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Low-Temperature Chlorination-Roasting–Acid-Leaching Uranium Process of Uranium Tailings: Comparison Between Microwave Roasting and Conventional Roasting

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Abstract: A new and efficient low-temperature chlorination-roasting–acid-leaching uranium process was proposed to solve the problems of low leaching efficiency, since the leaching residue does not meet the discharge standard in the traditional nitric acid leaching-uranium tailings process, compared with conventional chlorination roasting. XRD, SEM, particle size analysis, and other analytical methods were used to characterize and analyze the phase transformation and structural changes in the roasting process of uranium-containing tailings. An investigation was conducted to elucidate the influence of roasting temperature, NaCl addition, and roasting time on the leaching efficiency of uranium. Compared with conventional chlorination roasting, microwave chlorination roasting can effectively destroy the gangue mineral structure of dissolved slag; the surface cracks of uranium tailings increase, and the particle size is smaller, so that the uranium element is fully exposed, thereby improving the leaching effect. Because microwave heating has the characteristics of selective heating and rapid heating, when the microwave-roasting power is 2000 W, the sample only needs 12 min to be heated to optimal roasting temperature, which is 8 min shorter than the conventional heating time, and the leaching rate of uranium is further improved. In the microwave roasting experiment, the roasting temperature is set to 250 °C, roasting time is 90 min, and addition amount of NaCl is 25 wt % of the tailings mass. Under the optimal roasting conditions, the leaching rate of uranium is 94.84%.

Keywords: microwave; uranium-containing tailings; sodium chloride; roasting; leaching

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1. Introduction

Uranium is an important material basis for the development of the nuclear industry. It is very important for the stable development of the nuclear energy industry and nuclear industry in general [1–3]. It plays an important role in ensuring national energy security, promoting scientific and technological progress and achieving sustainable development. The purification and conversion of uranium is a key step in the preparation of nuclear fuel elements, which converts natural uranium ore into high-purity uranium compounds. However, a large amount of refractory uranium-containing purified dissolved slag will be produced during the purification and conversion of uranium [4–6]. As part of uranium-containing radioactive tailings, the main components of the uranium-purification dissolution residue not only include silicate, silica, and other heavy metal compounds

from the mine, but they also include emulsified sludge in the internal circulation liquid from uranium-purification production. The main components of the emulsified sludge are the emulsification products of organic phase extractants. The sources are complex and vary greatly, and it is difficult for them to be recovered by conventional means [6–8]. Before the slag discharge requirements are met, only temporary measures can be taken to deal with such uranium-containing tailings, and the uranium resources cannot be effectively recovered. At the same time, long-term open-air stacking will cause potential harm to the ecological security of the surrounding environment and ultimately endanger human health. In addition, China's uranium resources are in short supply, and its external dependence is increasing year by year. Therefore, the recovery of uranium from uranium-containing tailings can solve the problem of the uranium resource shortage and environmental pollution.

At present, the main methods for recovering uranium from such complex and difficult-to-treat uranium-containing waste residues are oxidative leaching, acid-alkali combined leaching, and roasting-acid leaching. The oxidation leaching method [9,10] uses oxidants such as MnO_2 and H_2O_2 to oxidize U^{4+} to U^{6+}. U^{6+} has better solubility in common leaching agents than U^{4+} and is easier to leach. Its characteristics improve the leaching rate. However, the addition of a large number of strong oxidants will increase the cost and secondary environmental pollution. At the same time, more ions will enter, which is not conducive to the subsequent treatment and purification of the leaching solution. The acid-alkali combined leaching method [11–13] is the first to use an alkaline solution to selectively destroy the gangue structure of the ore. Cracks and pores are formed, and the method of extracting uranium from ore by an acid-leaching agent is used again. The roasting-leaching method [14–16] first destroys the mineral structure of gangue in uranium tailings by roasting pretreatment, making it loose and increasing pores, increase the specific surface area of the mineral, promote the full reaction of the mineral and the leaching agent, and improve the leaching efficiency. During the pyrolysis of uranium waste, a resistance furnace is commonly employed for thermal transfer. However, this method suffers from extended heating durations, elevated production expenses, substantial energy usage, and environmental contamination [17–19]. Consequently, there is an urgent need to identify an alternative heating technique that is efficient, eco-friendly, and minimizes resource wastage.

Microwave is an electromagnetic wave with a frequency range between 300 MHz and 300 GHz, which makes polar molecules in the medium microwave field produce high-speed motion by the action of a high-frequency electric field, and the friction and collision between molecules generate heat [20,21]. It is different from the slow transfer of heat from outside to inside, microwave heating has the characteristics of volume heating, selective heating, fast heating, and easy control. Volume heating is reflected in the fact that the dielectric sample can be heated inside and outside simultaneously, thus greatly shortening the heat conduction time in conventional heating. In addition, due to the characteristics of volume heating, the temperature of the heated material is similar to the ambient temperature and the gas products produced by the chemical reaction of the molecules inside the material can escape through the surface. It will not seal the inside of the material due to surface melting or volume shrinkage [22–24]. The selective heating is reflected in the fact that the ability of different non-metallic substances to absorb microwaves is strong and weak. Therefore, for the mixture, the components with strong microwave-absorption ability heat up quickly, and the substances with low absorption capacity heat up slowly. The microwave absorption capacity of a substance is predominantly governed by its dielectric loss factor, a parameter indicative of the material's capacity to dissipate energy stored within it and transform it into thermal energy. In contrast to conventional thermal processing techniques, microwave heating facilitates selective material heating, thereby

conferring several advantages, including localized thermal generation, accelerated thermal transfer rates, enhanced thermal efficiency, the stimulation of chemical reactions, and a lowering of the temperatures necessary for such reactions to occur. This targeted heating approach stands in stark contrast to traditional methods, offering a more precise and efficient alternative for material processing [25–28]. Lu et al. [29] carried out research on microwave-assisted rock-breaking technology. The microwave electromagnetic differences of 11 kinds of diagenetic minerals were tested in a multimode cavity. It was found that the wave absorption of different mineral phases was different, and stress cracking occurred at lower temperatures. Amankwah et al. [30] proposed a new process of microwave roasting pretreatment of gold ore containing silicate and other impurities. Making full use of the characteristics of microwave selective heating, thermal stress is generated inside the gold mine, thus destroying the mineral structure. After microwave roasting treatment, the mineral strength and cohesiveness have been effectively reduced. Olubambi et al. [31] studied the mechanism of microwave pretreatment to improve the bioleaching behavior of low-grade complex sulfide ores. The number of cracks in the samples treated by microwave increased, the sulfur content decreased, and the pyrite phase increased. The change of mineral phase promoted the increase in solubility of microwave samples. The results show that microwave treatment improves the bioleaching behavior of the ore and greatly increases the leaching rate of copper and iron. Zhang et al. [32] recovered aluminum from fly ash by microwave treatment. The results show that microwave heating technology is an effective and energy-saving method for the treatment of fly ash to recover aluminum. After microwave roasting at 800 °C for 60 s, 95% of the aluminum in fly ash was released. Liu et al. [33] pretreated jarosite by microwave roasting technology, and recovered valuable metals by water leaching. Under the action of microwave heating, the elements in the mineral show a discrete distribution, which is conducive to the contact between the leaching solution and the mineral, and improves the leaching rate of valuable leaching. Zhang et al. [34] used microwave roasting molybdenum concentrate combined with nitric acid leaching to prepare ammonium molybdate. Compared with conventional roasting, microwave roasting can greatly shorten the roasting time under the best conditions, and the obtained ammonium molybdate has higher purity and better stability. Li et al. [35,36] used microwave roasting technology to treat cyanide tailings. It was found that CaCl had a good ability to absorb microwaves. When the microwave power is 1300 W, the roasting temperature is set to 1137 °C, and the roasting time lasts for 15 min, the gold extraction rate reaches the best of 85.2%. Compared with conventional roasting, the extraction rate of gold has been significantly improved, and the energy consumption has been greatly reduced. Zeng et al. [37] compared the conventional-roasting and microwave-roasting water-leaching process of red mud. The results show that under the same conditions, microwave roasting has a better alkali removal effect and lower power than conventional roasting. Microwave roasting can help to promote the crystal transformation of alkali components in red mud during the whole roasting process. Jena et al. [38] successfully recovered potassium from unconventional silicate rocks by microwave-assisted roasting and subsequent water immersion. In the roasting process, charcoal is used as a microwave absorbing material to absorb microwave heating as a reaction heat source between $CaCl_2$ and nepheline synthetic rock. Aiming at the problems of low uranium-leaching rate and high energy consumption in the conventional-roasting nitric acid-leaching method, a microwave chlorination-roasting nitric acid-leaching method was proposed to improve the leaching rate and roasting efficiency of uranium and reduce energy consumption.

In this paper, NaCl is used as a chlorinating agent; NaCl as a medium-low-temperature chlorinating agent has the advantages of low price and low reaction temperature. The effects of microwave power, roasting temperature, NaCl addition, and roasting time on uranium

leaching efficiency were studied. X-ray diffraction, differential thermal-thermogravimetric analysis, particle size analysis, scanning electron microscopy, and energy spectrum analysis were used to characterize the phase transition of tailings during microwave roasting, and the mechanism of sodium chloride destroying mineral structure during microwave roasting was explored. This was undertaken to achieve the purpose of efficient extraction of uranium from uranium tailings and also to provide technical support for the treatment of such radioactive solid waste.

2. Experimental Process

2.1. Materials

In this experiment, sodium chloride (NaCl) and nitric acid (HNO_3) were used as analytical pure reagents, and the test water was deionized water to improve the accuracy and repeatability of the experiment. The raw materials used in the experiment were the tailings produced by the extraction residue of a uranium-purification enterprise in southern China during production and processing and the filter plate and frame after the mixing and standing of the wastewater in the system. The composition was complex. In addition to the solid-waste residue and organic impurities produced during the uranium-leaching process, diatomite was also used as a filter aid. The content of U was 2.31%, and its main components and contents are shown in Table 1. The results of X-ray diffraction (XRD) analysis of uranium-bearing tailings are shown in Figure 1.

Table 1. Chemical composition of the uranium tailings.

Chemical Component	SiO_2	P_2O_5	Al_2O_3	Fe_2O_3	ZrO_2	CaO	U
Content (%)	63.36	16.04	3.72	3.83	5.03	1.16	2.31

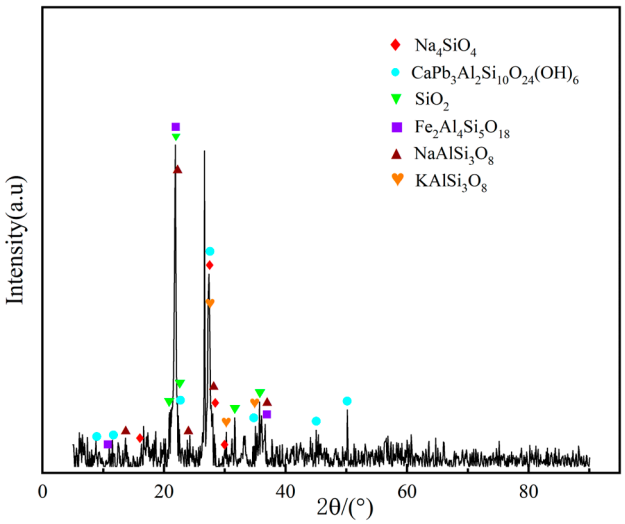

Figure 1. XRD pattern of uranium tailings.

2.2. Preparation and Experimental Process of Roasted Samples

The preparation process of the roasted material is shown in Figure 2a. The microwave equipment is mainly composed of a temperature control system, water cooling system, microwave generation system, microwave cavity, and other parts. The microwave generation system includes two SAMSUNG OM75P magnetrons, and the total microwave power can reach up to 3.0 kW. The experimental materials were dried at 100 °C for 6 h in a ventilated drying oven and then crushed and passed through a 100-mesh sieve (particle size less than 150 μm). A total of 100 g of uranium-bearing tailings were weighed each time, and sodium

chloride was added according to the predetermined mass percentage of tailings. Then, it was put into the mixing tank for full mixing, and then the mixed sample was put into the microwave oven. Set the operating power, start the equipment, raise the temperature to the target temperature and keep the temperature for a specified time, and take out the sample after cooling. Figure 2b shows the schematic diagram of nitric acid-leaching experiment. First, a nitric acid solution with a concentration of 5 mol/L was prepared for subsequent leaching experiments. Subsequently, 20 g of the sample was weighed and placed in a flask. Nitric acid was weighed according to the liquid–solid mass ratio of 5:1 and poured into the flask. The flask was placed on a constant-temperature magnetic stirrer, and the water bath temperature was set at 80 °C, the rotor speed was 200 r/min, and the stirring time was 2 h. Then, the slurry mixture after leaching was subjected to suction filtration treatment, and the filter cake obtained after suction filtration was rinsed with deionized water three times. The ratio of deionized water to nitric acid solution was 1:1. Finally, the wetting solution and the leaching solution were mixed evenly, the volume was weighed, and the uranium concentration was sampled and analyzed. The remaining filter residue was dried, weighed, ground, and sampled to analyze the uranium content.

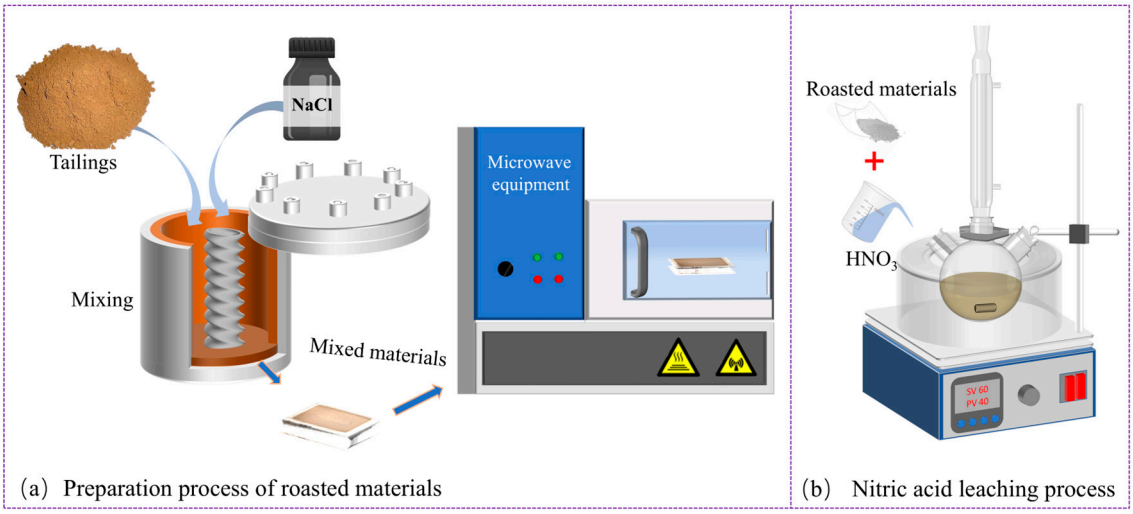

Figure 2. Preparation process of roasted materials and nitric acid-leaching experiment.

3. Results and Discussion

3.1. Effect of Microwave Power on Heating Behavior

The effects of microwave power on the heating behavior of materials were studied by setting the microwave powers of 1000 W, 1500 W, and 2000 W, respectively, and comparing them with the heating behavior of a conventional tube furnace under the condition of 1500 W. The results are shown in Figure 3. Under the condition of microwave powers of 1000 W, 1500 W, and 2000 W, the average heating rates of the material to the target temperature of 300 °C were 9.4 K/min, 15.8 K/min, and 21.5 K/min, respectively. However, under conventional-roasting conditions, when the power is 1500 W, the average heating rate of the material is 11.1 K/min. At the same power, the microwave heating rate is faster. It is mainly because the dielectric properties of minerals are the most prominent in the cavity of microwave equipment, so that the microwave energy interacting with the material is converted into heat energy through the dielectric effect, and the material is rapidly heated to the set value in the microwave cavity [35]. The heat-transfer mode of conventional roasting is heating conduction and heat convection. In this heat-transfer mode, there are inevitable heat dissipation and heat loss between the furnace body and the environment, the material and the environment, and the furnace body and the material,

which will lead to temperature hysteresis in the conventional tube furnace, resulting in uneven heating of the material during the heating process. According to Equation (1), the energy consumptions of microwave and conventional heating at different powers are 1920 kJ, 1710 kJ, 1680 kJ, and 2520 kJ, respectively.

$$Q = Pt \quad (1)$$

where the Q, P, and t denote energy expenditure, the magnitude of power, and the duration of the heating period, respectively. It has been observed that augmenting the microwave power input can significantly reduce the time required for heating. Adhering to the principle of minimizing energy usage while maximizing the efficacy of the process, a microwave power of 2000 W was selected for microwave roasting experiments in the subsequent study.

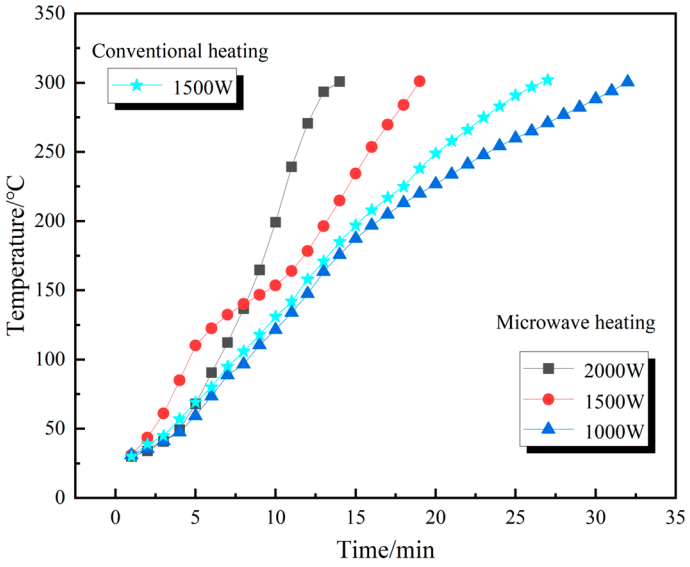

Figure 3. Heating curves of samples at different powers.

3.2. Effect of Chlorination-Roasting Temperature

Under the condition that the addition amount of Na Cl is 25 wt % of the tailings mass and the chlorination roasting time is 120 min. The effect of roasting temperature on leaching was investigated by microwave heating. The experimental results are shown in Figure 4. By comparison, it is not difficult to find that the leaching rate of microwave-roasting samples at different temperatures is higher than that of conventional roasting. The increasing on microwave-roasting temperature, the leaching rate of the sample increases first and then decreases. When the temperature reaches 250 °C, the leaching rate of uranium reaches a maximum of 94.75%. Under the microwave-roasting temperature of 200 °C, the leaching rate of uranium can reach 93.34%. In order to achieve the same effect, the temperature condition required for conventional roasting is 250 °C. This is mainly due to the fact that microwave heating has the characteristics of direct heating and rapid heating, so that the energy required for the reaction is quickly supplemented [26]. The phase changes of the calcined products at different roasting temperatures were analyzed by X-ray diffraction (XRD), and the results are shown in Figure 5. The main phases are SiO_2, $NaAlSi_3O_8$, $AlCl_3$, and FeOCl. The diffraction peak of SiO_2 phase decreases with the increase of temperature, which proves that some quartz structures are destroyed during microwave roasting, so that the wrapped uranium element will be further exposed, and the leaching rate will increase. However, when the temperature exceeds 250 °C, the diffraction peak of the $NaAlSi_3O_8$ phase begins to gradually strengthen, so the newly generated $NaAlSi_3O_8$ phase will form a

new package for uranium [14,39], and the corresponding leaching rate will also decrease. The diffraction peaks of AlCl3 and FeOCl phases gradually increase with the increase of temperature. The roasting of sodium chloride has high selectivity, which can effectively separate other metal components from oxides. With the progress of chlorination roasting, the complex metal components are chlorinated, which can further cause the destruction of the sample structure. The diffraction peak of the FeOCl phase is the most obvious at 250 °C. As the temperature continues to increase, the peak value gradually decreases, and the corresponding leaching rate also decreases. FeOCl may play a catalytic role in the leaching process. The particle size of tailings after different microwave roasting was compared, and the results are shown in Figure 6. As the increase in microwave roasting temperature, the average particle size of tailings after roasting showed a trend of decreasing first and then increasing. At 250 °C, the average particle size was 14. 33 μm. At this time, the specific surface area of the leaching sample is the largest, the contact with the leaching solution is the most sufficient, and the leaching efficiency is the highest. Compared with the average particle size of 15.74 μm of conventional calcined products, microwave roasting can further reduce the particle size of tailings after roasting and improve the leaching efficiency.

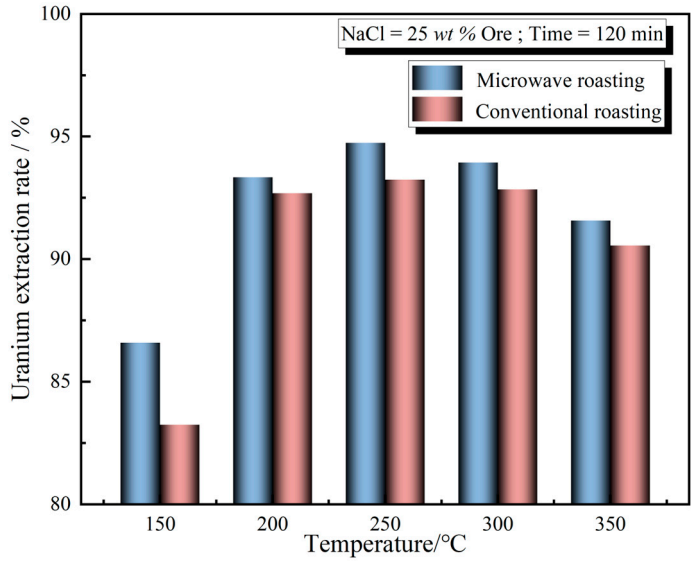

Figure 4. Effect of conventional roasting and microwave roasting on leaching at different temperatures.

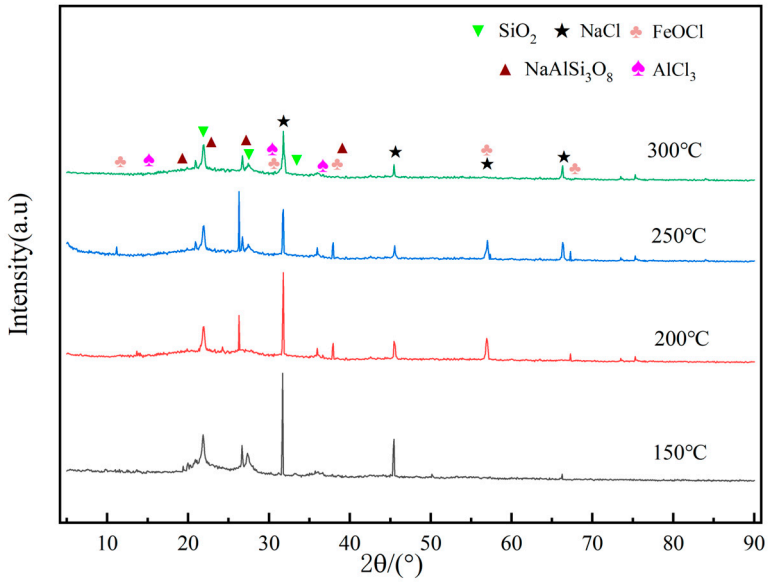

Figure 5. XRD analysis of microwave-roasting samples at different temperatures.

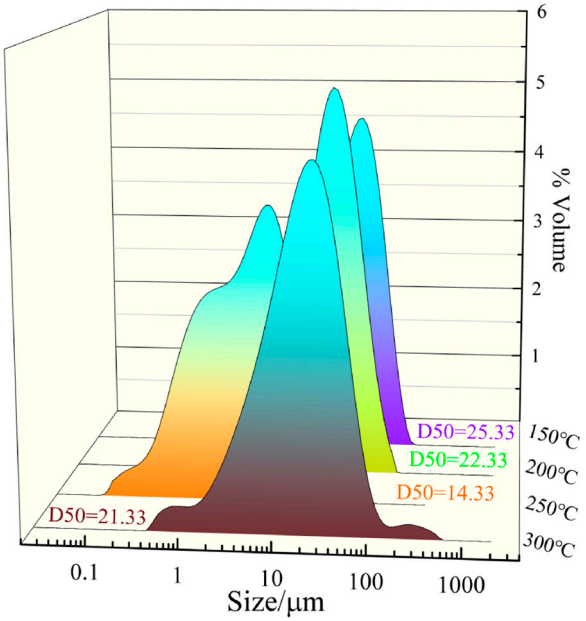

Figure 6. Particle size analysis of samples at different microwave-roasting temperatures.

3.3. Effect of NaCl Addition Amount

Under the conditions of microwave chlorination-roasting temperature 250 °C and roasting time 120 min, the effect of sodium chloride addition (10–30 wt %) on leaching are shown in Figure 7. The leaching rate of uranium increased gradually with the increase of sodium chloride dosage. When the amount of NaCl added was 25 wt % of the tailings mass, the leaching rate was up to 94.73%. Then, NaCl was added, and the leaching rate began to flatten, from 94.75% to 94.78%, an increase of only 0.03%. Under this condition, with the increase in sodium chloride addition, the leaching rate of uranium increased first and then decreased. When the addition of sodium chloride was 20 wt %, the leaching rate was up to 93.38%. By comparison, it is not difficult to find that the leaching rate of microwave-roasting pretreatment samples is higher than that of conventional-roasting pretreatment under the same additional amount of sodium chloride, and when the addition amount of NaCl exceeds 20 wt %, the leaching rate of microwave-roasting pretreatment samples does not show a downward trend. The SEM of microwave- and conventional-roasting pretreatment under different NaCl additions was compared and analyzed, as shown in Figure 8. In the conventional roasting experiment, when the addition amount of sodium chloride is 20 wt %, the particle size of tailings after roasting is the smallest, the degree of separation is the best, and cracks appear. However, when the addition amount of NaCl exceeds 20 wt %, the particle size of tailings after roasting increases, and the separation degree is poor. Under the condition of microwave roasting, when the addition amount of sodium chloride is 25 wt %, the particle size of tailings after roasting is the smallest, and the separation degree is the best. When the addition amount of NaCl is 30 wt %, the particle size of the sample hardly changes. Whether using conventional-roasting pretreatment or microwave-roasting pretreatment in the range of NaCl addition of 10–20 wt %, the role of NaCl in the roasting process is mainly to destroy the structure of the sample, increasing the specific surface area and porosity of the sample, thereby promoting the dissociation of uranium in the tailings. However, due to the different heating methods of the two roasting pretreatments, there are some differences in the degree of damage to the mineral structure during the heating process. The conventional-roasting pretreatment gradually heats up the mineral from the outside to the inside by means of heat conduction. At the beginning of the reaction, the transient thermal stress will cause cracks on the surface of the minerals, and the partial structure will be destroyed, which is conducive to the

reaction of NaCl with minerals. When the temperature inside and outside the mineral is the same, the thermal stress disappears, and the reaction between the mineral and NaCl is the only way to destroy the mineral structure. However, when the amount of NaCl added exceeds 20 wt %, excessive NaCl reacts with minerals to form $NaAlSi_3O_8$, $Na_2Pb_2O_7$, $Zr_7O_9F_{10}$, and other compounds [16], forming a secondary encapsulation of uranium, resulting in a decrease in leaching rate. The XRD analysis of the calcined products with different NaCl additions under the conventional-roasting pretreatment conditions is shown in Figure 9. The microwave heating specifically selects the heating property. The components with strong microwave absorption capacity in the mixture material rise faster, and the substances with lower absorption capacity rise slowly. The thermal stress inside the mineral will continue to destroy the mineral structure, and the cracks on the mineral surface will increase and extend to the interior of the mineral. The size of the mineral particles decreases; the specific surface area of the mineral increases, which is conducive to the contact between the mineral and NaCl; and the internal closure of the material will not be caused by surface melting or volume shrinkage during the reaction.

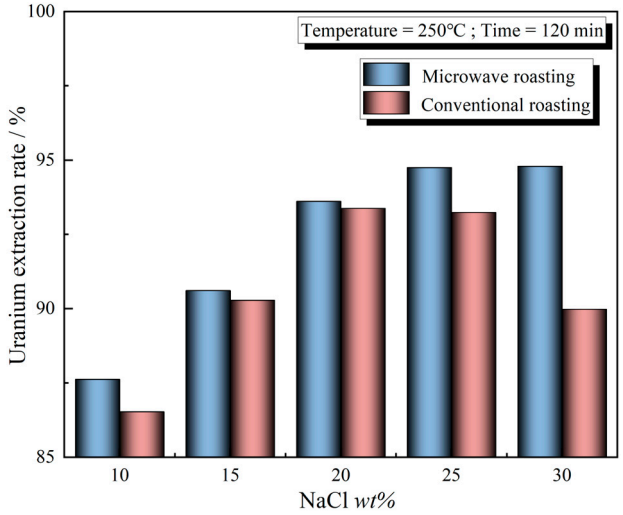

Figure 7. Effect of conventional roasting and microwave roasting on leaching at different NaCl addition amounts.

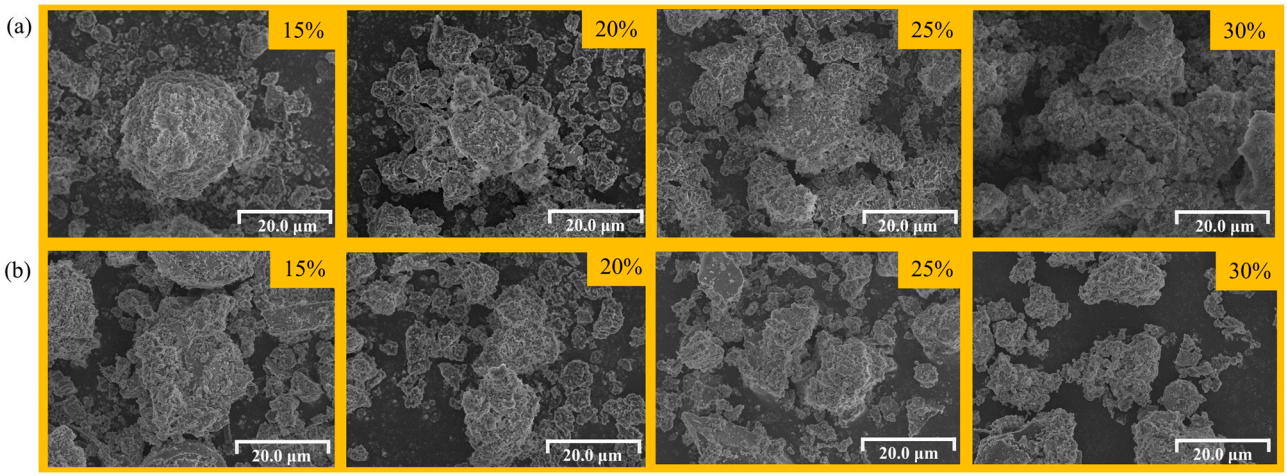

Figure 8. SEM graph of samples at different NaCl addition amounts: (**a**) conventional roasting, (**b**) microwave roasting.

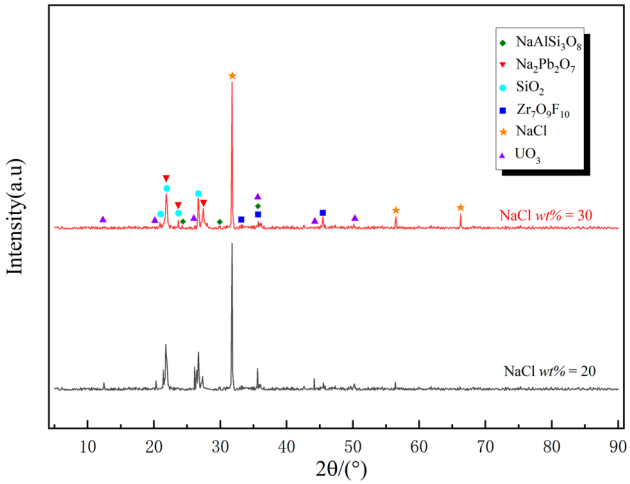

Figure 9. Conventional roasting: XRD graph of sample at different amounts of NaCl addition.

3.4. Effect of Chlorination-Roasting Time

Under the reaction conditions of roasting temperature of 250 °C and NaCl addition of 25 wt % of tailings mass. The effect of microwave chlorination-roasting time on uranium leaching rate is shown in Figure 10. As the extension of roasting time, the leaching efficiency is on the rise. When the roasting time is 90 min, the leaching rate is up to 94.84%. However, when the roasting time continues to extend, the leaching rate of uranium tends to be stable. Before 90 min, due to the short roasting time and insufficient chlorination reaction, most of the inclusion structure of uranium is not destroyed. After 90 min, the structure of uranium inclusions is destroyed more thoroughly under the thermal-stress damage caused by microwave heating, and the chlorination reaction tends to be stable. In the roasting reaction process, the roasting time is an important factor that directly affects the reaction process. More thorough chemical and physical reactions will be conducive to the destruction of the sample structure, and the exposed area of the uranium component will be further increased, thereby promoting the extraction of uranium. On the contrary, when the roasting time is not sufficient, it will lead to incomplete physical and chemical reactions, and it is difficult to achieve the best leaching effect. The SEM of the samples under different microwave-roasting times was compared, and the results are shown in Figure 11. The mixed material after microwave pretreatment has obvious pores and cracks. With the extension of roasting time, the surface cracks of the roasting products increase and expand, the mineral size decreases, and the reaction area increases, which can effectively release the wrapped uranium element and is beneficial to the leaching of uranium.

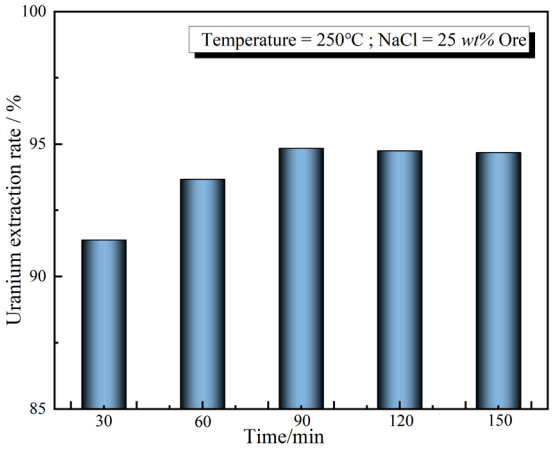

Figure 10. Effect of roasting time on uranium-leaching rate.

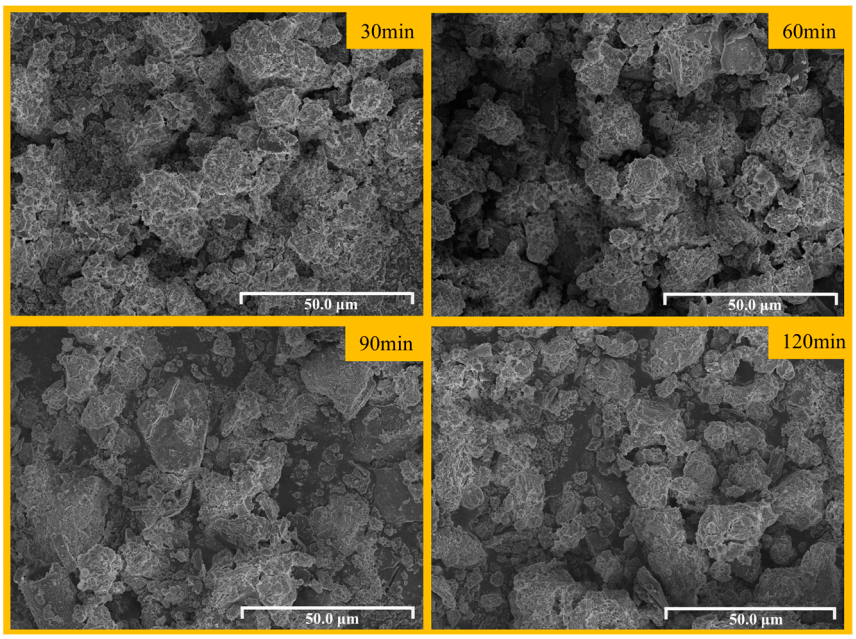

Figure 11. SEM graphs of samples at different roasting times.

3.5. Analysis of Microwave Chlorination-Roasting Process

B The mechanism of microwave roasting pretreatment process under optimal roasting conditions is shown in Figure 12. In the initial stage of the microwave-roasting experiment, the mixture is in a low-temperature state, its dielectric loss is low, and the penetration depth of the microwaves is high. Because a microwave has the characteristics of in situ heating and selective heating, the phases with strong dielectric properties such as Fe_2O_3 and Al_2O_3 in the material are preferentially heated, which makes the internal temperature rise rapidly, and the internal temperature is higher than the surface temperature [21,27]. However, the wave-absorption performance of gangue and other phases is weak, thus forming a temperature gradient, resulting in the generation of thermal stress, and thus achieving the purpose of destroying the structure of the uranium cladding. The chlorination reaction is carried out at the same time, which further damages the mineral structure and expands the exposed area of the uranium.

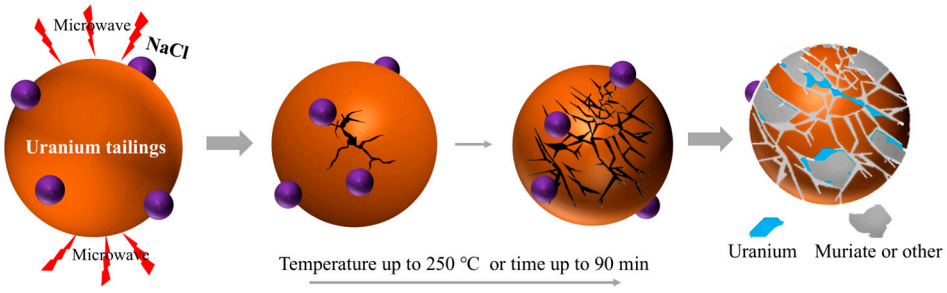

Figure 12. Schematic diagram of microwave chlorination-roasting process.

4. Conclusions

In this paper, uranium tailings are taken as the research object, and microwave chlorination roasting is adopted to make full use of the advantages of microwave selective heating and rapid heating. The structure of tailings is effectively destroyed under the pretreatment of microwave roasting. In contrast to the conventional chlorination-roasting, microwave chlorination-roasting has the advantages of short roasting time, low energy consumption, and higher degree of damage. The effects of roasting temperature, sodium

chloride addition, and roasting time on uranium leaching were explored. The following conclusions are obtained:

(1) The optimum process of microwave chlorination-roasting pretreatment involves roasting temperature at 250 °C, NaCl addition amount 25 wt % of tailings mass, and roasting time 90 min. Under the optimum roasting conditions, the leaching rate of uranium is 94.84%, which is 1.46% higher than that of 93.38% obtained by conventional-roasting pretreatment;

(2) In contrast to traditional roasting methodologies, microwave heating at a power level of 2000 W is capable of achieving a temperature of 250 °C within a 12-min timeframe, whereas conventional heating methods require a duration of 20 min to reach the same temperature. The subsequent roasting time was shortened from 120 min to 90 min, which significantly decreased energy consumption.

(3) The results of SEM and particle size analysis show that microwave chlorination-roasting pretreatment can effectively destroy the gangue mineral structure of dissolved slag, make the waste slag looser, make the size of tailings particles smaller, increase the specific surface area, and increase the microcracks, so that the uranium element is fully exposed and the leaching effect is improved.

Microwave-roasting pretreatment can improve the roasting efficiency of uranium tailings while reducing energy consumption, which provides a feasible method for industrial treatment of uraniu tec m tailings. Therefore, it is particularly important and urgent to promote the application of microwave-roasting hnology in environmental protection and resource utilization, especially regarding the recovery of important strategic resources.

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