Earth-abundant and environmentally friendly aluminosilicate minerals can be one of the promising alternatives to develop cost-effective energy conversion and storage devices. Herein, in situ growth of transition metal nanoparticles is proposed to modify two commonly available feldspar minerals, albite and microcline, for promoting electrocatalytic oxygen evolution reaction activity via a one-step thermal reduction strategy. Three types of transition metal nanoparticles, namely, Ni, Co, and Fe, are selected to modify the albite or microcline surfaces. As expected, these modified products deliver enhanced catalytic activities compared to the pristine minerals. Particularly, Co-modified microcline (C-KASO) demonstrates the best performance that even outperforms the commercial RuO$_2$ catalyst. This design by coupling low-cost aluminosilicate minerals with active transition metal nanoparticles offers a new insight into directly utilizing the natural abundant resources to address the current energy crisis.

1. Introduction

The current environmental and energy crisis urges the maximizing utilization of sustainable energy, whereas the economic cost of the current electrochemical energy conversion and storage technologies has impeded their widespread applications.$^{[1–3]}$ To fulfill this goal, it is always great passionate about the pursuit of advanced materials with satisfied efficiency but in reasonable production costs.$^{[4]}$ Among numerous efforts, exploring earth-abundant materials is a promising potential to achieve a good compromise between performance and cost.$^{[5–9]}$ In our early attempt, we found that the low-cost minerals after simple thermal treatment can present comparable solid-ion storage performance to the synthesized materials made from expensive analytical reagents in the typical sustainable energy storage devices of lithium-ion (Li-ion) batteries.$^{[6]}$ This attempt inspired us that it should also be approachable to directly use the low-cost minerals in sustainable fuel generations as economic catalysts.

Aluminosilicate minerals, a typical class of nontoxic, environmentally friendly, and abundantly available components in natural ores, are one of the promising alternatives to develop cost-effective energy conversion and storage devices. Until now, various aluminosilicate minerals have been studied for a wide range of energy and environment-related applications, such as 1) natural aluminosilicate minerals were used for boosting lithium storage capability after thermal reduction activation$^{[10]}$; 2) aqueous alteration of K-bearing aluminosilicate minerals can work as an alternative to both traditional K-fertilization and alumina production$^{[11]}$; 3) ultrafiltration membranes coupled with natural aluminosilicate minerals were used for metal removal from industrial wastewater$^{[12,13]}$; 4) zeolites derived from natural aluminosilicate minerals were used for fluid catalytic cracking applications$^{[14,15]}$; and 5) lanthanum-doped aluminosilicate minerals were explored for photocatalytic hydrogen evolution.$^{[16]}$ Unfortunately, due to the intrinsically chemical inertness, to best of our knowledge, currently no exploration on the direct use of aluminosilicate minerals for electrocatalytic oxygen evolution reaction (OER), a half rate-controlling step of water splitting, is found. Considering the inferior intrinsic catalytic activities of most aluminosilicate minerals, modification is usually needed for their use in electrocatalytic fuel generation reactions.

In this work, two commonly available feldspar minerals, albite (Na(AlSi$_3$)O$_8$) and microcline (K(AlSi$_3$)O$_8$), are chosen as raw materials. For promoting electrocatalytic OER activity, in situ growth of transition metal nanoparticles on the surface is proposed to modify the chosen aluminosilicate minerals. By a one-step thermal reduction strategy, with the presence of
metal-containing precursors and pristine minerals, three types of transition metal nanoparticles, Ni, Co, and Fe, are uniformly loaded onto the albite or microcline surfaces. In the previous reported works, Co, Ni, and Fe are capable to deliver satisfying catalytic activities for water-splitting reactions but with relatively low costs compared to the noble metals. These resultant products deliver enhanced catalytic activities compared to the pristine minerals. It is plausible that the Co-modified microcline (labeled as C-KASO) demonstrates the best performance that even outperforms the commercial RuO$_2$ catalyst. This design by coupling aluminosilicate minerals with transition metal nanoparticles offers us a practical solution to directly utilize the natural abundant resources to address the current energy challenges.

2. Results and Discussion

As shown in Scheme 1, white feldspar mineral powder was first mixed with glucose and transition metal-containing salts (e.g., Co (Ac)$_2$·4H$_2$O, Ni(NO$_3$)$_2$6H$_2$O, or FeCl$_3$·6H$_2$O), and then underwent a thermal annealing step at 900 °C for 2 h under a continuous Ar/H$_2$ mixture gas flow to produce metal-modified mineral products. This in situ growth strategy is simple and easy to be scaled up. Most importantly, it is capable to be adapted for various metal species or aluminosilicate minerals. It should be noteworthy that the glucose used in this synthetic protocol has several crucial roles, primarily including the following: 1) ensuring homogeneous mix of feldspar mineral powders with metal-containing salts as aqueous dispersions; 2) mediating the metal nanoparticle-support interaction during the growth process; 3) reducing the size or dimension of the resultant transition metal particles under thermal reduction process; 4) carbonizing and activating the electrochemical inert silicon oxide species within feldspar frameworks; and 5) stabilizing transition metal nanoparticles by forming metal–carbon bonds.$^{[17–19]}$ As a consequence, the resultant metal nanoparticles anchored onto feldspar miner surfaces are favorable for OER, which greatly promotes the added value of the natural abundant low-cost aluminosilicate minerals.

Scanning electron microscopy (SEM) coupled with atomic force microscopy (AFM) was used to identify the surface modifications preformed on the feldspar minerals. As shown in Figure 1, the pristine albite (P-NASO) and microcline (P-KASO) mineral particles exhibited sheet-like morphologies with relatively smooth surfaces. After in situ thermal reduction in the presence of transition metal-containing precursors, Ni-modified (N-NASO and N-KASO), Co-modified (C-NASO and C-KASO), and Fe-modified (F-NASO and F-KASO) feldspar minerals manifested rough surfaces, suggesting the successful loading of metal nanoparticles onto mineral surfaces. Moreover, the deposition of nanosize transition metals on the large-size feldspar mineral surfaces is favorable of creating rich active sites and abundant interfaces, which are beneficial for surface-dominant electrocatalytic reactions. It is also expected that the relatively good electrical conductivity of transition metals can greatly reduce the charge-transfer resistance and further promote the overall catalytic performance.

To examine the specific chemical composition in these modified feldspar mineral particles, bright-field transmission electron microscope (BF–TEM) equipped with element mapping accessory was further applied. As shown in Figure 2a,b, the P-NASO mainly consists of Si, Al, Na, and O, whereas P-KASO is mainly composed of Si, Al, K, and O. It is obviously identified that the major difference of P-NASO and P-KASO is the cations of Na and K. After modified by transition metal nanoparticles, Ni, Co, or Fe particles were uniformly loaded onto the feldspar mineral surfaces as evidenced by the homogeneous distribution of metallic elements in the corresponding element mapping patterns of N-NASO and N-KASO samples. These metallic nanoparticles onto feldspar mineral surfaces exhibited similar shapes and sizes. Further characterizations on high-resolution TEM (HR–TEM, Figure 2e–j) confirm the lattice spacing of 0.203, 0.20, and 0.206 nm, for N-NASO and N-KASO, C-NASO and C-KASO, and F-NASO and F-KASO samples, indicating the exposed faces of (111) for metallic Ni, (111) for metallic Co, and (110) for metallic Fe species, respectively. This indicates that this thermal reduction strategy should be a generalized approach for depositing various metal species onto aluminosilicate minerals and can be extended to modify other electrochemical inert inorganic composites with transition metallic active centers.

X-ray diffractometer (XRD) was subsequently used to monitor the phase changes before and after surface modifications. It has been verified that the feldspar minerals can be tuned to be electrochemically active by thermally treated under reducing conditions without the addition of any metal-containing precursor (labeled as T-NASO and T-KASO samples), by which the fully oxide silicon species of the feldspar mineral are reduced into active SiO$_x$. The partially reduced SiO$_x$ possesses an obvious electrons localization below the Fermi level, which increases the electronic conductivity, lowers the coordination numbers of cations, and benefits to the electrochemical reactions.$^{[19]}$ As shown in Figure 3a,b, after modified by transition metal nanoparticles, two typical peaks attributed to metallic Ni, Co, or Fe were identified in the XRD patterns. Specially, all metallic nanoparticles presented a face-centered cubic (fcc) structure, i.e., Ni on N-NASO and N-KASO samples possesses crystal parameters...
of \(a = b = c = 3.524\ \text{Å} \) (PDF No. 04-0850); Co on C-NASO and C-KASO samples has \(a = b = c = 3.545\ \text{Å} \) (PDF No. 15-0806); and Fe on C-NASO and C-KASO samples has \(a = b = c = 2.866\ \text{Å} \) (PDF No. 06-0696).\(^{[20–23]}\) These results agree well with the TEM analysis.

To accurately reveal the chemical states of the loaded transition metals on the feldspar mineral surfaces, X-ray photoelectron spectroscopy (XPS) was used. As shown in Figure 4a, the metallic state of the loaded transition metal nanoparticles on albite is confirmed by the peaks at lowest energy, where the core levels located at 850.2 eV in N-NASO, 776.2 eV in C-NASO, and 707.1 eV in F-NASO were attributed to the metallic Ni, Co, and Fe, respectively. There are yet some peaks appeared at higher binding energies, which should be ascribed to the chemical bonds formed between the metallic nanoparticles and the oxide substrate, the carbonized metallic species, together with the partial surface oxidation and adsorbed oxygen-containing surface groups, and satellite peaks.\(^{[24–27]}\) For example, in the C-NASO, two pairs of peaks for Co 2p centered at \(\approx 777/\approx 792\) and \(\approx 780/\approx 795\) eV should be assigned to Co\(^{3+}\) and Co\(^{2+}\), respectively.\(^{[28]}\) Similar chemical states were also identified for microcline-based N-KASO, C-KASO, and C-KASO particles (Figure 4b). Even though partial oxidation or carbonization of the metallic nanoparticles resulted by the extremely high surface activity of the nanosized particles has been observed, the metallic states will be expected to dominantly contribute to the electrocatalytic activity.\(^{[29]}\) Meanwhile, due to the abundant valence states of transition metal elements, some metastable phases formed in a mild reducing environment have exhibited significant catalytic activity.\(^{[30]}\) The residual chalcogen and nitrogen atoms originated from the metal-containing salts might also react with the sensitive metallic surfaces and the feldspar minerals, which can also contribute to the activity of the materials.\(^{[31,32]}\)

Based on the aforementioned characterizations, nanosized metallic crystallites can be easily deposited on the smooth mineral surfaces by using an in situ growth strategy with the presence of metal-containing precursors via a rapid chemical transformation at an elevated temperature.\(^{[33]}\) Due to the unique geometric nanosize effects, these active metallic nanoparticles are quite possible to be partly oxidized or carbonized on the surfaces.\(^{[29]}\) In the meanwhile, considering the abundant valence states of transition metal-based materials, some metastable phases may coexist under a mild reducing environment.\(^{[30]}\) In addition, some possible residual chalcogen and nitrogen atoms originated from the metal-containing salts may make
the chemical states of these sensitive metallic surfaces in the modified feldspar minerals more complex.\textsuperscript{[31,32]} From the viewpoint of OER, the presence of metallic nanoparticles and the partially reduced mineral structure led to complex coordinate environments of both the deposited metal nanoparticles and the modified feldspar minerals, which can create more structural vacancies or defects, effectively modulate the adsorption energy of reaction intermediates, and promoted the electrochemical reactions.\textsuperscript{[34–36]} As we have known, a four sequential proton-coupled electron-transfer process is involved in the OER accompanied by the break of O─H bonds and the formation of O═O bonds. At current research stage, Ir or Ru and their derivations have been considered to be the state-of-the-art OER catalysts; however, the exorbitant cost and scarcity of these noble metals seriously limit their practical applications.\textsuperscript{[37,38]} By depositing the transition metal nanoparticles onto the activated feldspar minerals, it is expected that the formation of nanosized transition metal active centers together with the modified minerals can boost the electrochemical catalysis activity and push the widespread use of low-cost catalysts for sustainable energy applications.

Electrocatalytic oxygen evolution performance of these transition metal-modified feldspar mineral catalysts was evaluated in a three-electrode system using linear sweep voltammetry (LSV) in the solution with 1.0 M KOH as electrolyzer. As shown in Figure 5a, the C-KASO and C-NASO catalysts delivered the highest current densities at a fixed potential, followed by the N-NASO and N-KASO catalysts, whereas the F-NASO and F-KASO catalysts demonstrated the weakest performance. It suggests that metallic cobalt is the most preferable modifier for improving the catalytic activities of feldspar minerals. At an overpotential of 470 mV, the cobalt-modified microcline can boost the current...
densities to over 90 mA cm$^{-2}$. To achieve the required density of 10 mA cm$^{-2}$, only 380 and 400 mV were needed for C-KASO and C-NASO catalysts, respectively, which are better than other modified minerals and the commercial RuO$_2$ catalyst (≈410 mV). Furthermore, only 420 mV is required to achieve a high density of 40 mA cm$^{-2}$ for C-KASO catalyst. Tafel analysis was then used to understand the reaction kinetics and to compare the catalytic activity of these modified feldspar mineral catalysts. Tafel plots can be fitted based on the Tafel equation $\eta = b \log j + a$, where $b$ represents the Tafel slope, and $\eta$ and $j$ stand for the overpotential and the current density, respectively. The Tafel slopes of the C-KASO and C-NASO catalysts were 50.6 and 66.7 mV dec$^{-1}$, respectively, much higher than that of RuO$_2$ catalyst (90.0 mV dec$^{-1}$) (Figure 5b), indicating much improved reaction kinetics toward OER. This performance is much comparable to these Ni- or Co-containing electrocatalysts in the alkaline system (Table S1, Supporting Information).

To confirm its practicability for large-scale application, C-KASO catalyst was loaded onto a piece of carbon cloth (1 $\times$ 1 cm$^2$) and worked as a working electrode for oxygen evolution measurements. As shown in Figure 5c, an overpotential as low as 330 mV was needed to achieve the required density of 10 mA cm$^{-2}$, and the current density reached over 60 mA cm$^{-2}$ at an overpotential of 380 mV. After 2000 cycles, only a slight performance decay was identified. The stability of the C-KASO electrocatalyst was studied by a chronopotentiometry technique.
by monitoring the required potential to produce a constant current density of 10 mA cm$^{-2}$. From the stability test of C-KASO catalyst in Figure 5d, it was found that the overpotential increased at the very initial stage and then stabilized for up to 70 h. These results reveal that the C-KASO catalyst exhibits a good long-term catalytic stability toward OER. Structural examination on C-KASO sample after long-term cycling verified that the percentage of these peaks at higher binding energies increases compared to those at low energies, suggesting the presence of surface oxidization after long-time cycling test in alkaline electrolyte (Figure S1, Supporting Information). Finally, to further verify the fast charge transfer behavior contributed by the metal nanoparticles, the electrochemical impedance spectra (EIS) of C-KASO and P-KASO catalyst-based electrodes were analyzed. As shown in the inset in Figure 5d, a semicircular is identified in the high-frequency region of the Nyquist plot, which is attributed to the charge-transfer resistance ($R_{ct}$) induced by the redox reaction that occurs on the electrocatalyst surfaces and/or interfaces. The estimated $R_{ct}$ values of C-KASO and T-KASO catalyst-based electrodes were 8.5 and 43.0 $\Omega$, respectively, which implies that the metallic Co-modified microcline mineral possesses an improved charge-transfer rate and an enhanced catalytic activity.

3. Conclusions

In summary, three types of transition metal nanoparticles, namely, Ni, Co, and Fe, were in situ implanted to functionalize two feldspar minerals, albite and microcline, for effective electrocatalytic OER, via a simple thermal reduction strategy. The presence of these metal nanoparticles with complex chemical coordination environments is favorable for modulating adsorption behaviors, increasing active sites, and boosting charge transfer during electrocatalytic reactions. Compared with the pristine electrochemical inert aluminosilicate minerals, these modified feldspar minerals exhibit improved catalytic activities. Particularly, the Co-modified microcline mineral (C-KASO) manifests the best performance with an overpotential of 330 mV at a current density of 10 mA cm$^{-2}$ and an excellent activity stability.
for 70 h. This work opens new avenues for rationally utilizing natural available resources to address current energy-related crises and offers a general approach for modifying aluminosilicate minerals by introducing metallic species for electrochemical applications.

4. Experimental Section

**Synthesis of Transition Metal-Modified Aluminosilicate Minerals:** Typically, the pristine microcline (K(AlSi3O8), P-KASO, Keane Ceramics, 100 mg), glucose (100 mg, Sigma-Aldrich), and metal-containing salts (0.34 mol, Co(Ac)2·4H2O, Ni(NO3)2·6H2O or FeCl3·6H2O, Sigma-Aldrich) were uniformly mixed in the presence of deionized water (1.0 mL) and stirred for 5 h. After the removal of water by dried in an oven at 80 °C for 24 h, the resultant mixture powder was calcinated at 900 °C under Ar/H2 flowing for 2 h using a heating rate of 5 °C min⁻¹ to produce the final products, which were labeled as C-KASO, N-KASO, and F-KASO for the Co, Ni, and Fe-modified P-KASO particles, respectively. For reference, the sample without the addition of any metal-containing precursor was synthesized (labeled as T-KASO). Co-modified albite (labeled as C-NASO), Ni-modified albite (labeled as N-NASO), Fe-modified albite (labeled as F-NASO), and referenced treated albite (labeled as T-NASO) samples were synthesized using the similar procedures except for the pristine albite (Na(AlSi3O8), P-NASO, Keane Ceramics) powder as raw material.

**Material Characterization:** Surface morphologies of the transition metal-modified aluminosilicate mineral particles were characterized on a Zeiss field-emission SEM (FE-SEM) equipped with a SE2 detector and a Bruker Icon Dimension atomic force microscope (AFM). The element mapping patterns of the transition metal-modified aluminosilicate mineral particles were recorded on a JEOI 2100 TEM coupled with an energy-dispersive spectrometric detector (EDS) at an accelerating voltage of 200 kV. Crystal structures of the transition metal-modified aluminosilicate mineral particles were examined on a Rigaku SmartLab XRD by using a Cu Kα radiation under a tube voltage and potential of 40 kV and 40 mA, respectively. Valence states of transition metal species in these modified aluminosilicate mineral particles were identified on a Kratos AXIS Supra XPS.

**Preparation of Working Electrode:** To prepare the catalyst ink, a 2 mg of the catalyst powder was dispersed into an ultrapure water and isopropanol (4:1, v/v) mixture with the addition of 5 μL Nafion binder solution under ultrasonic treatment. Then, 4 μL of the mixture was dipped onto a polished glassy carbon (GC) disc electrode (a diameter of ≈3 mm). After completely dried, the modified GC electrode was used as the working electrode for the following electrocatalytic tests.

**Electrochemical Measurements for OER:** Electrochemical measurements were conducted on a CHI 760E electrochemical workstation by using a three-electrode setup, consisting of a working electrode, a graphite rod electrode, and a saturated calomel electrode (SCE) in the 1.0 M KOH aqueous solutions as electrolyzer. LSV plots were obtained at a scan rate of 0.5 mV s⁻¹ after several cyclic voltammetry (CV) cycles for steady states, and the polarization curves were iR-corrected with an automatic compensation level of 95%. All potentials were calibrated to reversible hydrogen electrode (RHE) based on the equation: E(RHE) = E(SCE) + 0.0591 × pH + 0.0967. Chronopotentiometry tests were conducted at a constant current density of 10 mA cm⁻² for 70 h by loading the catalyst onto carbon cloth substrate. EIS were collected on a CHI 604E workstation at an amplitude potential of 5 mV in a frequency range between 100 kHz and 5 mHz.

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

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**Conflict of Interest**

The authors declare no conflict of interest.

**Data Availability Statement**

Research data are not shared.

**Keywords**

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