

Improvement of molybdenum trioxide quality via ultrasound-enhanced water washing: Targeted removal of structural potassium under neutral conditions

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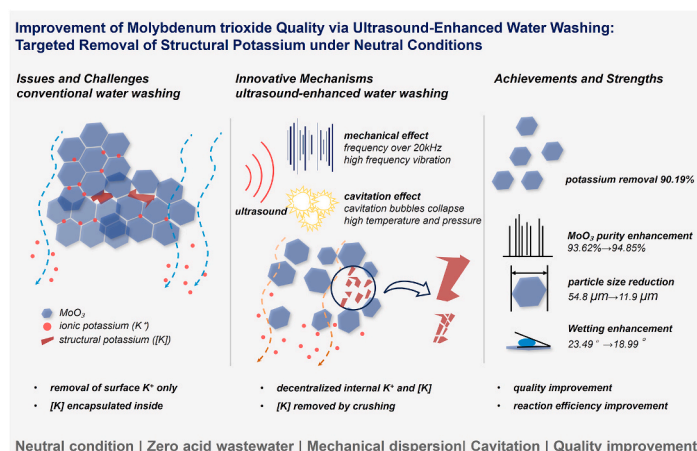
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HIGHLIGHTS

- A green potassium removal technique under neutral conditions was developed.
- A potassium removal efficiency of 90.19 % with minimal molybdenum loss was achieved.
- Potassium impurities were identified to exist in both ionic (K^+) and structural ([K]) forms.
- Ultrasound was demonstrated to be superior in the selective and efficient removal of [K].
- The quality of molybdenum trioxide was improved by the developed technique.

GRAPHICAL ABSTRACT



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ABSTRACT

To address the common drawbacks of high costs, environmental issues, and low potassium removal efficiency in conventional potassium removal processes for molybdenum trioxide (MoO_3), this study introduced a novel ultrasound-enhanced water washing technique under neutral conditions, which significantly reduced potassium impurities in MoO_3 and thereby improved its quality. By combining ultrasonic cavitation and mechanical effects, this technique achieved 90.19 % potassium removal, which was 7.69 % higher than conventional washing, with

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Neutral conditions
Quality improvement

only 5.51 % molybdenum loss. The optimized molybdenum trioxide was in the form of lamellar, the static contact angle was reduced by 19.15 %, and the particle size was reduced by 78 %, which was conducive to the subsequent further processing. Systematic characterization reveals that potassium exists as both ionic potassium (K^+) and structural potassium ([K]) in the MoO_3 raw material. Ultrasound selectively disrupted encapsulated [K] by dispersing aggregated particles and exposing impurities, while neutral conditions eliminated acidic wastewater generation. This technique provided a green and efficient alternative to conventional acid or inefficient water washing, meeting the requirements of sustainable production.

1. Introduction

Molybdenum trioxide (MoO_3) is a vital compound in the molybdenum industry. As a key precursor, it can be processed into high-end molybdenum products such as ammonium molybdate, high-purity MoO_3 , and molybdenum targets (An et al., 2024; He et al., 2024; Zhao et al., 2025). High-quality MoO_3 is crucial for smooth downstream processing and advancing the R&D (research and development), production, and application of advanced molybdenum products (Yao et al., 2016; Zhang et al., 2022).

The vast majority of MoO_3 is obtained from the processing of naturally mined molybdenite (Liu, 2018). During this process, the emergence of various impurities is inevitable. One of the key impurities is potassium, the presence of which has a significant impact on the subsequent processing of MoO_3 (Wang, 2014). In the preparation of high-purity molybdenum compounds or molybdenum-based functional materials such as high-purity molybdenum trioxide and high-purity molybdc acid from MoO_3 , potassium impurities can alter the reaction kinetics, affecting the crystalline behavior and microstructure of the material, and consequently decreasing the purity and homogeneity of the final product (Wang, 2014). In the crystallization process of ammonium molybdate, potassium impurities are prone to compete with ammonium ions for adsorption or co-precipitation, leading to a decrease in the purity of ammonium molybdate products and affecting their application performance in the fields of electronics and chemical industry (Zhang et al., 2021). In the preparation of molybdenum targets, potassium impurities will cause internal structural defects in the target, reducing the target density and sputtering uniformity, thus affecting the quality and performance stability of sputtering coating (Liu et al., 2024; Tian, 2010). Therefore, efficient removal of potassium impurities from MoO_3 is of great importance.

Against this backdrop, traditional methods for removing potassium are confronted with numerous difficulties. In the actual industrial production, for the removal of potassium from MoO_3 , chemical or physical methods such as ion exchange, extraction and the addition of additives are generally not applicable due to the excessively high cost or the introduction of new impurity ions (Sun et al., 2018; Fan et al., 2014). Alkaline washing (e.g., with NaOH solution) reacts with MoO_3 to form soluble molybdates (e.g., Na_2MoO_4), leading to co-dissolution of molybdenum and impurities, which not only results in molybdenum loss but also complicates subsequent separation. Acid washing at a pH of 1 can effectively remove potassium impurities, however, the large amount of acidic wastewater containing Cl^- , SO_4^{2-} , or NO_3^- produced poses significant hazards to both production systems and the environment (Shakib et al., 2025; Wei et al., 2024). Therefore, potassium removal by water washing under neutral conditions has become the current mainstream research direction. Relevant studies on the analysis of MoO_3 raw material have shown that potassium generally exists in two forms. The first type is present in simple ionic compounds (defined in this study as K^+), such as KCl and K_2SO_4 . K^+ is connected to anions by ionic bonds that have no directionality and saturation. Under the polar action of water molecules, the ionic bonds are prone to breakage (Zhou et al., 2020). Therefore, K^+ is relatively easily removed by the water washing method during the impurity removal process. The second type is potassium existing in a structural form in minerals (defined in this study as [K]). In such minerals, [K] is combined with elements like aluminum,

silicon, and oxygen through ionic bonds to form a networked silicate tetrahedral framework, and [K] is located in the "cage" of the tetrahedron to balance the charges in the framework structure (Li et al., 2023; Ma et al., 2017; Mugnaioli et al., 2020). Thus, it is difficult to remove by water washing. Yang et al. attempted to remove potassium from MoO_3 raw material by means of multi-stage water washing and prolonging the water washing time. Under the optimal conditions, the removal degree of potassium merely increased from 63.7 % to 76.6 % (Q. Liu et al., 2023). Owing to the lack of in-depth studies on the form and distribution of potassium (K^+ and [K]) in MoO_3 raw material, coupled with the lack of feasible techniques for the deep removal of difficult-to-water-wash [K], the progress of potassium removal by water washing is still limited.

Ultrasonic waves, which are mechanical waves with frequencies exceeding 20,000 Hz, find extensive applications in a wide range of chemical reactions and physical processes by virtue of their unique mechanical and cavitation effects (Devos et al., 2025; Ileri et al., 2015; Liu et al., 2023). In the domain of impurity removal via the water washing process, many studies have shown that the intensifying effect of ultrasound can remarkably increase the degree of impurity elimination (Di et al., 2024; Liu et al., 2023). The mechanical effect of ultrasound produces high-frequency oscillations that are effective in dispersing or disrupting the complex structure of substances, thereby exposing impurities (Guillemin et al., 2012; Marković et al., 2008; Wang et al., 2023). Concurrently, the cavitation effect produces cavitation bubbles that, upon collapse, generate high-temperature (~ 5000 K) and high-pressure (>100 MPa) reaction micro-regions (Priyadarshi et al., 2021; Suslick et al., 2011; Wang et al., 2024). This optimization of the reaction environment promotes the occurrence of reactions that are otherwise difficult to proceed from the perspectives of kinetics and thermodynamics, enabling them to progress smoothly. In the study of gold leaching from pyrite by Gui et al., the ultrasound-enhanced ozone pretreatment process effectively destroys the structure of pyrite and increases the rate of gold leaching from 86.88 % to 92.06 % (Gui et al., 2022). Ma et al. realized the decomposition of large ore particles into small particles by ultrasound-assisted acid leaching of potassium feldspar, thus making it easier for the acid to penetrate into the ore particles (Ma et al., 2017). Consequently, based on the existing water washing process, it is both feasible and highly valuable to explore the intensifying effect and role of ultrasound in the process of removing potassium and improving the quality of MoO_3 . Additionally, it is convenient for industrial operation and does not give rise to environmental concerns. However, the promoting effect of ultrasound on the potassium removal process during water washing, especially the removal effect on the [K]-type potassium that is structurally bound within the mineral lattice, has not been reported yet.

This study addressed the environmental and efficiency issues identified in previous research on the removal of potassium from MoO_3 and proposed a novel ultrasound-enhanced water washing technique. Systematic analytical methods such as EPMA, SEM-EDS, MLA, XRD and XPS were used to investigate the presence form and distribution of potassium in the MoO_3 raw material. The degrees of potassium removal and molybdenum loss were evaluated during both the ultrasound-enhanced water washing process and the conventional water washing process under varying conditions of water washing temperature, time, liquid-solid ratio, and ultrasonic power, with a focus on comparing the two, to obtain the optimal process parameters. The removal mechanism of

different types of potassium by ultrasound was explored, the migration behavior of these potassium types was defined, and the intensifying mechanism of ultrasound in the potassium removal process via water washing was disclosed. The aim of this study is to provide a viable industrial application template for the potassium removal process in the actual industrial water washing of MoO_3 , which not only improves the quality of MoO_3 but also aligns with both corporate economic benefits and social environmental protection requirements.

2. Experimental

2.1. Materials

The MoO_3 raw material used in this study was sourced from an ammonium molybdate production facility in Shaanxi, China. The production process involves water-washing purification of MoO_3 followed by ammonia leaching to obtain an ammonium molybdate solution, which subsequently undergoes crystallization. X-ray fluorescence spectrometer analysis of the raw MoO_3 (Table 1) shows that the primary component is Mo, while impurities exceeding 0.1 % include Si, Fe, Al, Cu, Ca, and K. During ammonia leaching, other impurities such as Fe, Si, and Al form insoluble hydroxides/oxides (e.g., $\text{Fe}(\text{OH})_3$ and $\text{Al}(\text{OH})_3$) that are effectively retained in the leaching residue, whereas potassium dissolves into the ammonium molybdate solution in ionic form. This dissolved potassium significantly impacts crystallization behavior by altering crystal growth kinetics and inducing microstructural defects, ultimately compromising product purity and homogeneity. Furthermore, failure to remove potassium prior to ammonia leaching necessitates additional costly purification steps (e.g., ion exchange or solvent extraction), substantially increasing production costs and posing major industrial processing challenges. Therefore, enhancing removal of potassium impurities is the focus of this study.

All experimental procedures utilized distilled water prepared in the laboratory.

2.2. Experimental mechanisms and procedures

As illustrated in Fig. 1, the ultrasound-enhanced washing process combines cavitation and mechanical effects to selectively remove both ionic (K^+) and structural ([K]) potassium impurities. The collapse of cavitation bubbles generates localized high-pressure microjets, disrupting aggregated MoO_3 particles and exposing encapsulated [K], while mechanical vibrations further disperse the particles, facilitating impurity dissolution under neutral conditions.

The ultrasound-enhanced water washing process of the MoO_3 raw material was carried out as follows (as shown in Fig. 2a): During the experiment, 50 g of MoO_3 raw material was placed into a 200 mL beaker each time. A magnetic rotor and distilled water were added, and then the beaker was sealed with plastic wrap to prevent water evaporation. The beaker was placed in a constant temperature water bath equipped with magnetic stirring, and the stirring rate was adjusted and fixed at 200 rpm. In this study, the ultrasonic device (20 kHz, maximum 1800 W) was operated in continuous-wave mode throughout all experiments. This device was independently developed by the Key Laboratory of Unconventional Metallurgy, Ministry of Education, Kunming University

of Science and Technology (as shown in Fig. 2b). The ultrasonic transducer was vertically placed in the beaker, approximately 1 cm from the bottom. By adjusting parameters such as treatment time (ranging from 30 to 120 min), temperature (ranging from 50 to 90 °C), liquid-solid ratio (ranging from 1:1 to 3:1), and ultrasonic power (ranging from 150 to 1650 W), various condition tests were conducted. After sufficient water washing, the sample in the beaker was transferred to a filtration device connected to a vacuum pump for filtration. Subsequently, various analyses were performed on the filtrate and the residue. The water washing process of MoO_3 under conventional conditions was the same as the above except that the ultrasonic power was set at 0 W. Each experiment was conducted in triplicate to ensure data reliability.

2.3. Analytical methods

The contents of different elements in the samples were determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Agilent 5110, USA). Each set of data was tested three times and the average value was taken. The phases of the different samples were investigated by a powder X-ray diffractometer (XRD, Rigaku dX 2000, Japan) with an operating voltage of 40 kV and an operating current of 25 mA, using $\text{Cu-K}\alpha$ as the ray source. The scanning angle of the XRD ranged from 5 to 90° and the scanning speed was 2°/minute. The XRD data were analyzed using MDI Jade software. X-ray photoelectron spectroscopy (XPS, PHI PHI-5300, USA) was used to analyze the binding energies of different elements in samples, with an operating voltage of 12 kV and an operating current of 6 mA, using $\text{Al-K}\alpha$ as the ray source. The results detected by the XPS instrument were analyzed using Avantage software and calibrated at the C1s peak position at 284.8 eV. The types and contents of elements in MoO_3 raw material before and after water washing were analyzed using an X-ray fluorescence spectrometer (XRF, Rigaku ZSX Primus, Japan). The operating voltage was 50 kV, and the operating current was 60 mA. The test diameter was 30 mm. The X-ray tube used a Rh target with a maximum power of 4 kW. The range of elements analyzed was from 9F to 92U, using a scintillation counter (SC) and a proportional counter (PC). Scanning electron microscope-energy dispersive spectrometer (SEM-EDS, ZEISS Sigma 300, Germany) was utilized to investigate the microscopic morphologies and elemental distributions of different samples. The accelerating voltage for morphology imaging was 3 kV, and the accelerating voltage for energy spectrum mapping was 15 kV. The detector was an SE2 secondary electron detector. Different samples were embedded in resin, sectioned and polished, and then electron probe microanalysis (EPMA, JEOL JXA-iHP200F, Japan) was used to analyze the microscopic morphologies, elemental distribution and contents of the sections. The electron gun measures standard samples at fixed voltages (10 kV and 15 kV) and fixed currents (10 mA and 20 mA). Under the same conditions, the characteristic X-ray intensities of elements in different samples were measured, compared with standard samples, and corrected and calculated to obtain the mass fractions of the elements. Mineral liberation analysis (MLA, ZEISS Sigma 300, Germany) and automated mineralogy information classification system (AMICS) were employed to determine the mineral phase compositions and contents of different samples. A laser particle size analyzer (Malvern Mastersizer 3000+ Ultra, UK) was adopted to measure the particle size and its distribution of the samples with water as the dispersion medium. After being pelletized, different samples were tested for static contact angles using a contact angle measuring instrument (Dataphysics OCA 20, Germany) with the sessile drop method (with water as the medium). To ensure data quality, all instruments were calibrated prior to use.

The removal degree of **potassium** or the loss degree of **molybdenum** was calculated by Equation (1).

$$\text{Degree of removal(loss)}(\%) = C_t \times V_t / m_0 \times \omega_0 \times 100\% \quad (1)$$

where C_t represents the concentration of elements (g/L) in the filtrate

Table 1

XRF detection results of different element contents in MoO_3 raw material.

Element	Content (wt. %)	Element	Content (wt. %)
Mo	61.15	Th	0.09
Si	2.19	Pb	0.06
Fe	1.19	Zn	0.05
Al	0.59	Mg	0.09
Cu	0.24	Ti	0.03
Ca	0.36	Mn	0.02
K	0.13	Bi	0.02

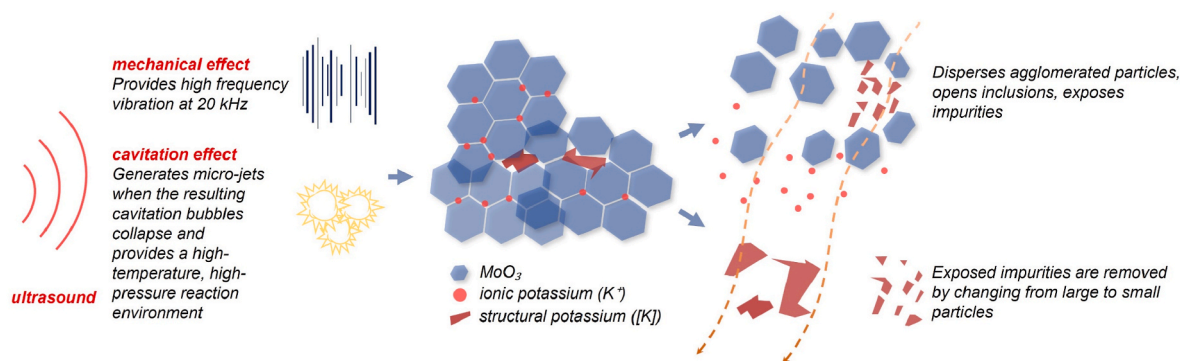


Fig. 1. Schematic diagram of the ultrasound-enhanced water washing mechanism for potassium removal from MoO₃.

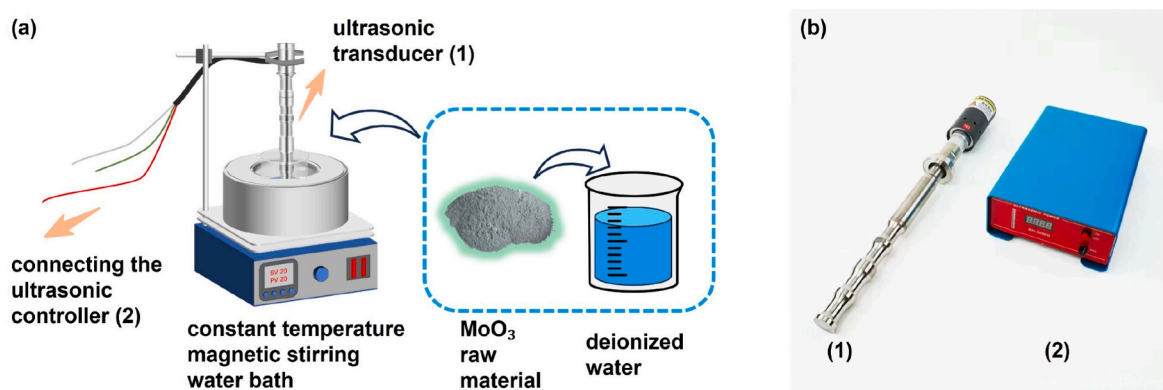


Fig. 2. Flow chart of the experiment (a), the ultrasonic equipment used; (b), where (1) is the ultrasonic transducer and (2) is the ultrasonic control unit.

after water washing, and V_f represents the volume (L) of the filtrate. Meanwhile, m_0 represents the mass of the initial MoO₃ raw material (g) taken before water washing, and ω_0 represents the element content (%) in the MoO₃ raw material (Upon testing, the content of molybdenum is 61.15 %, and the content of potassium is 0.13 %).

3. Results and discussion

3.1. Forms of potassium impurities

Mineral liberation analysis (MLA) is a method for liberating minerals and analyzing their composition. The Automated Mineralogical Information Classification System (AMICS) is a software system that, when used in conjunction with MLA, can automatically identify and quantitatively analyze the mineral composition of a sample (Zhang et al., 2019). The analysis results for MoO₃ raw material are shown in Table 2 (only mineral information related to the removal of potassium is listed in this table for comparison purposes, and a complete mineral analysis table is provided in Table S1 in the Supplementary Material). The mass fractions of potassium feldspar and biotite in the MoO₃ raw material confirm the presence of [K]. The potassium feldspar content is 1.19 %, and the biotite content is 0.15 %. Fig. 3a presents the cross-sectional view of the MoO₃ raw material. Besides MoO₃, the exterior of [K]

(potassium) is also surrounded by other minerals such as MoSiO₃ (molybdenum silicate) and Al₂Si₂O₅(OH)₅ (aluminum silicate hydroxide). These minerals aggregate and encapsulate the already challenging-to-treat [K] within, increasing the difficulty of removal. This analysis provides direct evidence for the presence of [K] in the MoO₃ raw material.

The XRD patterns are shown in Fig. 3b, along with the standard PDF reference patterns for MoO₃ (PDF#97–015–8256), SiO₂ (PDF#97–003–9830), and KAlSiO₄ (PDF#00–048–1028). The MoO₃ raw material sample showed characteristic peaks of KAlSiO₄, indicating the presence of [K] in MoO₃. This is consistent with the conclusions of MLA-AMICS. It is worth noting that the presence of K⁺ (such as KCl) was not detected in the XRD detection. This might be due to the low content of potassium, which fails to reach the detection limit of XRD. As for [K], its presence can be indirectly detected by detecting elements such as Si with relatively high contents.

The XPS results of MoO₃ raw material are shown in Fig. 3c–e. The K 2p spectra (Fig. 3c) revealed that the characteristic peaks of KCl (binding energies of 295.6 ± 0.1 eV and 292.7 ± 0.1 eV) appeared in the MoO₃ raw material (Ma et al., 2016; Qiao et al., 2021), indicating that a portion of the potassium impurities in the MoO₃ raw material exists in the form of K⁺. By analyzing the Si 2p spectra (Fig. 3d), the characteristic peak of SiO₂ was found at a binding energy of 103.9 ± 0.2 eV, and the characteristic peak of aluminosilicate was found at a binding energy of 103.1 ± 0.2 eV (Dubey et al., 2009; Li et al., 2024; Mugnaioli et al., 2020; Péliouss-Schecker et al., 2012). This is consistent with the detection of KAlSi_xO_y by MLA-AMICS and XRD. XPS analysis of Mo (Fig. 3e) in MoO₃ raw material shows binding energies of 236.0 ± 0.1 eV (Mo 3d_{5/2}) and 239.3 ± 0.1 eV (Mo 3d_{3/2}), which correspond to Mo in the +6-oxidation state and are consistent with the valence state of Mo in MLA-AMICS. It is demonstrated that the Mo of the material is in a stable and homogeneous oxidation state.

Table 2
Major minerals in MoO₃ raw material analyzed by MLA-AMICS (wt.%).

Name	Chemical formula	Mass fraction
Molybdenum trioxide	MoO ₃	90.64
Quartz	SiO ₂	2.37
Potassium feldspar	KAlSi ₃ O ₈	1.19
Biotite	K(Mg,Fe) ₃ (AlSi ₃ O ₁₀)(F,OH) ₂	0.15
Other minerals	–	5.65

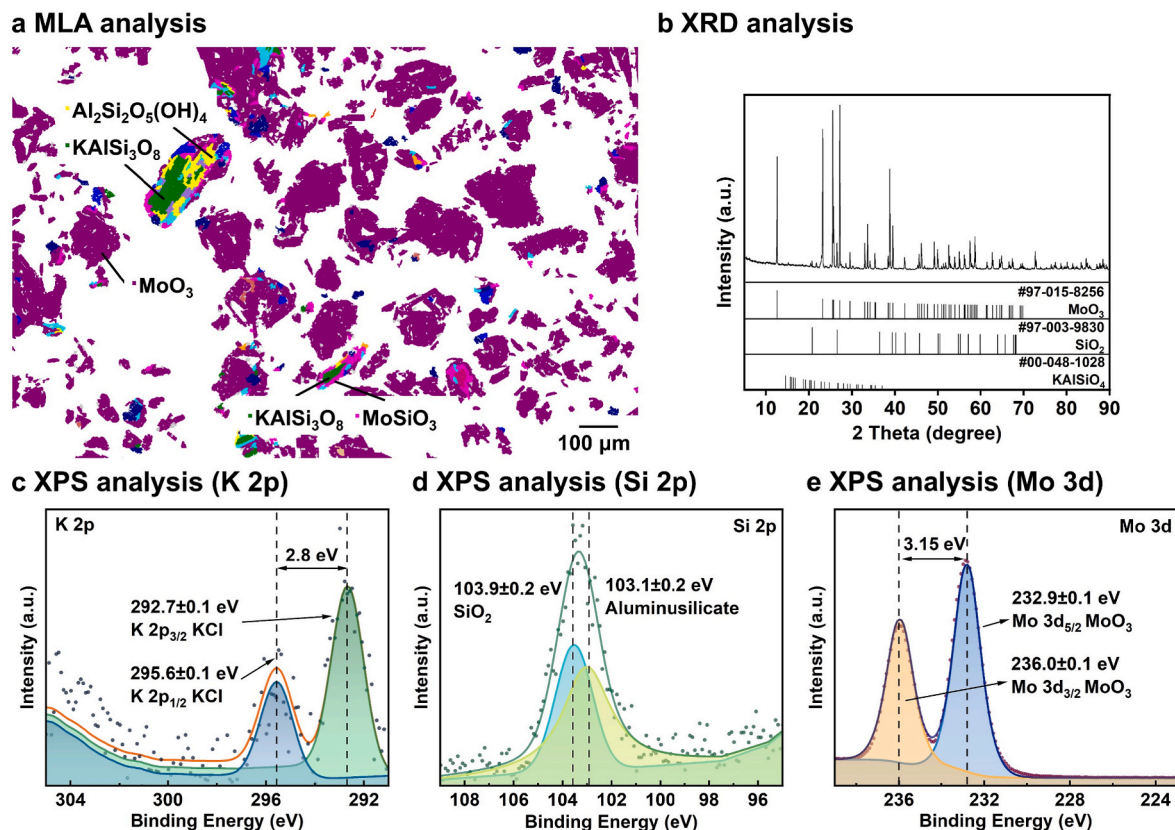


Fig. 3. Different analyses of MoO₃ raw material: (a) MLA result; (b) XRD result; (c) K 2p results of XPS analysis; (d) Si 2p results of XPS analysis; (e) Mo 3d results of XPS analysis.

The above analysis demonstrates that potassium in the **MoO₃ raw material** exists in two forms: as structural potassium ([K]) within minerals like potassium feldspar and biotite, and as ionic potassium

(K⁺). This characterization is supported by MLA-AMICS, XRD, and XPS results.

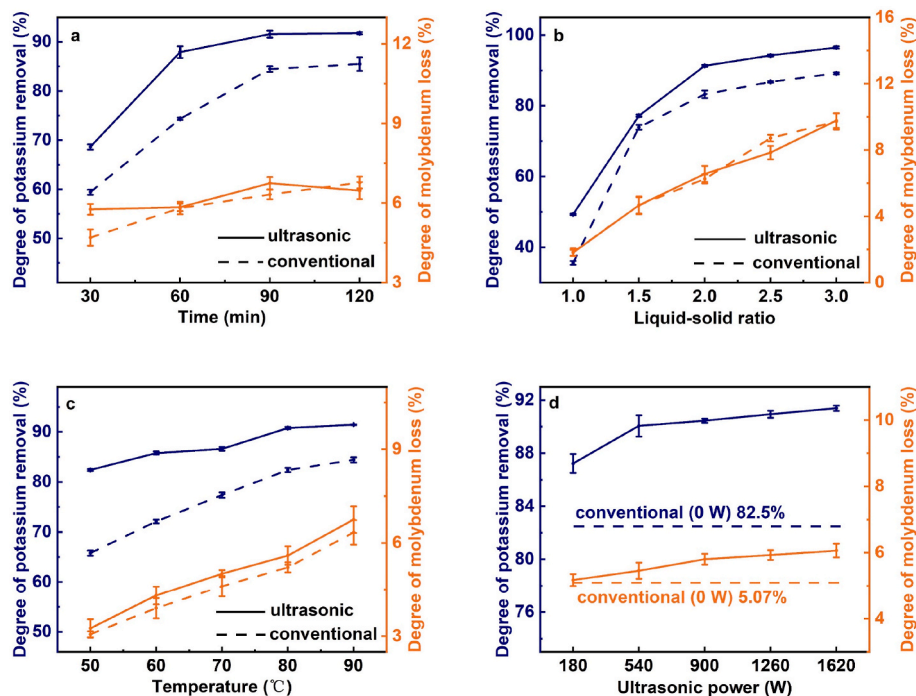


Fig. 4. Effect of different factors on the removal (loss) degree of potassium and molybdenum (data are presented as mean ± standard deviation, *n* = 3): (a) time; (b) liquid-solid ratio; (c) temperature; (d) ultrasonic power.

3.2. Effect of ultrasound-enhanced potassium removal under different conditions

3.2.1. Effect of time

The water washing process was carried out at a temperature of 90 °C, a liquid-solid ratio of 2:1, and an ultrasonic power of 1260 W (Fig. 4a). Under the continuous action of ultrasound, the removal degree of potassium consistently exceeds that under conventional conditions. According to the research by Lian et al., the cavitation effect of ultrasound has been found to generate high-speed microjets, effectively dispersing potassium impurities that are attached to the MoO₃ structure or encapsulated within it (Lian et al., 2021). The high-frequency mechanical vibrations of ultrasound (mechanical effect) also enhance the process of dispersing or opening encapsulated structures (Vikash and Kumar, 2020). Additionally, ultrasound produce cavitation bubbles in the liquid medium (Devos et al., 2025). When these bubbles collapse near the surface of MoO₃, they create intense local pressure and temperature changes, providing conditions for the removal of difficult-to-water-wash [K]. In contrast, conventional water washing relies solely on mechanical stirring, which has low mass transfer efficiency and cannot provide sufficient energy to remove potassium encapsulated within the internal structure, nor can they remove the difficult-to-water washing [K]. Therefore, the rate of increase in potassium removal is relatively slow. Regarding the loss degree of molybdenum, both water washing methods exhibit relatively low levels. This can be attributed to the chemical stability of molybdenum in the reaction system. According to the report by De Castro et al., molybdenum forms stable chemical bonds (covalent bonds) within molybdenum's oxide structure, the potassium impurities primarily targeted by ultrasound are typically located at the grain boundaries of MoO₃, and the energy of ultrasound is not sufficient to break the strong chemical bonds of molybdenum, resulting in minimal molybdenum loss (De Castro et al., 2017). By contrast, the gentler conventional water washing conditions are even less likely to cause significant decomposition or dissolution of MoO₃. From an economic perspective, the optimized water washing time is 90 min.

3.2.2. Effect of liquid-solid ratio

The effect of different liquid-solid ratios on the removal (loss) degree of potassium and molybdenum was investigated at a water washing temperature of 90 °C, a time of 90 min and an ultrasonic power of 1260 W. The results are shown in Fig. 4b. As the liquid-solid ratio increases, the removal of potassium improves in both ultrasonic and conventional water washing processes. For the ultrasound-enhanced water washing process, a higher liquid-solid ratio not only enhances the dispersion of MoO₃ but also provides more space for the formation and collapse of cavitation bubbles (Devos et al., 2025). Research by Meroni et al. has shown that in systems with a higher liquid-solid ratio, the enhanced dispersion of solid particles reduces the propagation resistance of ultrasound, allowing energy to be more evenly distributed throughout the liquid phase (Meroni et al., 2022). This facilitates the uniform application of the cavitation and mechanical effects of ultrasound on more MoO₃ particles. Consequently, the microjets and shock waves generated by cavitation bubbles can more effectively remove potassium impurities from the surface and interior of MoO₃. In the conventional water washing process, a higher liquid-solid ratio increases the contact area between the liquid and MoO₃, thereby increasing the degree of potassium removal. The loss degree of molybdenum under both water washing methods increases gradually with the increase in the liquid-solid ratio. This is because the dispersed particles and the increased solid-liquid contact area also promote the dissolution of molybdenum into the liquid phase. Considering water consumption costs and potassium removal efficiency, the optimized liquid-solid ratio is 2:1.

3.2.3. Effect of temperature

As shown in Fig. 4c, the relationship between the removal (loss)

degree of potassium and molybdenum and the water washing temperature was examined at a time of 90 min, a liquid-solid ratio of 2:1, and an ultrasonic power of 1260 W. As the temperature increases, the removal degree of potassium in both ultrasonic and conventional water washing processes shows an upward trend. In the ultrasound-enhanced water washing process, the rise in temperature leads to a decrease in the surface tension of the liquid medium and an increase in the vapor pressure inside the cavitation bubbles. This facilitates the formation of cavitation bubbles and causes them to collapse more violently, generating stronger microjets and shock waves (Contamine et al., 1995; Hong and Son, 2023; Osterman et al., 2009). These enhanced physical effects are conducive to the detachment of potassium impurities from MoO₃ at higher temperatures. In the conventional water washing, the increase in temperature accelerates the kinetic energy of the molecules (Aquilanti et al., 2018; Li and Merz, 2022), thereby increasing the potassium removal rate. Regarding the loss degree of molybdenum, both water washing methods maintain relatively low levels. MoO₃ exhibits high thermal stability, maintaining its chemical structure within a certain temperature range. Additionally, as the temperature increases, the solubility of MoO₃ also increases (from 0.138 g/L at 20 °C to 2.106 g/L at 80 °C), thus the loss degree of molybdenum will slowly increase. The optimized temperature is 80 °C.

3.2.4. Effect of ultrasonic power

The effect of ultrasonic power on the removal (loss) degree of potassium and molybdenum was investigated at a water washing time of 90 min, a liquid-solid ratio of 2:1, and a temperature of 80 °C, and the results are shown in Fig. 4d. As the ultrasonic power increases, the intensity of the cavitation and mechanical effects also increases (Marković et al., 2008). This not only aids in the removal of potassium but also promotes the loss of molybdenum. Consequently, both curves exhibit an upward trend. Due to the threshold of the cavitation effect of ultrasound, and the increasingly stringent reaction environment required for the removal of the remaining small portion of difficult-to-water-wash [K], the enhancement in potassium impurity removal becomes limited when the ultrasonic power reaches 540 W. It only increases from 90.19 % at 540 W to 91.17 % at 1620 W. In addition, the high ultrasonic power brings additional power consumption as well as molybdenum losses, and therefore the optimized ultrasonic power is 540 W.

3.2.5. Solid-phase characterization

Fig. 5a shows the morphologies of the three samples, with the ultrasonically water washed sample primarily exhibiting a lamellar form, while the other two samples are in the form of blocks or block-lamellar aggregates, which corroborates the XRD findings. The lamellar form has the advantage of being less prone to agglomeration, which can prevent issues with reduced reaction effectiveness due to agglomeration. As shown in Fig. 5b, the static contact angle of the ultrasonically water washed sample is 18.99°, which is a 19.15 % reduction compared to that of the MoO₃ raw material and a 16.78 % reduction compared to that of the conventionally water washed sample. A smaller contact angle indicates better wettability, so the MoO₃ raw material, after ultrasound-enhanced water washing, will have better contact with the solution in subsequent leaching and other solid-liquid reactions. As shown in Fig. 5c, the particle size of the ultrasonically water washed sample is 11.9 μm, which is a nearly 78.28 % reduction compared to the 54.8 μm of the MoO₃ raw material and a 20.67 % reduction compared to the 15.0 μm of the conventionally water washed sample. The significantly reduced particle size leads to a substantially larger specific surface area, enhancing contact with the solution and providing more reactive sites during the reaction process. This is particularly beneficial in catalytic reactions where the active sites on the catalyst's surface interact with reactant molecules. The increased surface area facilitates more frequent and effective interactions, thereby improving the catalytic efficiency (Sharma et al., 2021). In addition, reducing the particle size of MoO₃ enables higher specific capacitance and better capacitance retention

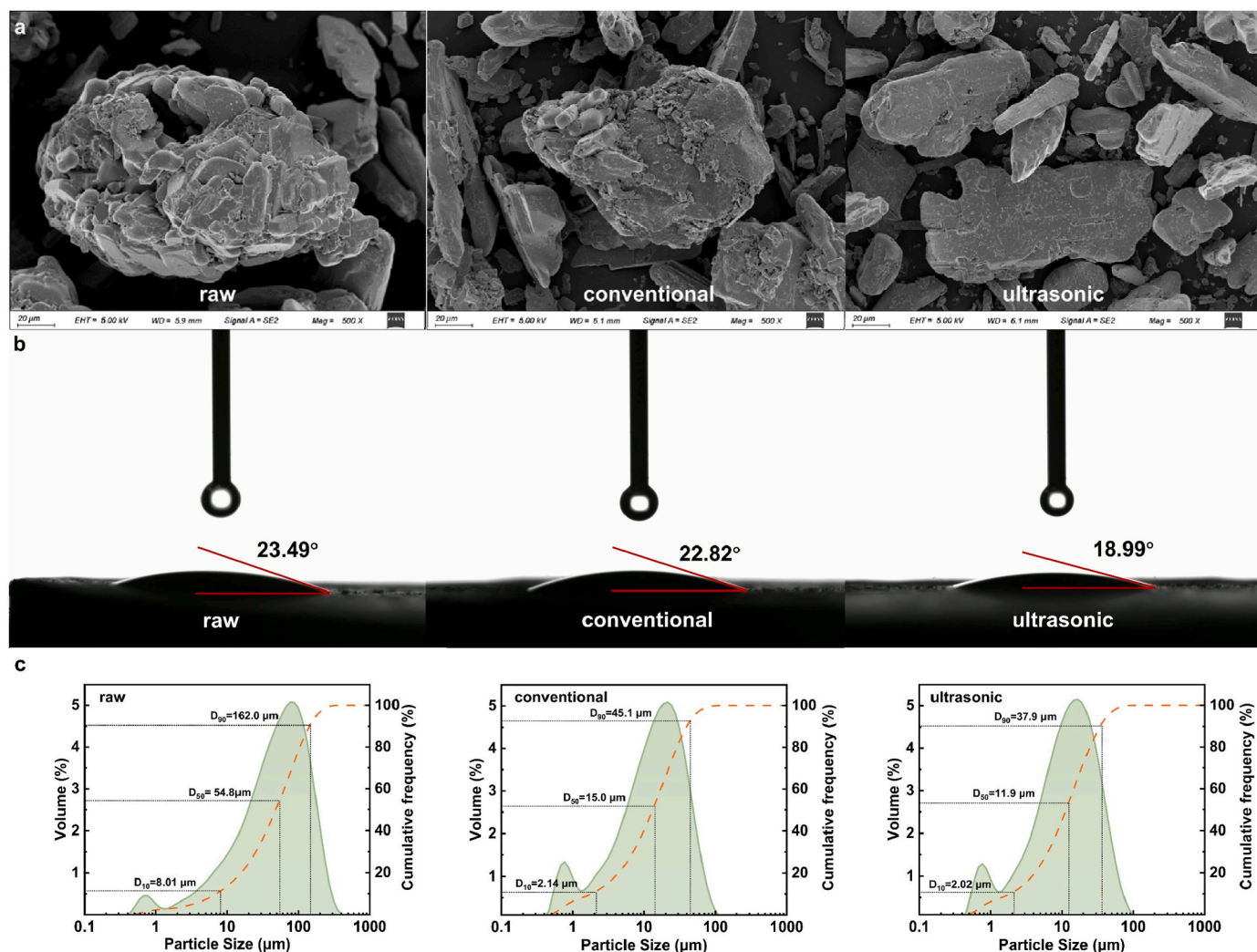


Fig. 5. Different characterization results for MoO₃ raw material, conventionally water washed samples and ultrasonically water washed samples: (a) SEM analysis results; (b) static contact angle analysis results; (c) particle size analysis results.

performance in supercapacitors (Alam et al., 2022).

In summary, the optimized conditions and effects of the ultrasound-enhanced water washing technique were summarized in Table 3. Under neutral conditions, the potassium removal rate reached 90.19 %, which is 7.69 % higher than the traditional water washing method (82.5 %), while ensuring that the molybdenum loss rate was as low as 5.51 %. This technique not only significantly improves the degree of potassium removal, but also promotes the lamellar morphology of MoO₃ and optimizes the contact angle and particle size. Compared with the acid-base treatment methods used in other studies, this technology operated under neutral conditions and had significant environmental and economic

Table 3

Summary of optimization washing parameters and corresponding performance indicators.

Category	Conventional water washing	Ultrasound-enhanced water washing
Optimal conditions	90 min, 80 °C, liquid-solid ratio 2:1, 540 W (only ultrasound-enhanced water washing)	
Potassium removal	82.5 %	90.19 %
Molybdenum loss	5.07 %	5.51 %
Microscopic morphology	agglomerated	lamellar
Static contact angle	22.82°	18.99°
Particle size (D ₅₀)	15.0 μm	11.9 μm

advantages. By completely avoiding the production of acidic/alkaline wastewater, it significantly reduced treatment costs and operational complexity in industrial applications. The process only introduces minimal additional energy consumption from ultrasonic equipment, representing an insignificant fraction of the total operational expenses, while delivering substantially enhanced potassium removal efficiency that reduces downstream purification costs. These combined benefits make the technique both environmentally sustainable and economically advantageous. Industrial applications have also demonstrated that the ultrasonic technique provides consistent economic benefits (Ran et al., 2024). It is important to note that corrosion-resistant materials, optimized ultrasonic power and regular maintenance should be implemented to ensure the long-term operational stability and reliability of ultrasonic equipment.

3.3. Mechanism of ultrasound-enhanced removal of different forms of potassium

3.3.1. SEM-EDS analysis

SEM-EDS analysis provides valuable insights into the microstructural changes and surface elemental distribution of MoO₃ raw material during the ultrasonic or conventional water washing processes.

Fig. 6a shows the microscopic morphology of the MoO₃ raw material. It can be seen that small mineral particles of different shapes aggregate

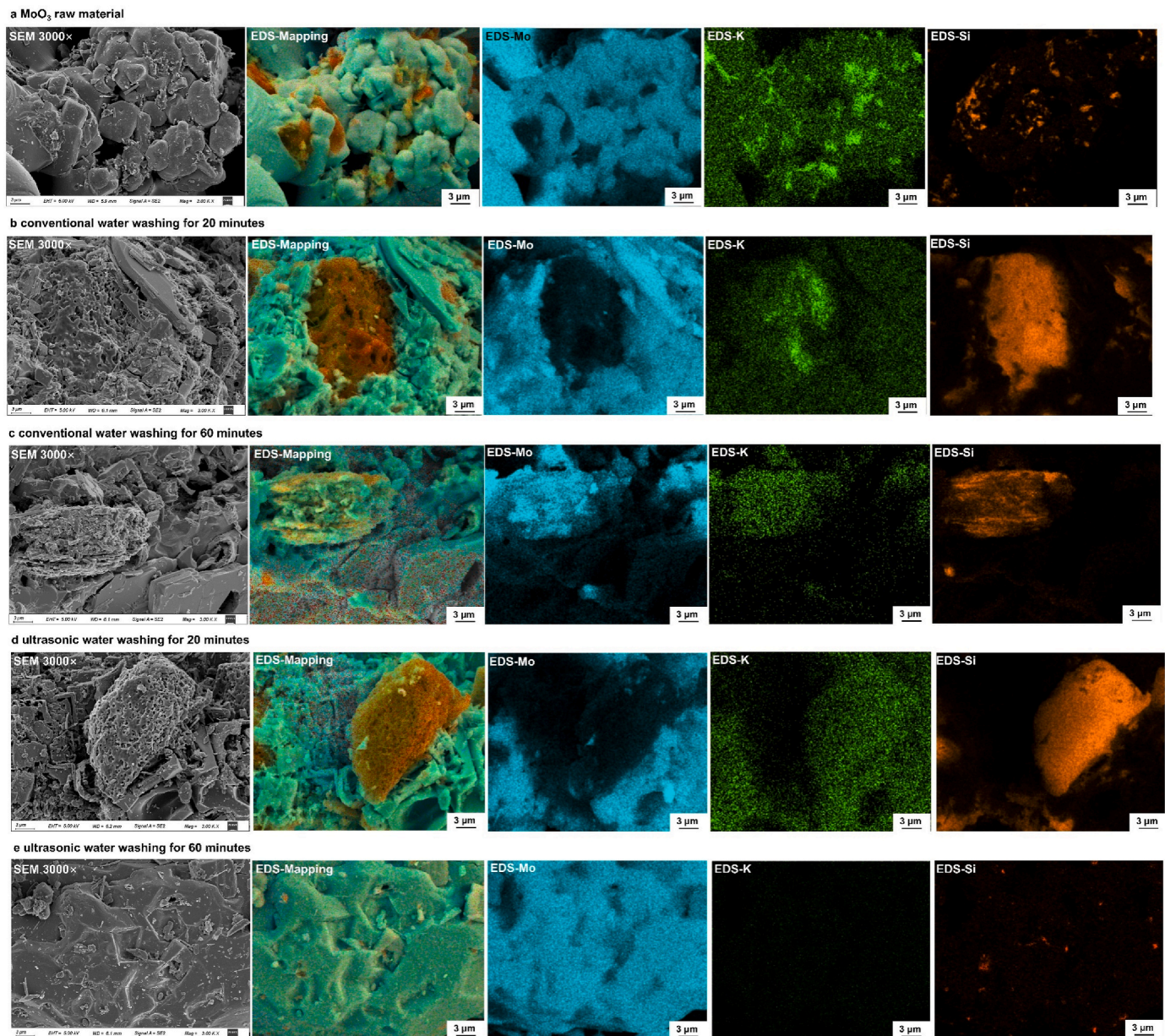


Fig. 6. Microscopic morphology and element distribution results of different samples: (a) MoO_3 raw material; (b) conventional water washing for 20 min; (c) conventional water washing for 60 min; (d) ultrasound-enhanced water washing for 20 min; (e) ultrasound-enhanced water washing for 60 min.

to form large particles with rough and uneven surfaces and complex structures such as stacking and wrapping. EDS-mapping analysis shows that the aggregated small mineral particles are MoO_3 , while potassium and silicon are distributed in the interstices and even inside the MoO_3 particles. It is noteworthy that part of the potassium in the EDS diagram overlaps with silicon, while the other part of potassium is present alone and dispersed at different locations. This intricate structure offers abundant sites for the adsorption or encapsulation of impurity potassium, simultaneously posing significant challenges to the removal of potassium during the water washing process.

The results after conventional water washing for 20 and 60 min are shown in Fig. 6b and c, respectively. After 20 min of conventional water washing, it is evident that potassium is encapsulated within the outer layer of MoO_3 . Following 60 min of conventional water washing, the potassium is separated from the MoO_3 and becomes exposed on the surface. The results after ultrasound-enhanced water washing for 20 and 60 min are depicted in Fig. 6d and e, respectively. After 20 min of ultrasound-enhanced water washing, the potassium has been exposed to

the surface and the outer layer is unobstructed by inclusions. When subjected to ultrasound-enhanced water washing for 60 min, the surface structure of MoO_3 becomes smooth, with no complex stacking or encapsulation structures present, and no significant aggregation or adsorption of potassium is observed within this area.

From the SEM-EDS results, it can be seen that the conventional water washing method is difficult to effectively treat potassium located in complex structures, especially potassium encapsulated in the interior. Ultrasound-enhanced water washing, relying on its unique cavitation effect and mechanical effect, has a significant destructive effect on complex structures. Wang et al. proposed a modified model of the Rayleigh-Plesset equation for the collapse of cavitation bubbles, and demonstrated that transient high pressures (>100 MPa) are generated upon the collapse of cavitation bubbles (Wang et al., 2024). This phenomenon can disperse the aggregation of minerals and open the encapsulations, thereby exposing the impurities inside. Additionally, a reactive microregion up to about 5000 K is formed around the cavitation bubble after its collapse (Suslick et al., 2011), promoting potassium

release into the solution and enhancing its removal.

3.3.2. Electron probe microanalysis (EPMA) analysis

The electron probe microanalysis (EPMA) detection method has high sensitivity and accuracy for detecting trace elements, allowing for the accurate determination of elements with very low concentrations in samples. The three samples were embedded in resin and then cut. Through EPMA analysis, the microstructure and elemental distribution of the cross-sections of different samples were examined. This method can detect the impurities encapsulated within a material.

Fig. 7a illustrates a profile of the MoO_3 raw material. The aggregation of small particles of MoO_3 minerals into large particles is demonstrated. By analyzing the elemental composition at position 1#, the contents of K, Si, Al, and O are 12.34 %, 30.89 %, 9.91 %, and 46.86 %, respectively (see Table 4), which proves that the minerals in this area are $\text{KAl}_x\text{Si}_y\text{O}_z$ composed of [K], which is encapsulated in the inner part of MoO_3 raw material and is distributed in a concentrated manner. The elemental content of position 2# was found to be 0.13 % for K and 0.12 % for Cl. This proves that there is KCl composed of K^+ in this area, which can be seen in the image in the gap between the particles, and the distribution is more dispersed.

The profiles of the samples after 60 min of conventional water washing are shown in Fig. 7b. The structure of the large particles formed by aggregation is clearly not as compact as that in Fig. 7a, and the small trioxide particles begin to slowly disperse from the outside to the inside. The small MoO_3 particles around the minerals consisting of [K] in the interior (positions 3# and 4#) likewise begin to disperse, but remain encapsulated. Elemental analysis of position 5# revealed a K content of 0.03 % and a Cl content of 0.02 %, indicating a decrease in Cl. This proves that the dispersion of the particles has made the intergranular gaps wider, which provides favorable conditions for the removal of K^+ entrapped in the gaps.

The profiles of the samples after 60 min of ultrasound-enhanced water washing are shown in Fig. 7c. Compared to Fig. 7a and b, it can be clearly seen that even the small particles of MoO_3 were cracked under the intensification of ultrasound, which caused the small particles of MoO_3 to be further fragmented. K and Cl were not detected by elemental

Table 4

Elemental mass fractions at different points in EPMA analysis (wt.%).

No.	K	Si	Mo	Cl	Al	O
1#	12.34	30.89	–	–	9.91	46.86
2#	0.13	–	67.61	0.12	–	32.14
3#	0.48	27.19	22.41	–	0.74	49.19
4#	0.35	43.79	–	–	0.19	55.67
5#	0.03	–	67.32	0.02	–	32.63
6#	–	–	67.67	–	–	32.33
7#	–	42.69	–	–	–	57.31

analysis of position 6#, and the contents of K and Cl in the face-scan results were much lower than those of the MoO_3 raw material and the samples obtained after conventional water washing. This proves that the dispersing effect of ultrasound is very significant and that the K^+ present in the interstitial space of the particles is removed more thoroughly. Meanwhile, the presence of [K] was detected at position 7#, when [K] was already located at the edge (surface) of the MoO_3 particles.

The mechanism of K^+ removal from MoO_3 raw material was demonstrated by the analysis of three sample profiles. K^+ are dispersed and present in the interstices between MoO_3 particles that have aggregated to form larger grains. Conventional water washing can only work from the outside in, gradually loosening the MoO_3 particles layer by layer. Once the particles are loosened, the enlarged interstices expose the K^+ encapsulated within, allowing for further removal. Ultrasound-enhanced water washing, which relies on cavitation and mechanical effects, not only intensifies the process of separation between MoO_3 particles but also further fragments the MoO_3 particles. The process of transitioning from aggregated large particles to dispersed small particles and then to fragmented small particles significantly enhances the removal of K^+ from within.

3.3.3. Mineral liberation analysis (MLA)

The three prepared samples (selected and prepared using the same methods as described in Section 3.3.1) were analyzed using MLA to assess the release of potassium-bearing minerals from the MoO_3 structure. AMICS can be utilized to identify and quantify the potassium

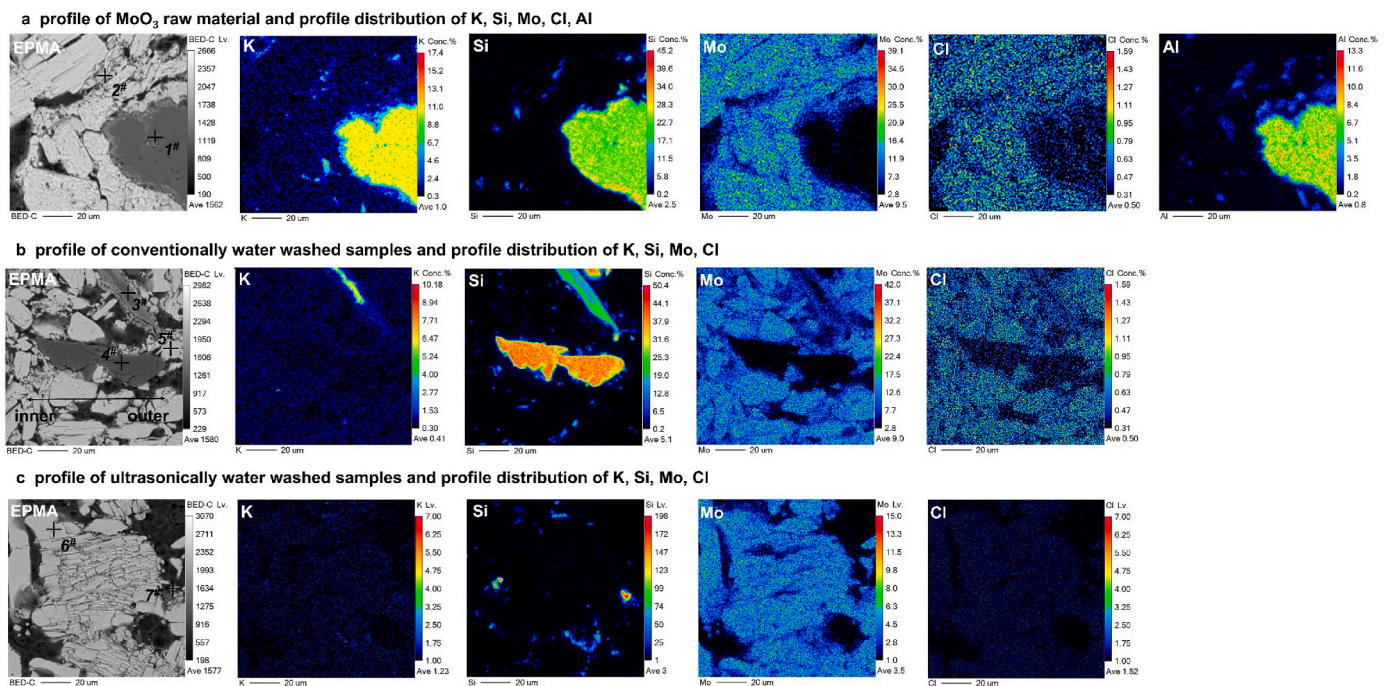


Fig. 7. EPMA analysis results of different samples: (a) profile of MoO_3 raw material and profile distribution of K, Si, Mo, Cl, Al; (b) profile of conventionally water washed samples and profile distribution of K, Si, Mo, Cl; (c) profile of ultrasonically water washed samples and profile distribution of K, Si, Mo, Cl.

impurities within the MoO_3 structure before and after the water washing process.

Fig. 8a shows the cross-section of the sample after conventional water washing, where it is evident that the mineral particles have become smaller. This is attributed to the conventional water washing process, which removes some of the outer minerals, gradually exposing the [K] inside. However, it can be seen that some [K] is still encapsulated, indicating that the dispersing capability of conventional water washing is limited. The situation after ultrasound-enhanced water washing is shown in Fig. 8b. The mineral particles obtained after ultrasound-enhanced water washing are significantly smaller than those of the previous two methods are, proving that the dispersing effect is the best among the methods. The encapsulations of [K] are completely opened, allowing [K] to be fully exposed. The breakdown of shells such as MoSiO_3 during ultrasonic treatment enhances potassium removal while causing minor molybdenum loss.

Combined with AMICS analysis, the potassium mineral content in conventionally washed and ultrasonically washed samples was obtained, as shown in Table 5 (complete mineral analysis table is provided in Table S1 in the Supplementary Material). Both conventional and ultrasound-enhanced water washing have a promoting effect on the removal of [K]. It is apparent that the intensifying effect of ultrasound-enhanced water washing significantly enhances the removal effect of [K]. After organizing the data, Fig. 9 was obtained, which shows that the sum of the two minerals composed of [K] accounts for 1.34 % of the MoO_3 raw material. This can be reduced by 58.21 % after ultrasound-enhanced water washing, which is a 25.37 % increase compared to conventional water washing. Specifically, about 54.62 % of potassium feldspar was removed after ultrasound-enhanced water washing, an improvement of 22.69 % over conventional water washing. Approximately 86.67 % of biotite was eliminated, nearly a 46.67 % increase compared to conventional water washing method.

4. Conclusion

This study proposed an ultrasound-enhanced water washing technique for potassium removal from molybdenum trioxide under neutral conditions, and the results were as follows.

1. The forms and distribution of potassium impurities were revealed. Potassium in MoO_3 raw material existed in two forms: as KCl, where potassium K^+ was present in ionic form, and as KAlSi_3O_8 (potassium feldspar, biotite), where [K] was present in a structural form.
2. Highly efficient removal of potassium impurities from MoO_3 was achieved. The ultrasound-enhanced water washing process achieved a potassium removal level of 90.19 %, 7.69 % higher than conventional water washing, while maintaining a low molybdenum loss.
3. High-quality MoO_3 was obtained. Compared with that after conventional water washing, the content of MoO_3 after ultrasound-enhanced water washing increased by 1.23 %, reaching 94.85 %.

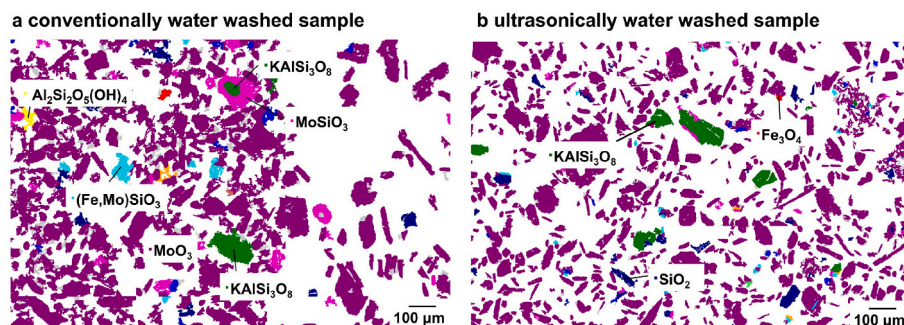


Fig. 8. MLA analysis results for different samples and labeling of major mineral phases: (a) conventionally water washed samples; (b) ultrasonically water washed samples.

Table 5

Names and mass fractions of major minerals in different samples analyzed by MLA-AMICS (wt.%).

Name	Chemical formula	Conventional	Ultrasonic
Molybdenum trioxide	MoO_3	93.62	94.85
Quartz	SiO_2	2.05	2.24
Potassium feldspar	KAlSi_3O_8	0.81	0.54
Biotite	$\text{K}(\text{Mg,Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$	0.09	0.02
Other minerals	–	3.43	2.35

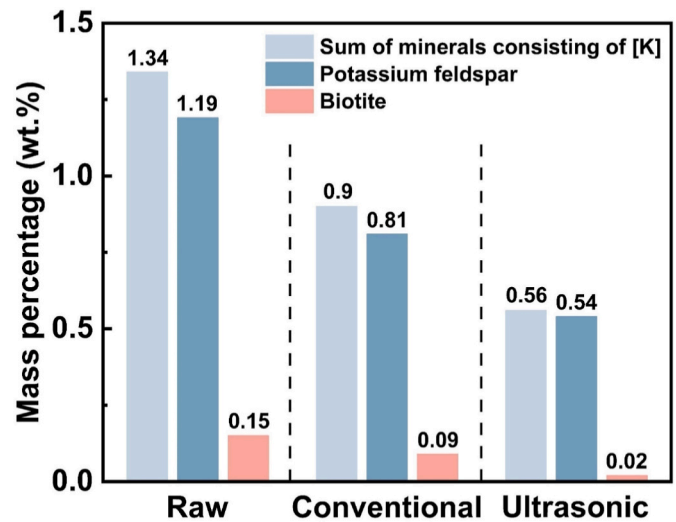


Fig. 9. Single and total mass fraction content of potassium-bearing minerals in different samples.

The main form of MoO_3 was lamellar. The contact angle reduced by 16.78 % compared to conventional water washing, to 18.99°, and the particle size was reduced by 20.67 %, to 11.9 μm . These properties are favorable for subsequent treatment processes of MoO_3 .

4. The mechanism of potassium removal by ultrasound for different forms of potassium were revealed. Cavitation and mechanical effects of ultrasound enhanced dispersion. For K^+ existing in the interstices of MoO_3 particles, ultrasound dispersed aggregated MoO_3 large particles into small particles, exposing trapped K^+ . For [K], ultrasound separated the minerals containing [K] from MoO_3 . Ultrasound-enhanced water washing removed 54.62 % of potassium feldspar and 86.67 % of biotite, which was 22.69 % and 46.67 % higher than conventional water washing, respectively.

This process addresses the shortcomings of traditional processes in

terms of environmental protection and efficiency, achieving a clean and efficient potassium removal process suitable for industrial production. However, the limitations of this study lie in its focus on a specific type of MoO₃ raw material; due to variations in impurity distribution, its effectiveness for other types of MoO₃ has not yet been verified. In subsequent studies, the ultrasound-enhanced washing process will be optimized for different MoO₃ raw materials, and parameters specific to impurities will be established to enhance its broad applicability.

CRedit authorship contribution statement

Tian Wang: Writing – original draft, Formal analysis. **Yuxi Xie:** Writing – review & editing. **Wenlong Miao:** Writing – review & editing. **Kaixue Sun:** Writing – review & editing. **You Ji:** Writing – review & editing. **Tran Van Duoc:** Writing – review & editing. **Thiquynhxuan Le:** Writing – review & editing, Project administration, Conceptualization. **Libo Zhang:** Supervision, Resources.

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.jclepro.2025.146465>.

Data availability

Data will be made available on request.

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