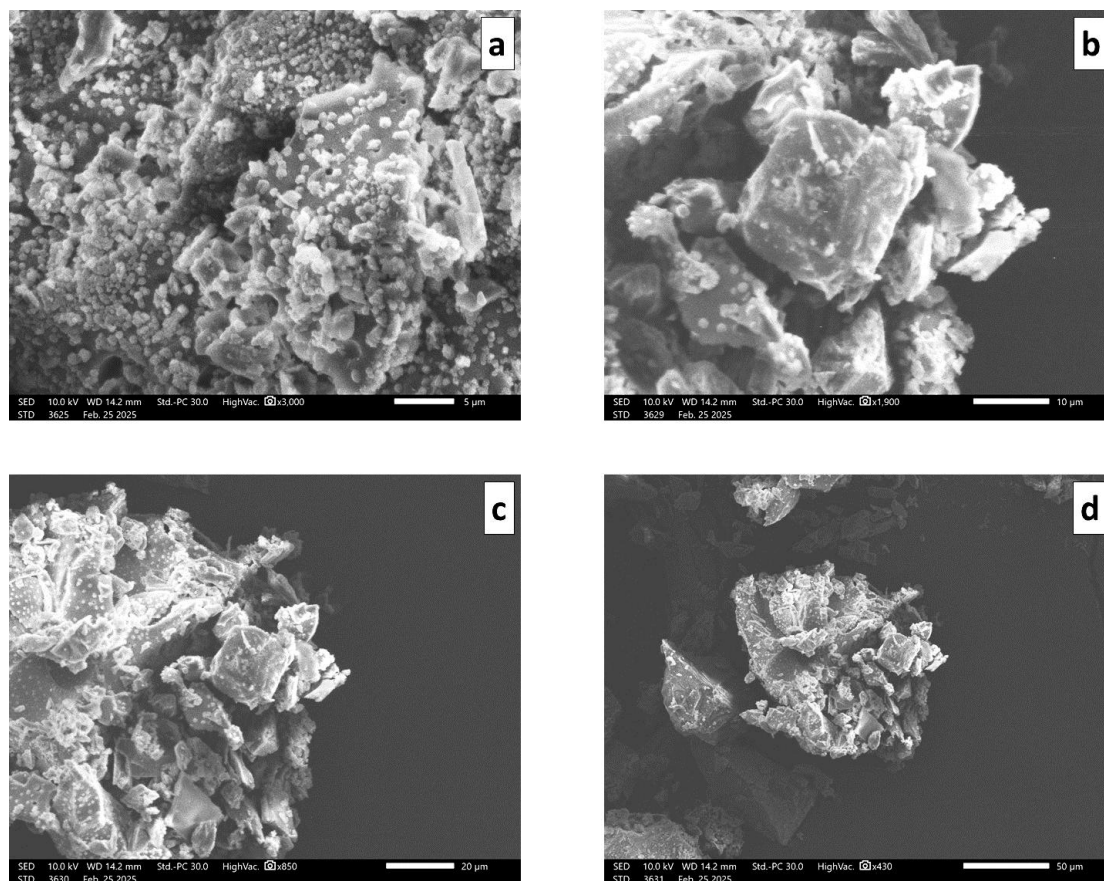


Figure 1. SEM images of the MOF formed based on Sodium tartrate and MnCl_2 salt at different scales: a) 5 μm , b) 10 μm , c) 20 μm , d) 50 μm .

At 1900 $\times$ magnification (10 μm scale), larger crystalline structures are observed with smaller particles dispersed on their surfaces. This morphology suggests the coexistence of particles with different sizes and shapes within the material. The crystal-like domains are irregularly distributed and surrounded by finer particles, indicating a heterogeneous growth process during material formation. The attachment of nanosized particles onto larger crystals further contributes to the complexity of the surface morphology. Images obtained at lower magnifications (850 $\times$ and 430 $\times$) corresponding to scales of 20 and 50 μm reveal large aggregates formed from smaller primary particles. The aggregates exhibit irregular geometries and rough surface textures, indicating a complex structural organization. The formation of these agglomerates may be associated with particle-particle interactions occurring during synthesis and subsequent drying of the material. Despite aggregation, individual structural components remain distinguishable within the larger clusters.

EDS Analysis

The EDS spectrum shown in Figure 2 was used to determine the elemental composition of the synthesized material. Strong peaks corresponding to carbon (C-K, ~ 0.28 keV) and oxygen (O-K, ~ 0.53 keV) were observed in the spectrum. The dominance of these signals is consistent with the presence of an organic ligand-based structure within the material.

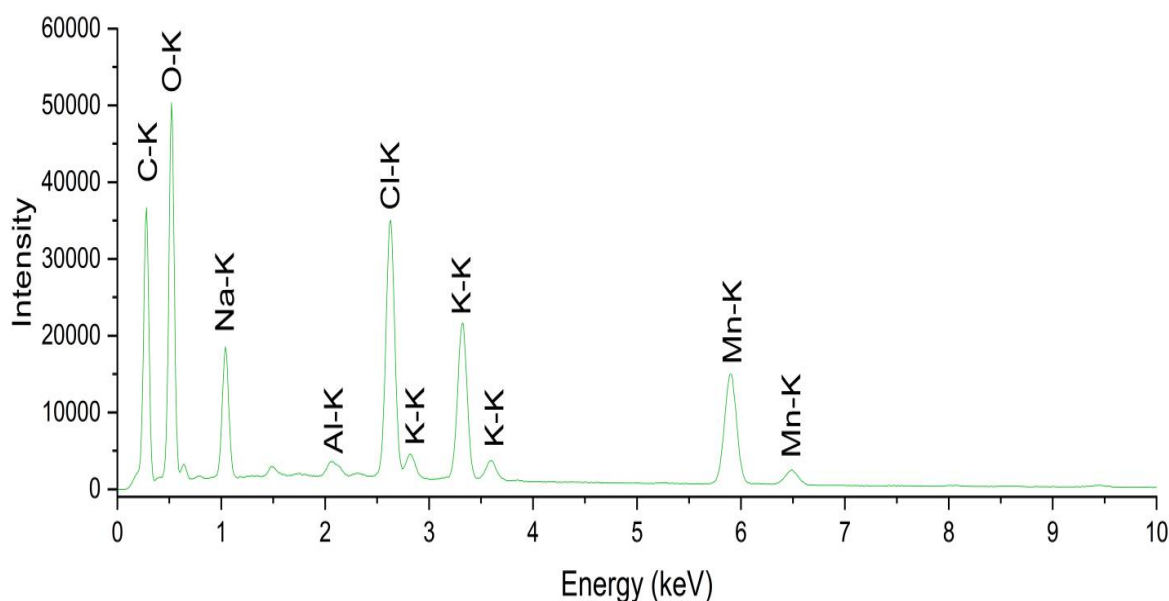


Figure 2. Elemental content of MOFs formed based on MnCl_2 salt with Sodium tartrate

Additional peaks corresponding to sodium (Na-K, ~ 1.04 keV), chlorine (Cl-K, ~ 2.62 keV), and potassium (K-K, ~ 3.31 - 3.59 keV) were also detected. The presence of these elements may be associated with residual precursor species remaining after the synthesis process. Prominent manganese peaks were observed at approximately 5.90 and 6.49 keV. These peaks indicate that manganese is one of the major constituents of the synthesized material and confirm its presence within the obtained product. A weak aluminum signal was also detected in the spectrum. Since aluminum was not intentionally introduced during synthesis, its presence may originate from external contamination, sample preparation procedures, the sample holder, or trace impurities from laboratory materials.

Quantitative Elemental Composition

Carbon and oxygen constitute approximately 75% of the total elemental composition, indicating that these elements are the major constituents of the synthesized material. The manganese content was determined to be 9.51 wt.%, confirming the presence of manganese as one of the principal metallic elements in the sample. Sodium, potassium, and chlorine were detected in moderate concentrations and may be associated with residual precursor species remaining after the synthesis process. The low aluminum content (0.17 wt.%) suggests that this element is present only in trace amounts and is unlikely to contribute significantly to the overall composition of the material.

Table 1.

Results of elemental analysis of MOF, formed based on MnCl_2 salt with Sodium tartrate

Element	Line	Mass%	Atom%
C	K	37.80 ± 0.07	51.34 ± 0.10
O	K	36.94 ± 0.11	37.67 ± 0.11
Na	K	4.49 ± 0.03	3.19 ± 0.02
Al	K	0.17 ± 0.00	0.10 ± 0.00
Cl	K	5.95 ± 0.02	2.74 ± 0.01
K	K	5.15 ± 0.02	2.15 ± 0.01
Mn	K	9.51 ± 0.04	2.82 ± 0.01
Total		100.00	100.00

Elemental Mapping Analysis

Figure 3 presents the SEM image and corresponding EDS elemental mapping results of the synthesized material. Elemental mapping was performed to evaluate the spatial distribution of the detected elements and to assess the compositional homogeneity of the sample. The carbon (C-K) and oxygen (O-K) maps shown in Figures 3b and 3c exhibit widespread distribution throughout the analyzed region. The relatively continuous distribution of these elements indicates that carbon and oxygen are major constituents of the material and are present across most particles observed in the SEM image. The similarity between the carbon and oxygen distributions suggests their association within the same structural phase. The sodium (Na-K) map (Figure 3d) shows a lower signal intensity compared with carbon and oxygen. Sodium appears in localized regions of the sample, indicating that it is present in smaller quantities. Likewise, the aluminum (Al-K) map (Figure 3e) exhibits only weak and scattered signals. The very low concentration and irregular distribution of aluminum suggest that it is present only as a trace component and may originate from external sources or sample preparation procedures.

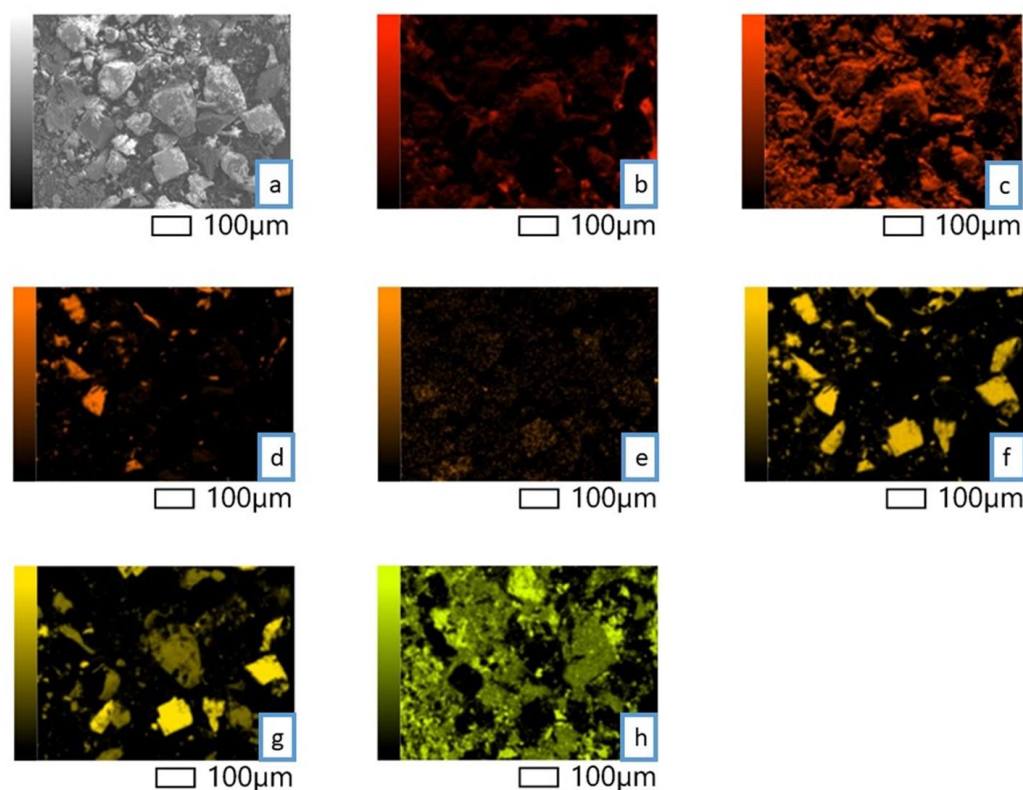


Figure 3. SEM and EDS mapping analysis of MOFs formed based on Sodium tartrate and MnCl_2 a) 100 μm sample; b) C-K (Carbon); c) O-K (Oxygen); d) Na-K (Sodium); e) Al-K (Aluminum); f) Cl-K (Chlorine); g) K-K (Potassium); h) Mn-K (Manganese).

Figures 3f and 3g show the distribution of chlorine (Cl-K) and potassium (K-K), respectively. These elements are detected in several distinct particles and localized regions throughout the sample. Their distribution is less uniform than that of carbon and oxygen, which may be associated with residual precursor species remaining after synthesis. The presence of chlorine and potassium in similar regions suggests a possible relationship between these elements within the material. The manganese (Mn-K) mapping image presented in Figure 3h reveals the

presence of manganese throughout a large portion of the analyzed area. The Mn signal is observed across numerous particles, indicating that manganese is widely distributed within the synthesized material. This observation is consistent with the EDS spectrum and quantitative elemental analysis, which identified manganese as one of the major metallic constituents of the sample.

Conclusion

A manganese-containing material synthesized from Sodium tartrate and manganese chloride was characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS).

SEM analysis revealed a heterogeneous morphology consisting of porous regions, nanosized particles, crystalline domains, and larger agglomerated structures. The observed microstructure demonstrates the presence of particles with different sizes and shapes distributed throughout the sample, indicating a complex surface morphology.

EDS analysis identified carbon, oxygen, sodium, potassium, chlorine, aluminum, and manganese as the constituent elements of the material. Quantitative elemental analysis showed that carbon and oxygen were the dominant components, accounting for approximately 75% of the total composition, while manganese was detected as the major metallic element with a concentration of 9.51 wt.%.

Elemental mapping further demonstrated the distribution of carbon, oxygen, manganese, sodium, potassium, chlorine, and aluminum within the analyzed region. Carbon, oxygen, and manganese were observed throughout a large portion of the sample, whereas sodium, potassium, chlorine, and aluminum exhibited lower concentrations and more localized distributions.

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