

Research Article

Design of iron(II) molybdate/sodium molybdate nanocomposites as advanced anode materials for lithium-ion batteries

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ABSTRACT

Iron(II) molybdate/sodium molybdate ($\text{FeMoO}_4/\text{Na}_2\text{MoO}_4$) nanocomposites are synthesized using a simple fabrication process and investigated for the first time as anode materials for lithium-ion batteries. Structural and morphological analyses reveal the coexistence of FeMoO_4 and Na_2MoO_4 domains within nanosheet-based architectures containing both semi-crystalline and amorphous regions. By controlling the reaction time, the phase composition, morphology, and degree of amorphous region of the nanocomposites are effectively regulated. The optimized electrodes exhibit high reversible capacities, stable cycling, favorable rate capability, and substantial pseudocapacitive contributions. In particular, the samples synthesized for 3 and 6 h deliver reversible capacities of 1146 and 923 mAh g^{-1} , respectively, after 100 cycles at 0.1 A g^{-1} , while all nanocomposite electrodes exhibit initial coulombic efficiencies above 77%. The enhanced lithium-storage performance is attributed to the synergistic interaction between the two molybdate phases and the controllable partially amorphous nanosheet structure. The nanoscale architecture shortens Li^+ transport pathways, whereas the Fe- and Mo-containing species formed during cycling facilitate charge transfer and the reversible decomposition of Li_2O . These findings establish a general design strategy combining dual-molybdate phase synergy with controllable amorphous nanostructure for high-capacity anodes.

1. Introduction

In recent decades, the increase in unusual climate events, along with the increasing frequency and intensity of natural disasters, has been closely linked to human-caused greenhouse gas emissions, particularly

from the energy sector [1,2]. Consequently, the transition to renewable energy sources and electrification of transportation systems have created an urgent need for efficient, reliable, and cost-effective energy storage technologies [3,4]. Lithium-ion batteries (LIBs) are currently among the most promising candidates owing to their high energy

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density, long cycle life, and high-speed operation [5,6]. However, the practical energy density of LIBs is still limited by the low theoretical capacity of commercially available graphite anodes (372 mAh g^{-1}), hindering their application in next-generation high-energy-density energy storage systems [7].

In recent times, transition metal-based materials have attracted considerable attention in various applications such as biomedical, photocatalyst, and electrochemical [8]. For instance, M. Masjedi-Arani [9] et al. successfully synthesized NiCo_2O_4 – based material with a hierarchical 3D structure. Benefiting from its unique structure, this material exhibits superior electrochemical performance when employed as a material hydrogen storage. Especially in the field of LIBs, due to their conversion reaction mechanisms, which enable significantly higher theoretical capacities [10–12]. For example, M. Ghiyasiyan-Arani [13] et al. synthesized and applied material AlV_3O_9 – hierarchical 3D structure as an anode electrode in LIBs, this material possesses specific capacity of $609.09 \text{ mAh g}^{-1}$ at a current density of 0.1 A g^{-1} . However, the transition metal materials encounter several serious issues such as material structure suffers from severe volume expansion during the electrochemical process, low electrical conductivity and ion conductivity [10]. In latest reports, the synthesis of composite with carbon materials or material with nanostructures has been recommended as approaches to address this problem [14]. Among these, iron(II) molybdate (FeMoO_4) has emerged as a promising anode material owing to the polyvalent oxidation–reduction chemical properties of Fe and Mo, along with its abundant reserves and low cost [15,16]. However, similar to other transition metal materials, the electrochemical performance of FeMoO_4 is limited by inherent drawbacks, including poor electrical conductivity, low specific surface area, and significant volume changes during repeated lithium oxidation/reduction [17]. Recent studies have suggested that composite design and nanoscale structural tuning are effective strategies for overcoming these limitations [17,18]. On the other hand, the preparation of a composite material from FeMoO_4 and carbon materials also presents certain limitations. First, the carbon materials generally possess large surface area, which triggers side reactions occur, resulting in consuming a large amount of Li^+ ion causing the low coulombic efficiency in initial cycle [19]. Second, doping carbon materials in large proportions may block Li^+ ion transport channels, thereby increasing impedance of electrode [20]. Third, the composite material synthesis method is commonly complex, making it challenging to precisely control material structure [21]. Additionally, several methods also necessitate high – temperature processing, leading to substantial energy consumption if applied at an industrial scale [22]. Furthermore, the synthesis of carbon materials such as CNT, graphene, and reduction graphene oxide encounters significant challenges regarding expense and environment [23]. In recent reports, sodium molybdate (Na_2MoO_4) has been identified as a potential anode material, particularly when integrated into composite systems with other metal oxides, where synergistic effects can improve conductivity, cycle stability, and Li^+ ion storage kinetics [24–26]. In addition, the inclusion of an amorphous phase in the material significantly improves cycle strength by allowing Li^+ ions to insert in an isotropic direction, reducing local stress and limiting structural collapse [27]. At the same time, the amorphous phase with a large free volume and interconnected channels enhances the electrode–ion interaction, improves the reaction kinetics, and effectively absorbs volume changes [26,28].

To leverage of the complementary properties of both materials, we present a simple and scalable synthesis strategy for fabricating $\text{FeMoO}_4/\text{Na}_2\text{MoO}_4$ nanocomposites (NFMO NCs) and systematically investigate their electrochemical performance as LIB anodes. The resulting NCs exhibit high reversibility, long cycle stability, superior rate capacity, and significant pseudocapacitive contribution. These advantages are attributed to the synergistic effects of the two molybdate phases, nanosheet structures, and partially amorphous phase. During cycling, nanoscale Mo and Fe particles were formed, increasing the electrical conductivity and catalyzing the decomposition of Li_2O , while the amorphous phase

acted as a buffer to absorb the volume change, enhancing the cycle stability of the NFMO NCs. The material properties were tuned according to the reaction time.

2. Materials and methods

2.1. Synthesis of $\text{FeMoO}_4/\text{Na}_2\text{MoO}_4$ NCs

A polymer-containing precursor (designated as Solution A) was obtained by dissolving 150 mg of poly(2-ethyl-2-oxazoline) (PEtOx, $M \approx 50,000$) and 700 mg of polyvinylpyrrolidone (PVP, $M \approx 360,000$) in 30 mL of triethylene glycol (TEG). The mixture was magnetically stirred at 90°C in a three-neck flask under Ar until a clear homogeneous solution was obtained. Simultaneously, $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ (4 mmol) was dissolved in 30 mL of TEG, then 10 mL of NaOH solution (2 M) was added to form Solution B. Separately, NaBH_4 (54 mmol) was dissolved in 40 mL of TEG to form Solution C. After achieving complete homogeneity, Solution B was rapidly poured into Solution A under continuous stirring. Next, Solution C was slowly dripped through a pressure-balanced funnel. The reaction was accompanied by significant gas release and was maintained for an additional 2 h after the end of the dripping process before being cooled naturally to room temperature. The resulting insoluble bipyramidal Na_2MoO_4 precipitate was separated by centrifugation at 9000 rpm for 10 min and thoroughly washed three times with ethanol.

The resulting solid was then redispersed in 70 mL of TEG by sonication to form Solution D, which was then heated to 180°C under Ar with continuous stirring. Simultaneously, Solution E was prepared by dissolving $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (4 mmol) in 30 mL of TEG. Solution E was then slowly added dropwise to Solution D while maintaining the reaction temperature at 180°C . The reaction was allowed to proceed for 3 or 6 h. After cooling to room temperature, the product was recovered by centrifugation (9000 rpm, 10 min) and washed three times with ethanol. The resulting powders were vacuum dried at 70°C for 4 h, then heat treated at 300°C for 6 h under Ar. The final samples were designated NFMO_3h and NFMO_6h and used for structural characterization and subsequent electrochemical property evaluation.

2.2. Materials characterization

The phase compositions and crystalline structures of the powder samples were identified by powder X-ray diffraction (XRD) analysis. The surface morphology, particle dimensions, and microstructural characteristics were investigated using transmission electron microscopy (TEM), high-resolution TEM (HRTEM), and selected area electron diffraction (SAED) analyses. The elemental composition and spatial distribution of the constituent elements were determined using energy-dispersive X-ray spectroscopy analysis.

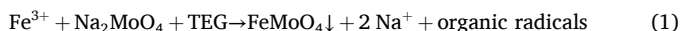
2.3. Electrochemical measurements

The working electrodes were fabricated by dispersing the active material (70% by weight), Super P conductive carbon (15% by weight), and poly(acrylic acid) binder (15% by weight) in ethanol under continuous stirring to obtain a homogeneous suspension. This suspension was evenly applied to the copper foil using a doctor-blade spreading method, then dried at 70°C for 3 h and further dried overnight at the same temperature to ensure complete removal of the solvent. After drying, the electrodes were stamped into 1.5-cm-diameter circular discs. The active material loading on the working electrode was approximately $0.90\text{--}1.0 \text{ mg cm}^{-2}$, and the corresponding areal capacity was approximately $1.20\text{--}1.26 \text{ mAh cm}^{-2}$. CR2032 button cell batteries were assembled in an argon-gas-filled glove box (MBRAUN) using the fabricated electrode as the working electrode, Li metal foil as both the counter and reference electrodes, a Celgard 2400 separator, and an electrolyte consisting of a 1 M LiPF_6 solution dissolved in a 1:1 volume mixture of ethylene carbonate and diethyl carbonate.

Cyclic voltammetry (CV) measurements were performed using an Arbin electrochemical system in the potential range of 0.01–3.00 V vs. Li/Li⁺, with an initial scan rate of 0.3 mV s^{−1}. After two activation cycles, additional CV measurements were performed at scan rates varying from 0.3 to 1.2 mV s^{−1} to investigate electrochemical kinetics. The galvanostatic charging and discharging process was performed at a current density of 0.1 A g^{−1} within the same voltage window. The rate capability was evaluated after the first five cycles by sequentially applying current densities of 0.1, 0.2, 0.5, 1.0, and 3.0 A g^{−1}. Electrochemical impedance spectroscopy (EIS) analysis was performed using a Gamry 1010E system in the frequency range of 100 kHz to 100 mHz.

3. Results and discussion

Initially, Na₂MoO₄·2H₂O dissolves in the TEG/PVP/PEtOx solution under alkaline conditions (NaOH), generating Na⁺ and MoO₄^{2−} ions. When the solution reaches supersaturation, Na⁺-MoO₄^{2−} complexes are formed and subsequently nucleate primary Na₂MoO₄ crystals. These crystal nuclei continue to grow anisotropically through the continuous addition of Na⁺ and MoO₄^{2−} ions. Simultaneously, PVP and PEtOx selectively adsorb onto specific crystal planes, orienting crystal growth and limiting particle aggregation. In addition, NaBH₄ maintains a reducing environment, facilitating the controlled nucleation and growth of Na₂MoO₄ crystals. The result was the acquisition of highly crystallized bipyramids of Na₂MoO₄ (Fig. S1) [26]. Next, upon the addition of FeCl₃, the bipyramids of Na₂MoO₄ acted as sacrificial precursors and gradually dissolved from the surface, releasing MoO₄^{2−} ions into the solution. These ions reacted with Fe³⁺ in the TEG medium to form FeMoO₄ nuclei on the particle surface (Eq. 1). After 3 h of reaction, the product consisted of residual Na₂MoO₄ and low-crystallization FeMoO₄ nanosheets. When the reaction time was extended to 6 h, the continuous dissolution of Na₂MoO₄ along with the growth of FeMoO₄ crystals and secondary nucleation continued, leading to the formation of FeMoO₄ nanosheets uniformly decorated with nanoparticles (Fig. 1). Therefore, both the morphology and phase composition of the NFMO NCs are strongly influenced by the reaction time.



XRD analysis was performed to determine the crystalline phases of the synthesized samples (Fig. 2). Characteristic diffraction peaks appearing at 2θ values of approximately 19°, 23°, 25.1°, 26.2°, 27°, 31.6°, 32.5°, 33.5°, and 39.7° can be assigned to the (201), (021), (201), (220), (112), (022), (222), and (330) crystallographic planes of FeMoO₄, respectively. The diffraction peaks at approximately 16.8°, 27.7°, 32.6°, 49.0°, 52.0°, 57.1°, and 60.0° correspond to the (111), (220), (311), (422), (511), (440), and (531) planes of Na₂MoO₄, respectively. The simultaneous appearance of these characteristic peaks confirmed the successful synthesis of NCs comprising both FeMoO₄ and Na₂MoO₄ phases. The crystalline and amorphous compositions of the two NFMO NCs were calculated using Eqs. S1 and S2 and are presented

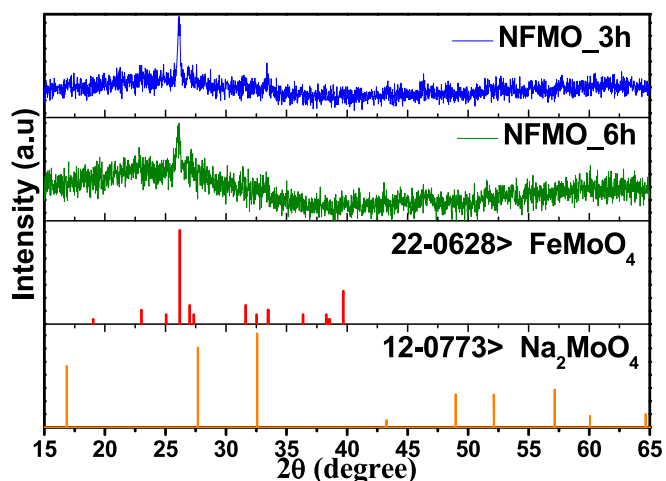


Fig. 2. XRD patterns of (a) NFMO_3h and (b) NFMO_6h NCs, with reference peaks of FeMoO₄ and Na₂MoO₄ indicated.

in Table S1. Both NFMO NCs showed high amorphous composition, accounting for approximately 80.15% and 72.73% for NFMO_3h and NFMO_6h NCs, respectively. Here, an increased crystallization rate was observed in the sample with a longer reaction time. This can be explained by the fact that maintaining a longer reaction time facilitates a more ordered structural arrangement within the NFMO particles. Differences in crystalline and amorphous formation between the two samples were further observed using HRTEM techniques.

The TEM images show that all the NC samples consist of numerous ultrafine particles smaller than 10 nm, along with many nanosheet-like structures. These nanoscale features are particularly advantageous for lithium storage because they can promote the reversible decomposition of Li₂O during electrochemical cycling when used as anodes in LIBs [29,30]. Owing to their high surface energies, these nanoparticles tended to self-assemble into nanosheet structures as the reaction time increased from 3 to 6 h. The HRTEM images combined with SAED patterns confirmed the simultaneous existence of crystalline and disordered regions in the NCs; at the same time, the number of crystalline phases in the NFMO samples increased with prolonged reaction time. Clear lattice patterns corresponding to both the FeMoO₄ and Na₂MoO₄ phases were observed, along with amorphous regions (Fig. 3). Specifically, for the NFMO_6h sample (Fig. 3b), lattice spacings of approximately 0.47 nm and 0.16 nm were identified, corresponding to the (201) plane of FeMoO₄ and the (440) plane of Na₂MoO₄, respectively. These microscopy images are consistent with the phase information obtained from XRD analysis. The elemental composition and spatial distribution within the NFMO samples were analyzed using scanning TEM (STEM) coupled with elemental mapping. As shown in Fig. S2 and summarized in Table S2, the samples contained varying amounts of Na, Fe, Mo, and O. The elemental mapping images (Fig. 4) show that these elements were uniformly distributed throughout the NFMO NCs, confirming the successful integration of the constituent phases at the nanoscale.

CV measurements were performed to clarify the lithiation/delithiation mechanism of the NFMO anodes. As shown in Fig. 5, the first CV cycle differed considerably from the subsequent cycles, indicating an activation process. During the initial cathodic sweep, multiple reduction peaks appeared, which were attributed to the intercalation of Li⁺ into the FeMoO₄ and Na₂MoO₄ phases (Eq. 2, Eq. 3) [15,31], the reduction of Mo⁶⁺ and Fe²⁺ to their respective metallic states (Eq. 4) [32,33], as well as the formation of a solid electrolyte interphase (SEI) layer [34]. In the anodic sweep branch, the first oxidation peak at ≈0.50 V corresponds to the oxidation of Mo⁰ to Mo^{+2z} (Eq. 5) [35], and a broad oxidation peak (from 1.0 to 2.5 V) is observed, related to the re-oxidation of Mo and Fe metals (Eq. 6, Eq. 7) [36]. In subsequent cycles, the disappearance of some of the initial reduction peaks indicated irreversible structural

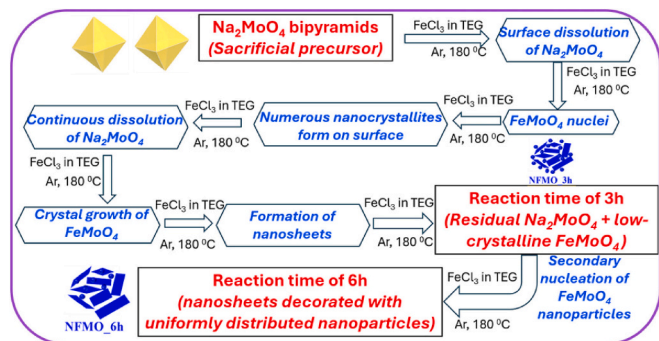


Fig. 1. Formation mechanism of NFMO NCs.

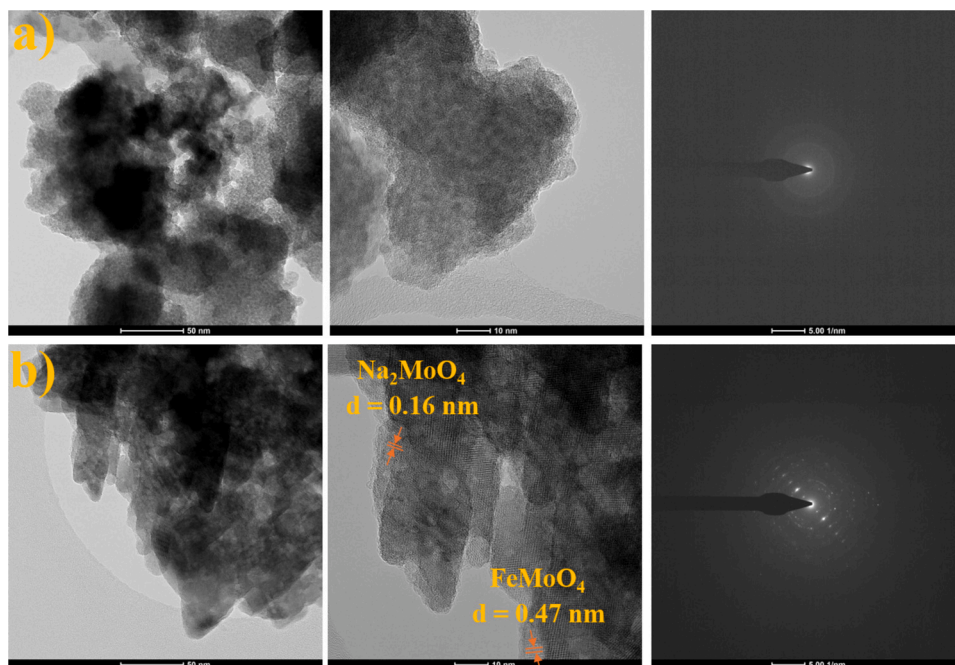


Fig. 3. TEM, HRTEM, and SAED images of (a) NFMO_3h and (b) NFMO_6h NCs.

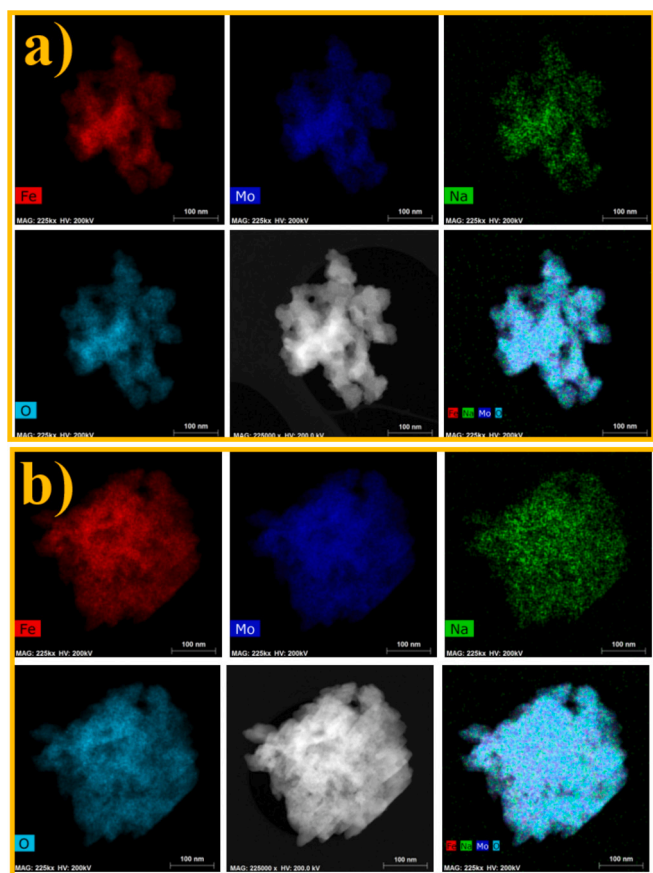
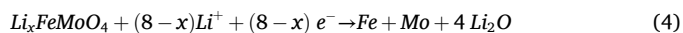
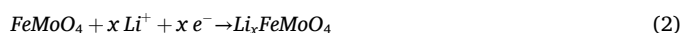


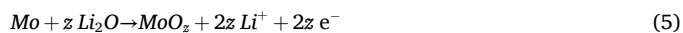
Fig. 4. STEM images with elemental mapping of (a) NFMO_3h and (b) NFMO_6h NCs.

transformations and stabilization of the SEI layer. The remaining redox peaks were attributed to the reversible transformation reactions of the Mo-based species (Eq. 8, Eq. 10) [36] and Fe (Eq. 9) [15,37]. The slight shift and broadening of the peaks in the later cycles mainly stem from electrode polarization and structural rearrangement. Overall, lithium storage in the NFMO electrodes is governed by a multistep conversion mechanism, where the initial lithium intercalation is not entirely reversible, whereas subsequent cycles exhibit good reversibility. The overlapping of the CV curves after the first cycle confirms the stable and reversible electrochemical performance of the NFMO electrodes.

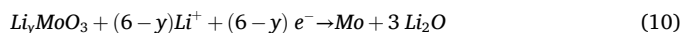
First discharge:



Charge process:



Discharge process:



During discharging at potentials lower than 0.50 V, reactions between Li^+ ions and FeMoO_4 nanoparticles lead to the formation of Mo and Fe metal nanoparticles and the creation of Li_2O phases [24,35,38–42]. The Mo and Fe nanoparticles then promote the

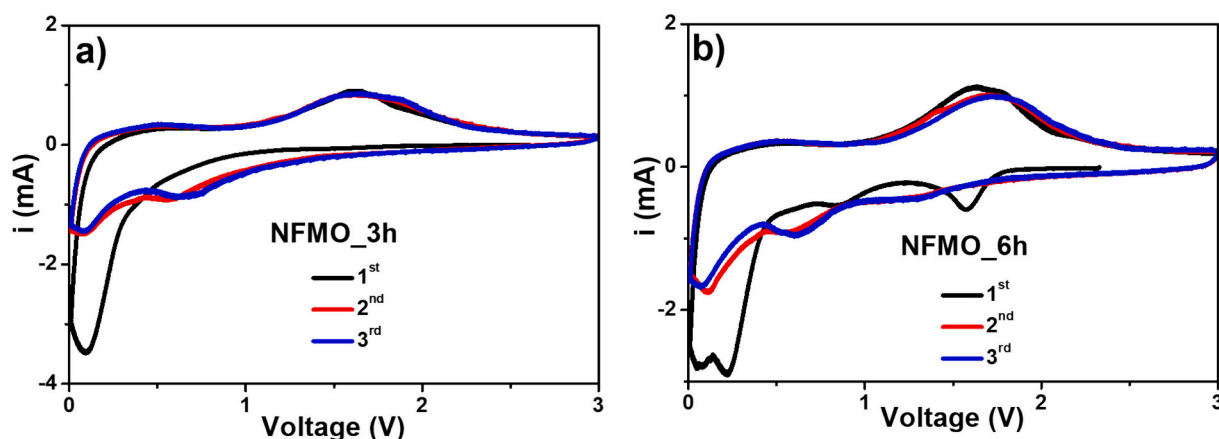


Fig. 5. CVs of (a) NFMO_3h and (b) NFMO_6h electrodes.

reversible decomposition of Li_2O during charging and effectively inhibit particle aggregation [43,44], improving the electrochemical performance. Furthermore, owing to the higher conductivity of Mo ($\approx 2 \times 10^7 \text{ S m}^{-1}$) compared with Fe ($\approx 10^7 \text{ S m}^{-1}$), molybdate-derived components enhance electron transport, reduce charge-transfer resistance, and improve Li^+ diffusion in the electrode. Overall, the molybdate species plays a key role in governing the electrochemical behavior of the NFMO electrodes.

Fig. 6 shows the initial galvanostatic charge–discharge curves of the NFMO electrodes. The sample synthesized for a reaction time of 3 h yielded initial discharge/charge capacities of $1481/1244 \text{ mAh g}^{-1}$, respectively, corresponding to an initial coulombic efficiency (ICE) of 84%. Conversely, the electrode obtained after reacting for 6 h showed a higher initial capacity of $1835/1424 \text{ mAh g}^{-1}$, but the ICE decreased slightly to 77.6%. These results demonstrate a clear tradeoff between the reversible capacity and initial yield as the synthesis time increases. This phenomenon is most likely related to the change in the relative ratio between the FeMoO_4 and Na_2MoO_4 phases (Table S2), differences in morphology and phase states, and differences in the formation and stability of the SEI layer during the first electrochemical cycle. After the first cycle, the coulombic efficiency (CE) of all electrodes increased rapidly and approached 100%, indicating the formation of a stable SEI layer. Once established, the SEI layer acts as an effective barrier, limiting direct electron leakage into the electrolyte and significantly reducing unwanted side reactions. Consequently, the irreversible Li^+ loss was virtually eliminated, allowing for stable and sustainable electrochemical operation in subsequent cycles. This stable behavior is clearly demonstrated in Fig. 6, where the charge–discharge curves of the second and

third cycles nearly coincide, proving the highly reversible lithium storage capability and excellent cycle repeatability of the NFMO electrodes.

The long-term cycle behavior and CE of the NFMO anodes were evaluated at a current density of 0.1 A g^{-1} , as shown in Fig. 7a. For the NFMO_3h electrode, a slight decrease in capacity is observed during the first 10 cycles, after which the capacity stabilizes and remains almost constant until cycle 100. By contrast, the NFMO_6h electrode exhibited a moderate capacity decrease in the initial cycles, followed by a gradual increase until 60 cycles, and a slow decline as it approached the 100th cycle. The initial capacity loss of both electrodes was mainly attributed to the incomplete reversibility of the Li_2O decomposition process during the early charging cycles. This behavior was most likely related to the insufficient generation of Mo/Fe metal nanoparticles during the initial discharge cycles, which limited the catalytic role of Mo/Fe in promoting the initial Li_2O decomposition. The observed capacity recovery upon prolonged cycle lengthening may stem from several synergistic effects, including electrochemical grinding [42,45,46], gradual formation of in situ metal nanoparticles [42,47–50], and the role of Mo/Fe species in catalyzing the reversible Li_2O conversion reaction [43,44]. Similar capacity activation has been reported for several other anodic conversion materials, such as the $\text{Fe}_2(\text{MoO}_4)_3$ system [42,46]. Although direct ex-situ characterization was not performed in this work, the electrochemical behavior observed here is consistent with these previously reported mechanisms. For the NFMO_6h electrode, the gradual capacity degradation after 60 cycles was most likely related to the strong volume change during repeated lithiation/delithiation, which may cause structural instability during long-term cycling. After 100 cycles, the NFMO_3h and NFMO_6h electrodes achieved reversible capacities of

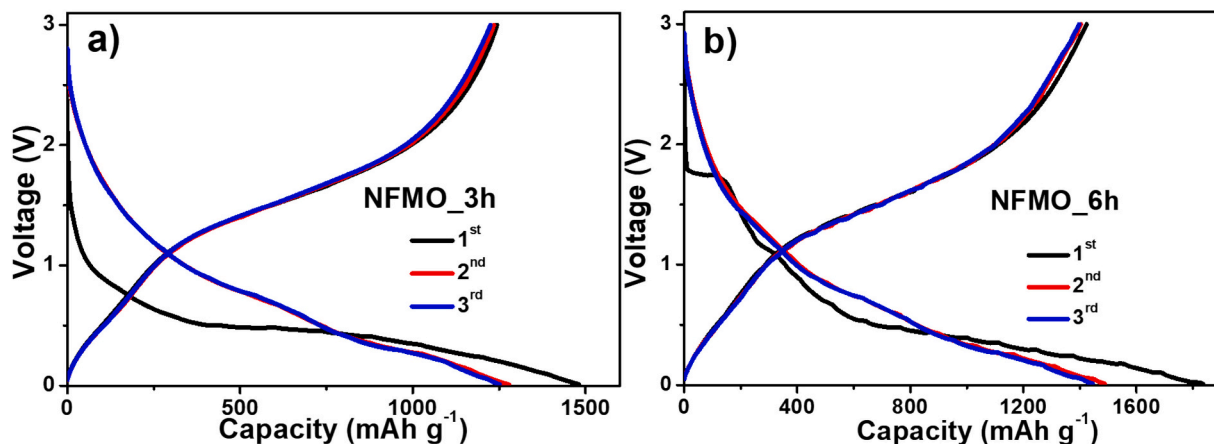


Fig. 6. Voltage profiles of (a) NFMO_3h and (b) NFMO_6h electrodes.

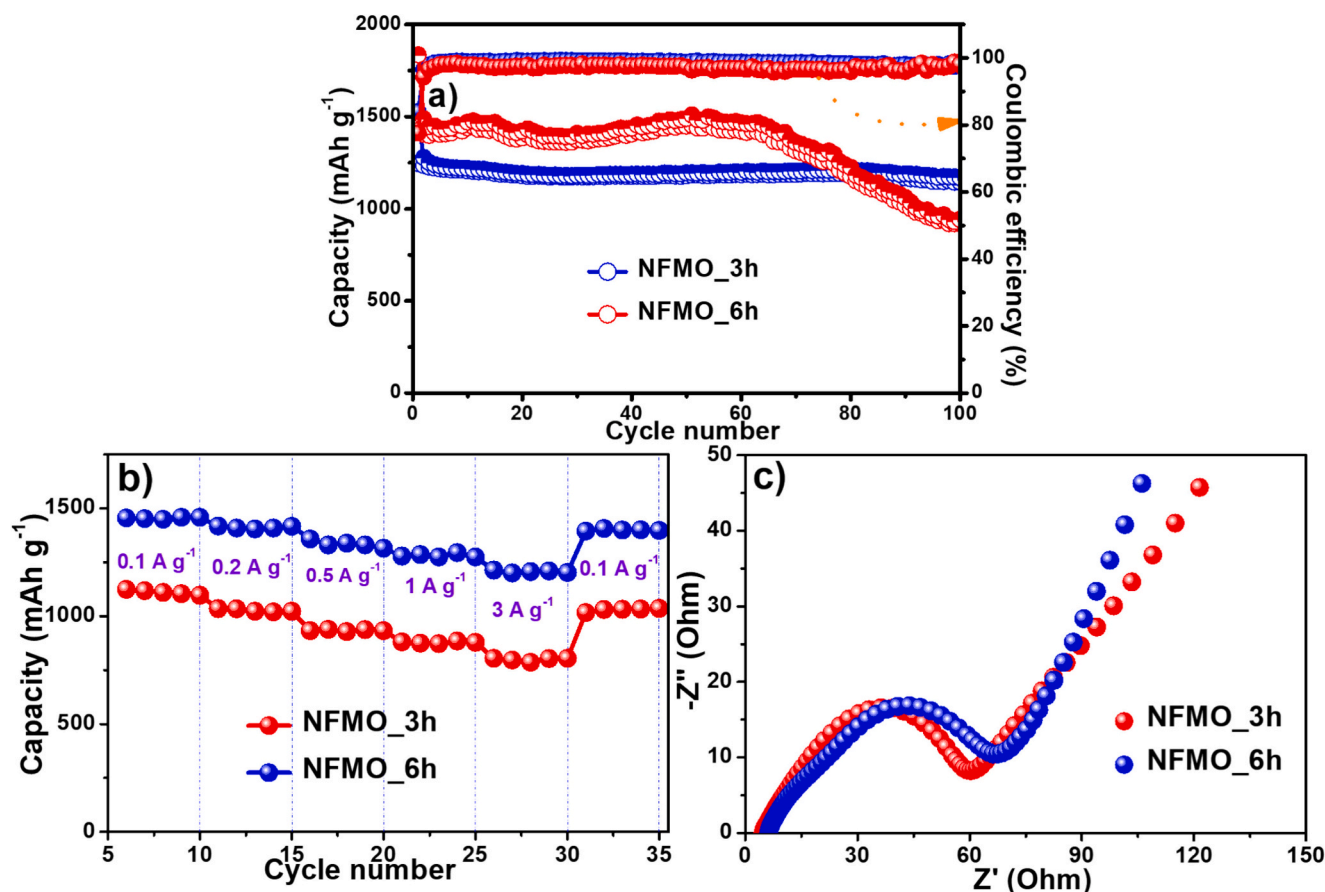


Fig. 7. (a) Cycle performance, (b) rate cycle performance, and (c) EIS of NFMO electrodes.

1146 and 923 mAh g⁻¹, respectively, corresponding to capacity retentions of 92.1% and 64.8%. The difference in the capacity retention over cycles of these two electrodes can be explained by their phase composition. Indeed, the NFMO_3h NC has a higher proportion of amorphous phase, which acts as a buffer, absorbing the stresses from volumetric expansion during battery operation. This helps the NFMO_3h NC maintain structural stability during lithiation/delithiation. Conversely, the NFMO_6h NC has a higher crystalline phase than the other sample, which improves the capacity in the initial stages. However, as the number of cycles increases, a lower amorphous phase content reduces the ability of the electrode material to accommodate volume expansion, leading to structural degradation and gradual capacity fading during cycling [27,28,51]. Notably, the electrochemical performance of the NFMO_3h electrode is superior to those of many recently reported molybdate-based anodes, as summarized in Table 1. Furthermore, the CE values of both NFMO electrodes increased rapidly in the early cycles and then stabilized. The NFMO_3h electrode achieved a CE above 99% after approximately eight cycles, whereas the NFMO_6h electrode maintained slightly lower CE values, ~97%, throughout the

cycles, indicating less stable interface kinetics.

Fig. S3 presents the surface morphology of the NFMO electrodes before and after 100 cycles. After the cycling process, the NFMO_3h electrode maintained the integrity of its electrode structure. A coating appeared on the surface, partially obscuring the original shape of the material particles. This coating is most likely a SEI layer, formed from the decomposition of the electrolyte in the first cycle. The continuous and stable SEI layer acts as a passive barrier, allowing Li⁺ ions to diffuse through but preventing direct contact between the electrode and the electrolyte. As a result, electrolytic decomposition reactions are limited, the consumption of active Li⁺ is significantly reduced, and the electrode surface is protected in subsequent cycles [34,52,53]. The formation of this stable SEI layer is consistent with the high capacity retention and superior cycle durability of the NFMO_3h electrode. Conversely, after 100 cycles, the NFMO_6h electrode showed surface cracks. This phenomenon reflects mechanical instability due to repeated volume changes in the active material during the lithiation/delithiation process. When the accumulated stress exceeds the tolerance limit, the active particles crack or detach from the conductive network, impairing the

Table 1

Summary of reported molybdate-based anode materials for Li-ion batteries.

Molybdate based anodes	Current density (mA g ⁻¹)	Mass loading (mg cm ⁻²)	Electrolyte	Cycling number	Capacity (mAh g ⁻¹)	Reference
FeMoO ₄	100	–	1 M LiPF ₆	80	926	[15]
3D Fe ₂ (MoO ₄) ₃	100	–	1 M LiPF ₆	50	745	[41]
FeMoO ₄ /Fe ₂ O ₃ -NC	100	1.0	1 M LiPF ₆	100	1021	[22]
ZnMoO ₄	100	1.17	1 M LiPF ₆	100	457.7	[57]
Bi ₂ O ₃ @Bi ₂ MoO ₆ _400°C	100	0.9–1.0	1 M LiPF ₆	80	701	[58]
CoMoO ₄	100	1–1.5	1 M LiPF ₆	50	990	[59]
P-NiMoO ₄	100	1.5–2	1 M LiPF ₆	100	1130	[60]
NFMO_3h	100	0.9–1.0	1 M LiPF ₆	100	1146	This work

continuity of the electron pathway and the diffusion pathway of Li^+ ions. Simultaneously, new cracked surfaces continuously come into contact with the electrolyte, promoting electrolytic decomposition and the formation of a secondary SEI layer. This process not only consumes more active Li^+ but also increases the charge transport resistance, thereby increasing the electrochemical polarization and hindering reaction kinetics of the electrode. The combination of mechanical integrity degradation, electrical disconnection, increased resistance, and the continuous growth of SEI layer on fractured surfaces leads to the rapid and continuous degradation of the NFMO_6h electrode capacity during the cycling process [54–56].

Fig. 7b illustrates the rate performance of the NFMO electrodes evaluated under a series of increasing current densities. Both the NFMO_3h and NFMO_6h electrodes delivered stable capacities over a wide current range of $0.1\text{--}3.0\text{ A g}^{-1}$. Specifically, the NFMO_3h electrode maintained capacities of 1111, 1027, 934, 878, and 799 mAh g^{-1} , whereas the NFMO_6h electrode showed corresponding values of 1455, 1411, 1335, 1282, and 1207 mAh g^{-1} at 0.1, 0.2, 0.5, 1.0, and 3.0 A g^{-1} , respectively. After the current density was reverted to 0.1 A g^{-1} , the charge capacities recovered to 1030 mAh g^{-1} for NFMO_3h and 1400 mAh g^{-1} for NFMO_6h, demonstrating excellent structural robustness under high-rate operation. The superior rate reversibility of the NFMO electrodes can be attributed to the formation of a stable SEI layer, favorable nanostructured architecture, coexistence of micro- and mesoporous features, and optimal proportion of FeMoO_4 and Na_2MoO_4 active phases.

The electrochemical impedance behaviors of the NFMO electrodes fabricated under different conditions are shown in Fig. 7c. All Nyquist spectra exhibit a flattened semicircle in the mid-frequency region, related to charge transfer at the interface (R_{CT}) and resistance related to the SEI layer (R_{SEI}), followed by a sloping line in the low-frequency region, corresponding to the diffusion of Li^+ in the electrode lattice. The

similarity of the spectral shapes indicates that the electrodes share the same electrochemical reaction mechanisms. However, the marked differences in the sizes of the semicircles reflect significant differences in the interface kinetics. Quantitatively, the total resistance values of the NFMO_3h and NFMO_6h electrodes were estimated to be 53.4 Ω and 57.1 Ω , respectively (Fig. S4 and Table S3). Among the investigated samples, the NFMO_3h electrode exhibited the lowest resistance. The prolonged reaction time promotes the transformation from incompletely grown FeMoO_4 nanosheets to fully grown FeMoO_4 nanosheets decorated with nanoparticles. This structural evolution increases the contact area between the electrode and the electrolyte, while shortening the diffusion path of Li^+ ions, thereby contributing to an increased Li^+ diffusion coefficient and improved electrochemical kinetics of the NFMO_6h electrode. However, the larger contact surface area can also promote electrolyte degradation and the continuous growth of the SEI film during long cycles, which is detrimental to long-term cycle stability. Conversely, the NFMO_3h electrode with its two-phase structure maintains a more stable electrode structure, thereby achieving superior capacity retention. Therefore, the optimal reaction time reflects the balance between the ability to rapidly transport Li^+ ions and the structural stability of the electrode, thereby determining the long-term electrochemical performance of the material.

To elucidate the charge storage kinetics, the NFMO electrodes were systematically evaluated by separating the diffusion-dominant and capacitance-dominant contributions through CV measurements at varying scan rates (Fig. 8(a and b)). The analysis was performed using Eqs. S3 and S4, followed by the numerical solution of Eq. S5. By applying the same mathematical procedure, the CV responses were separated into diffusion and capacitance components at each scan rate, as shown in Fig. S5 and S6. The relative contribution of these two charge storage mechanisms were calculated using Eqs. S6 and S7 and are summarized in Fig. 8(c and d). Notably, for all electrodes

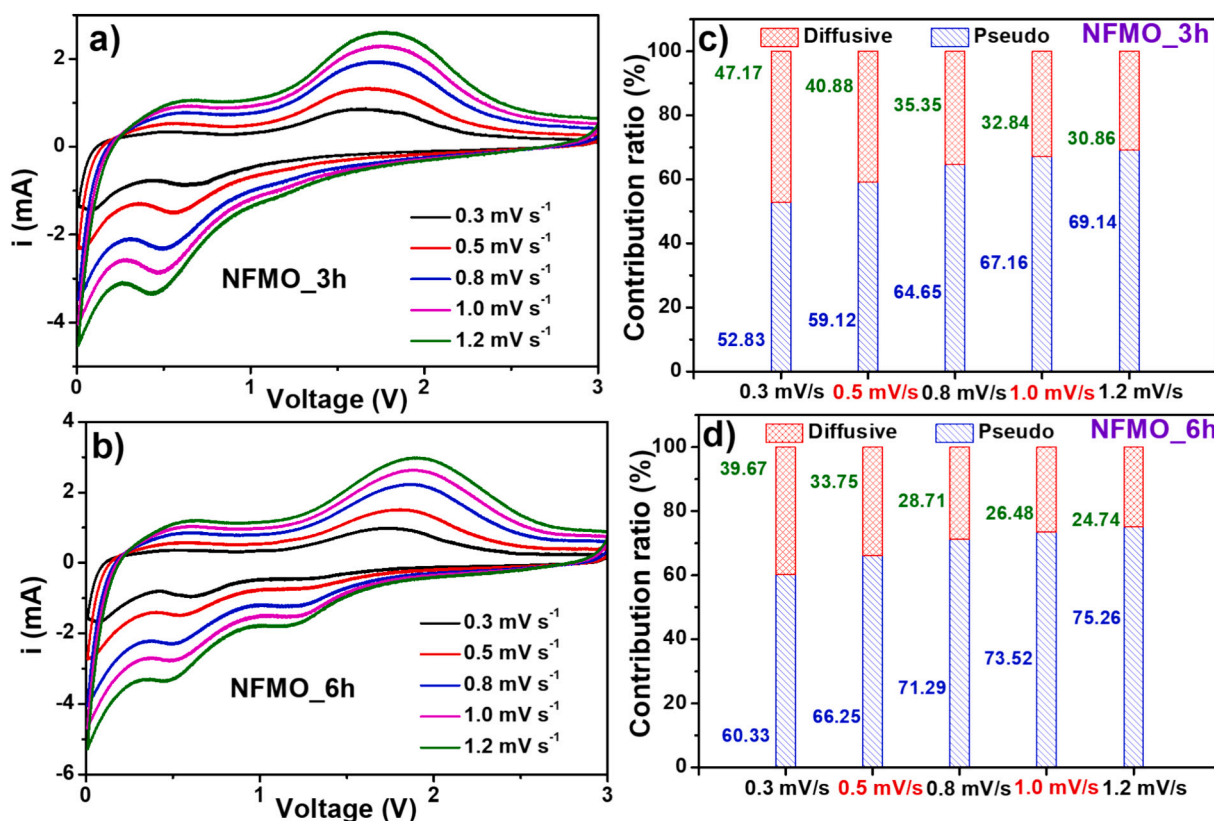


Fig. 8. CVs of (a) NFMO_3h and (b) NFMO_6h electrodes at varying scan rates. The contribution rate of the pseudo and diffusion for (c) NFMO_3h and (d) NFMO_6h electrodes.

investigated, the capacitance-dominant process accounted for more than 52% of the total current response, indicating that charge storage behavior was primarily dominated by surface mechanisms. Specifically, the NFMO_6h electrode exhibits a slightly higher pseudocapacitance contribution ($\approx 7\%$) than the NFMO_3h electrode at the same scan rate. This enhanced pseudocapacitive behavior is beneficial for accelerating the charge-discharge process and improving the performance at high current densities. These kinetic characteristics are in close agreement with the observed results for cycle stability, rate capability, and EIS, confirming the favorable charge-transfer kinetics of the NFMO electrodes. To gain a deeper understanding of the ion transport mechanism, the charge storage behavior was analyzed by differentiating the contributions of diffusion and pseudocapacitive, as shown in Fig. S7a. The relationship between peak current (i) and scan rate (ν) (Eq. S8) was evaluated through $\log(i) - \log(\nu)$ plots (Eq. S9) of the anode peaks of the NFMO electrode. Linear interpolation of these plots yielded slope values (b) of 0.77 and 0.81 for the NFMO_3h and NFMO_6h electrodes, respectively. Because these b values are close to 1, the electrochemical processes in the NFMO electrodes are primarily governed by surface-dominated pseudocapacitive mechanisms rather than by diffusion-limited Li^+ ion intercalation mechanisms. Fig. S7b shows the dependence of peak current (I_p) on the square root of the scan rate ($\nu^{1/2}$). According to Eq. S10, the slope obtained from the $I_p - \nu^{1/2}$ interpolation is directly related to the square root of the ion diffusion coefficient (D). Therefore, the slope was used as a qualitative indicator to compare the ion diffusion capacities of the different electrodes. As shown in Fig. S7b, the NFMO_6h electrode showed a larger slope than the NFMO_3h electrode, indicating that the Li^+ ion diffusion capacity followed the order NFMO_6h > NFMO_3h. These results are in close agreement with the kinetic characteristics presented in Fig. 8.

4. Conclusions

In this study, $\text{FeMoO}_4/\text{Na}_2\text{MoO}_4$ nanocomposites with reaction-time-dependent morphologies, phase compositions, and degrees of amorphous region were successfully synthesized and evaluated for the first time as anode materials for lithium-ion batteries. The optimized NFMO_3h and NFMO_6h electrodes delivered reversible capacities of 1146 and 923 mAh g^{-1} , respectively, after 100 cycles at 0.1 A g^{-1} , together with favorable cycling stability, rate capability, and pseudocapacitive behavior. The enhanced electrochemical performance is attributed to the synergistic effects of the dual-molybdate phases and the partially amorphous nanosheet structure. The nanoscale architecture accommodates volume changes, increases the electrode-electrolyte interfacial area, and shortens Li^+ transport pathways. In addition, Fe and Mo species generated during cycling enhance charge transport and promote the reversible decomposition of Li_2O , thereby improving reaction kinetics and reversible lithium storage. These findings suggest a general design strategy based on dual-molybdate phase synergy combined with controllable amorphous nanostructures for high-capacity electrodes. Further development should focus on carbon coating or conductive-network integration, metal doping, suppression of initial irreversible lithium loss, and fabrication of high-area-loading thick electrodes. Full-cell evaluation with practical cathodes and subsequent pouch-cell validation will be required to assess the potential of $\text{FeMoO}_4/\text{Na}_2\text{MoO}_4$ nanocomposites for high-energy and high-power lithium-ion batteries.

CRedit authorship contribution statement

Tuan Loi Nguyen: Writing – original draft, Investigation, Funding acquisition. **To Giang Tran:** Formal analysis. **Thuy-An Nguyen:** Software. **Dinh Quan Nguyen:** Visualization, Resources. **Hieu Trung Bui:** Formal analysis, Data curation. **Dongwon Chun:** Validation, Formal analysis. **Kwang J. Kim:** Resources, Methodology. **Tran Thi Kieu Ngan:** Formal analysis. **Il Tae Kim:** Writing – original draft, Conceptualization.

Jaeyun Moon: Writing – review & editing, Resources, Project administration, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.inoche.2026.117513>.

Data availability

The data supporting the findings of this study are available from the corresponding author upon request.

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