Preparation of Potassium Dichromate Crystals from the Chromite Concentrate by Microwave Assisted Leaching

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Abstract: In the present investigation, the oxidizing roasting process of chromite with sodium carbonate to prepare potassium dichromate crystals was studied in the microwave field with air, by heating the chromite and sodium carbonate. The chromite and sodium carbonate heated separately at 1000 °C in the microwave oven (frequency: 2.45 GHz; power 1.5 kW) in order to study the microwave absorption properties. The dielectric constant and dielectric loss factor of the chromite and sodium carbonate examined. Then, chromite with sodium carbonate taken in (1:2) ratio and heated at 750 °C. Thus obtained samples were characterized using various techniques includes Powder-XRD (XRD), Scanning Electron Microscopy (SEM), and X-ray fluorescence (XRF). The XRD pattern reveals the existence of Fe_3O_4, Fe_2O_3, $NaAlO_2$, and Na_2CrO_4. The iron and aluminum were leached out as Fe_2O_3 and $Al(OH)_3$ respectively. The resulting sample treated with the KCl to prepare potassium dichromate crystals. Finally, potassium dichromate crystals formed.

1. Introduction

The application of microwave heating in metallurgy has been developed in recent years. Some of the world's developed countries such as United States, United Kingdom, Japan, and Canada have given the great importance to this new technology. China has started the research work in this field from 1980s. Tiny although the application of microwave heating in metallurgy is still in the development stage [1,2], many attractive research results have been obtained, and microwave heating has been used in the pretreatment of the ore crushing, refractory gold ore, and the recovery of gold from tailings, extraction of rare metals from ores and heavy metals [3–6].

The microwave is an electromagnetic wave, with the frequency of 0.3–300 GHz (wavelength in 1–1000 mm), and located in the electromagnetic spectrum of red between the external radiation and radio wave. The heating effects of microwave are characterized as follows: (1) the powder material can be heated quickly, and the heating effect can shorten reaction time; (2) can selectively heat the powdery material to make the ore mine and the gangue minerals produce thermal dissociation; (3) can be used without contact with thermal powder material to avoid the external pollution of the material; (4) with

molecular ruler degree of stirring, can create a strong solid-solid state reduction kinetics, microwave energy is a new type of heating energy, which has obvious advantages.

It is possible to realize that the rapid and selective heating of powder material [7–9]. In order to avoid the traditional heating method, it is required to bring the powder material heat and mass transfer to the phenomenon of uneven, using microwave heating method, which can effectively heat material [10].

Potassium dichromate crystals are orange-red monoclinic crystal plate-shaped crystals [11]. The main raw material mainly used for the production of chromate products such as Cr_2O_3 etc. [12]. In the chemical industry, it is used as an oxidizing agent to make a match head. Manufacture of enamel powder used for enamel, enamel to green. The glass industry as a colorant. Printing and dyeing industry as a mordant. In the spice industry it is used as an oxidizing agent. In addition, it is one of the important reagents to test the Chemical Oxygen Demand (COD) of the water body. The acid water solution can detect the ethanol, and the ethanol can quickly generate the blue and green trivalent Cr ion [13,14], and the solution becomes green. Acidification of potassium dichromate can be ethanol oxidation and discoloration, to test whether the driver drunk driving [15].

In nature, it is easy to form a symbiosis with iron Chromium Picotite ($Cr_2O_3 \cdot FeO$), this type of symbiotic ore is commensurate with the chromium iron ore (chromite) [16,17]. Chromite is China's shortage of mineral resources, reserves accounted for only 15% of the world, and the size of deposit is small, scattered and ore grade is low [18–20], the dependence on the import of chrome ore is high up to about 95% in our country.

Chromite easily forms a symbiosis with picotite $[(Mg, Fe)Cr_2O_4]$ in nature, this type of symbiotic ore is commensurate with the chromite [21,22]. The Chromite is the main raw material for the production of chromium compounds, which is the main component of $FeO \cdot Cr_2O_3$, in general, chromite content is about 40%. It is commonly used in the chromite preparation of potassium dichromate in Laboratory [23]. But the conventional heating and extraction are slow in this process. Importantly, the conversion rate of Cr is not very high. Ultrasonic extraction can accelerate the conversion rate and shorten the extraction time and strengthen the effect of extraction and also can improve the conversion rate of some elements [24–28].

In this paper, mainly research using chromite to prepare potassium dichromate crystals in the field of the microwave is reasonable and by microwave heating and ultrasonic extraction can get a high purity of potassium dichromate crystals.

2. Experimental Work

2.1. Materials and Characterization

The chromite concentrate sample originated from South Africa and provided by Jinzhou Company in Liaoning Province, China. The brief chemical components of chromite analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES) and the results listed in Table 1. Phase analysis of the chromite concentrate was performed with the XRD (Bruker, Billerica, MA, USA; $CuK\alpha$, wavelength: 1.5405980) and the patterns were shown in Figure 1. The SEM images of the chromite concentrate were recorded using JSM-35CF SEM equipment (JAPAN Electron Optics Laboratory Co, Ltd., Tokyo, Japan) and shown in Figure 2. The particle size distribution was measured using particle size analyzer (LS230, Beckman Coulter, Brea, CA, USA).

Table 1. Brief chemical components of chromite concentrate (wt. %).

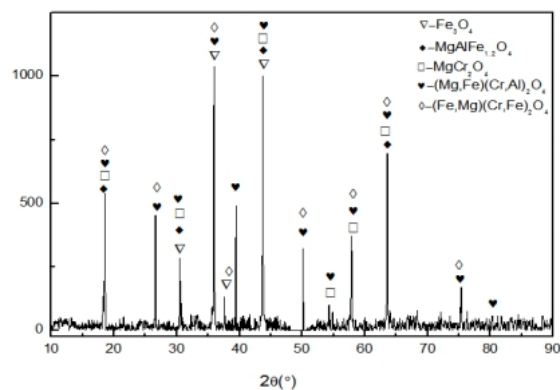| Cr_2O_3	Fe	Al_2O_3	MgO	H_2O	SiO_2	CaO	P	S
51.44	13.56	11.11	10.44	10.44	3.95	1.45	0.05	0.05

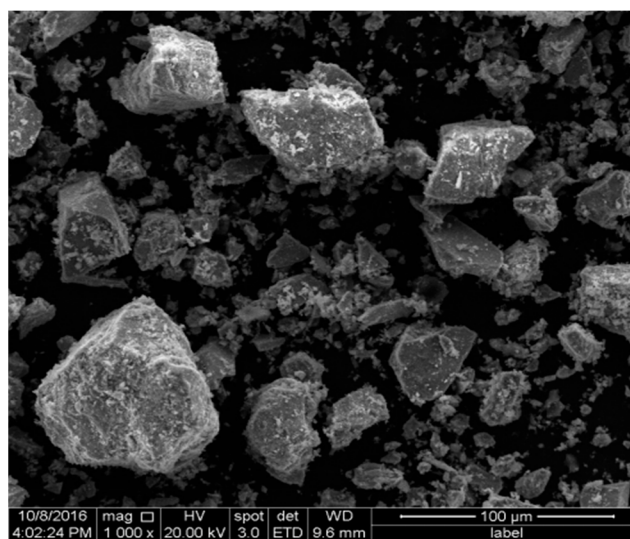**Figure 1.** XRD pattern of chromite concentrate.

Figure 2. SEM images of chromite concentrate.

*2.2. Dielectric Property Measurement System*

The microwave absorbing property of the material is an important physical indicator in the field of microwave chemistry. In this work, a hybrid experimental/computational permittivity measuring system developed by the Institute of Applied Electromagnetics at Sichuan University used to determine the complex permittivity of the chromite and sodium carbonate at different sample temperature. First, the quartz tube without measuring material calibrated to get the resonant frequency and the quality factor of empty cylindrical cavity, then the sample was placed in the calibration, which has already completed with the diameter of 4 mm quartz tube. At the beginning of the experiment, the quartz tube kept into the test chamber for calibration, the calibration is completed. Sample materials kept into quartz tube measured at the start 2.45 GHz electromagnetic wave propagation along the waveguide section. When it encountered the material of the quartz tube, the energy of the wave absorption, transmission, and reflection, change the microwave output frequency and quality factor of the material observed before and after the entry of the quartz tube, which can calculate the absorbing ability of the size of the quartz tube material. The structure of dielectric constant measurement device shown in Figure 3.
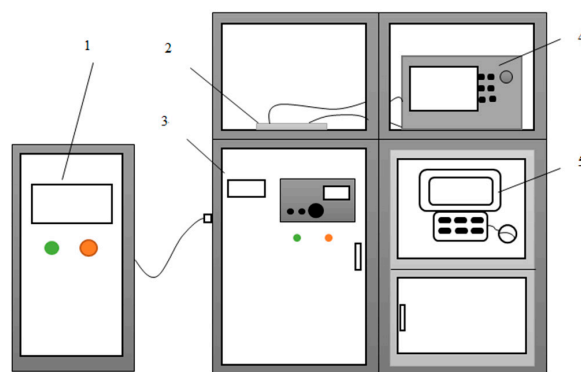
Figure 3. The structure diagram of dielectric constant measurement device (1-water cooling system, 2-reaction system, 3-control system, 4-microwave network analyzer, 5-PC collection system).

2.3. Experimental Setup and Procedure

All experiments were carried out in a microwave oven, which was developed by Kunming University and it has a good performance. Its adjustable power range was 0–6 kW, microwave frequency was 2.45 GHz; with a tungsten-rhenium thermocouple (measuring temperature range: 0–1800 °C). The particle size of chromite was 74 μm and it was measured using particle size analyzer. The chromite powder (50 g) and sodium carbonate (50 g) separately heated in microwave heating furnace (frequency 2.45 GHz, power 1.5 kW) using silicon carbide crucible. Similarly, chromite and sodium carbonate mixed (weight ratio of 1:2) and roasted using a microwave oven for 30 min at 750 °C. Thus, the obtained sample contains Fe_3O_4, Fe_2O_3, $NaAlO_2$, and Na_2CrO_4. The sample was ground, water added and ultrasonic extraction carried to remove Iron particles. Then the iron-free sample was neutralized (pH: 7 to 8) using glacial acetic acid to leach out the aluminum particles. Further, glacial acetic acid was added (pH: 5) in order to convert CrO_4^{2-} into $Cr_2O_7^{2-}$. Then the KCl solution added and heated. When crystals precipitation appeared, the heating stopped and crystals washed to remove KCl in a hot environment. Afterwards, the filtrate cooled and transferred into the ice bath. Then a large amount of $K_2Cr_2O_7$ formed [29]. It washed with ice water, at this time because $K_2Cr_2O_7$ has a relatively large solubility and retained in the filtrate. Finally, the orange-red crystals obtained (Figure 4).
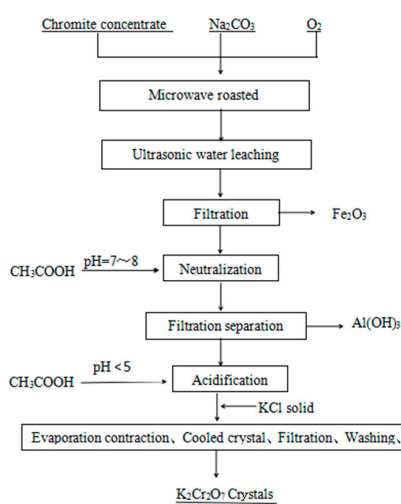
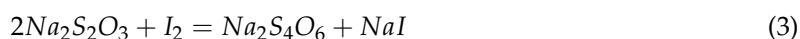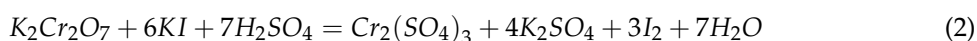**Figure 4.** Flowchart for the preparation of $K_2Cr_2O_7$ from chromite concentrate with Na_2CO_3.

The waste liquid must be recycled because Cr is poisonous. The conversion rate of chromite calculated according to the following formula [30]:

$$\%\eta(\text{Cr}) = [\text{Cr}]_r \cdot V_r / m_0 \times 100 \tag{1}$$

where $[\text{Cr}]_r$, V_r and m_0 denotes the Cr concentration (g/L) in the leach liquor, the volume (L) of the leach liquor, and the Cr mass (g) in the chromite concentrate, respectively.

2.4. Determination of Purity of the Resultant Product

2.5 g (m_{sample}) of $K_2Cr_2O_7$ weighed, dissolved in 250 mL volumetric flask ($V_{K_2Cr_2O_7}$), and transferred 25 mL solution to an iodine flask ($V_{1K_2Cr_2O_7}$). Then, put 10 mL (2 mol/L) H_2SO_4 solution and 2 g KI solid mixture for 5 min in the dark and added to 100 mL of distilled water, the solution changed to yellow-green with the prepared standard 0.1 mol/L $Na_2S_2O_3$($C_{Na_2S_2O_3}$) solution. Then, starch 3 mL (density 1.5 g/cm^3) added and continued titration solution until the blue faded presenting a bright green, according to $Na_2S_2O_3$ standard the concentration and use of the volume ($V_{Na_2S_2O_3}$) to calculate the conversion rate of the product. The main reactions involved in this process as follows:
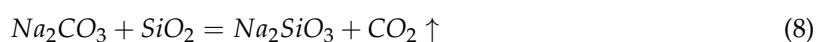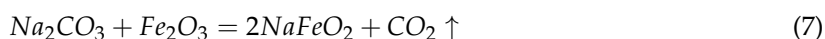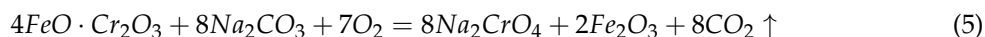
$$K_2Cr_2O_7 + 6KI + 7H_2SO_4 = Cr_2(SO_4)_3 + 4K_2SO_4 + 3I_2 + 7H_2O \tag{2}$$

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + NaI \tag{3}$$

$$K_2Cr_2O_7\% = \frac{C_{Na_2S_2O_3} V_{Na_2S_2O_3} M_{K_2Cr_2O_7}/6}{m_{sample}\left(V_{1K_2Cr_2O_7}/V_{K_2Cr_2O_7}\right)} \times 100\% \tag{4}$$

3. Results and Discussion

According to the previous reports, in the microwave field, the process of material heating mainly decided by the dielectric loss in the electromagnetic field. The dielectric properties characterized by two parameters (1) dielectric constant and (2) dielectric loss factor. The numerical value reflects the interaction between the reduced material and the electromagnetic field. When the reducing material placed in the microwave field, the dielectric polarization will occur due to the presence of a series of different particle reduction materials, namely the dielectric polarization when the particles were not the same, polarization and elimination time was not the same. The dielectric constant and dielectric loss factor of the material calculated theoretically to explain the strength of the material wave absorbing property [31–37].
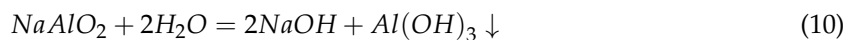
During the microwave heating process, the FeO·Cr$_2$O$_3$ was oxidized to Na$_2$CrO$_4$ in the presence of O$_2$ and Na$_2$CO$_3$. At the same time, the components of Al$_2$O$_3$, Fe$_2$O$_3$, and SiO$_2$ all transformed into the corresponding soluble salts NaAlO$_2$, NaFeO$_2$ and Na$_2$SiO$_3$, all of them dissolve easily in water. Action equations of the main reactions are as follows:

$$4FeO \cdot Cr_2O_3 + 8Na_2CO_3 + 7O_2 = 8Na_2CrO_4 + 2Fe_2O_3 + 8CO_2 \uparrow \tag{5}$$

$$Na_2CO_3 + Al_2O_3 = 2NaAlO_2 + CO_2 \uparrow \tag{6}$$

$$Na_2CO_3 + Fe_2O_3 = 2NaFeO_2 + CO_2 \uparrow \tag{7}$$

$$Na_2CO_3 + SiO_2 = Na_2SiO_3 + CO_2 \uparrow \tag{8}$$

Thus formed NaFeO$_2$ is leached in the form of Fe(OH)$_3$ the main reaction equation is as follows:

$$NaFeO_2 + 2H_2O = NaOH + Fe(OH)_3 \downarrow \tag{9}$$

Controlling the pH in the process helps in the leaching of $NaAlO_2$ and Na_2SiO_3 by precipitation of $Al(OH)_3$ and H_2SiO_3, while Na_2CrO_4 converted to $Na_2Cr_2O_7$. The reaction equations of the main reactions are as follows:

$$NaAlO_2 + 2H_2O = 2NaOH + Al(OH)_3 \downarrow \tag{10}$$

$$Na_2SiO_3 + 2H_2O = 2NaOH + H_2SiO_3 \downarrow \tag{11}$$

$$2CrO_4^{2-} + 2H^+ = Cr_2O_7^{2-} + H_2O \tag{12}$$

The $Na_2Cr_2O_7$ react with KCl and forms the $K_2Cr_2O_7$ as fellows [38]:

$$Na_2Cr_2O_7 + KCl = K_2Cr_2O_7 + 2NaCl \tag{13}$$

3.1. The Absorption Properties of Raw Materials

Figure 5 shows the temperature rise characteristic curves of the chromite and the solid sodium carbonate respectively in the microwave field. Chromite concentrate heated to 1000 °C in 6 min and the heating rate was 167 °C/min, then the solid sodium carbonate was raised 1000 °C in 13 min and the heating rate was 77 °C/min. From the temperature rise curves, chromite and solid sodium carbonate have good wave absorbing properties.

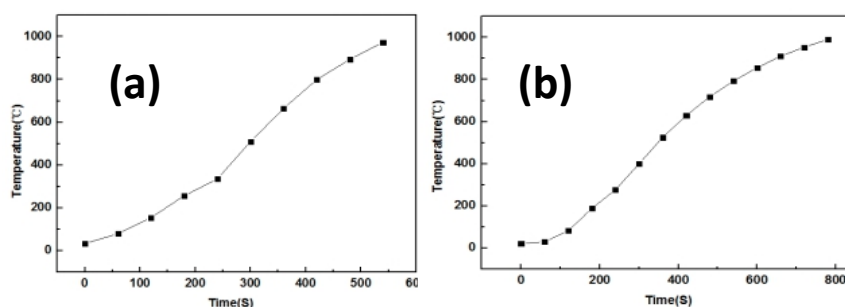**Figure 5.** Temperature rise characteristic curves in the microwave field of chromite (**a**) and Na_2CO_3 (**b**).

3.2. The Study Dielectric Constant and Dielectric Loss Factor of Raw Material

Figures 6 and 7 show the dielectric constant and dielectric loss factor of the chromite and sodium carbonate respectively. The increasing trend of dielectric loss and dielectric loss factor with the temperature, indicates the chromite concentrate and sodium carbonate have good wave absorbing properties.
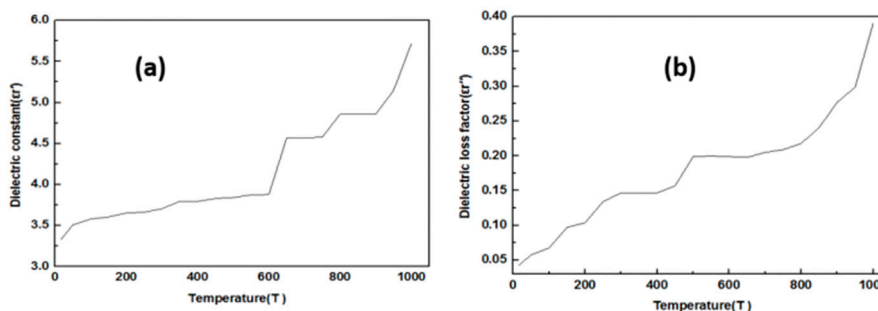

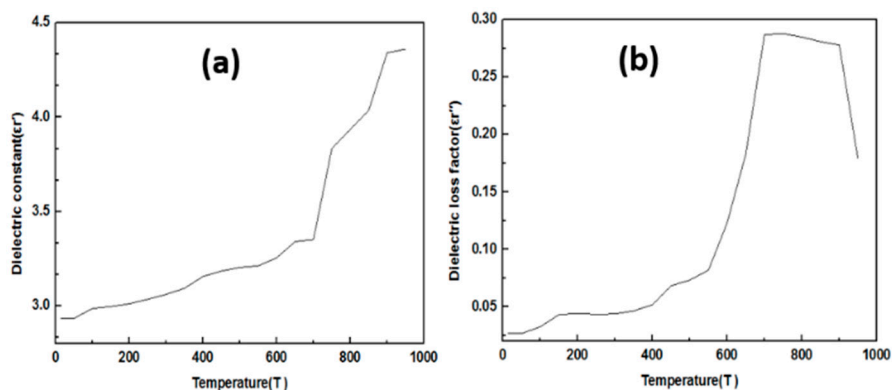**Figure 6.** Dielectric constant (**a**) and loss factor (**b**) of chromite with temperature.

Figure 7. Dielectric constant (**a**) and loss factor (**b**) of Na_2CO_3 with temperature.

3.3. Characterization of the Roasted Material

3.3.1. SEM Analysis

Figure 8 represents the SEM image of the roasted products. It clearly shows that the surface of roasted products covered by a moderate quantity of melt after roasted. It can also be observed that the micro thermal fragmentation of the particles in the mixture of sodium carbonate and chromite. The SEM images show the aggregation of the particles with some pores.
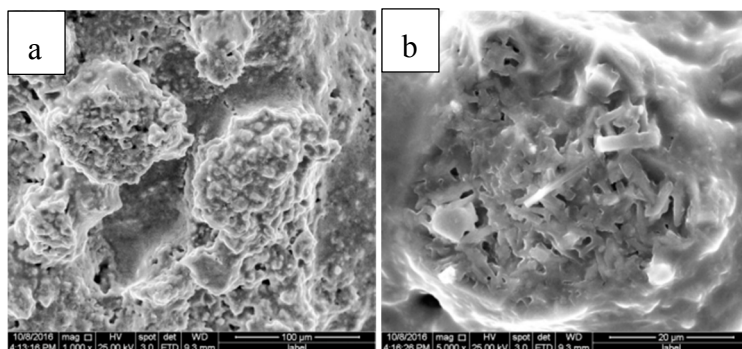

Figure 8. SEM images (**a**,**b**)of microwave roasted reaction products.

3.3.2. XRD Analysis

Figure 9 shows the XRD pattern of the product after microwave roasted. The $FeO \cdot Cr_2O_3$ and Na_2CO_3 reacted chemically producing Na_2CrO_4. Additionally, Al_2O_3 and Na_2CO_3 reacted to generate $NaAlO_2$. At the same time, Fe_2O_3 also existed.
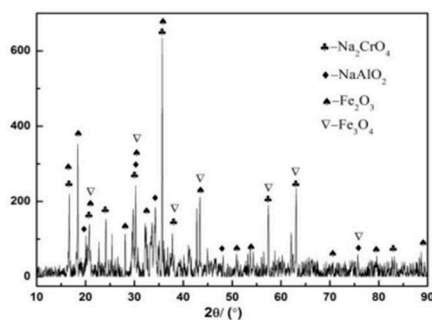

Figure 9. XRD pattern of microwave roasted product.

Figure 10 shows the XRD pattern of slag after ultrasonic extraction, which shows the existence of Fe_2O_3, Fe_3O_4 and $Al(OH)_3$. Compared to the XRD patterns of chromite concentrate before microwave roasted and ultrasonic extraction, only Fe_2O_3 and Fe_3O_4 are existing. Other components removed.
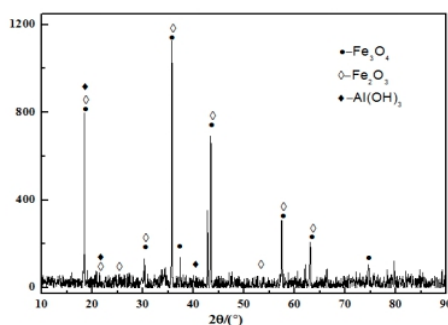

Figure 10. XRD pattern of slag after ultrasonic extraction.

Figure 11 illustrates the XRD pattern of the final product. The XRD pattern clearly suggests the formation of $K_2Cr_2O_7$. The synthesized potassium dichromate crystals existed in monoclinic crystal system. However, the existence of the small amount of K_2CrO_4 indicates that the pH not controlled well; therefore, Na_2CrO_4 not completely transformed into $Na_2Cr_2O_7$. From the determination, the purity of the resultant product, the potassium dichromate crystals purity is 67.4%.
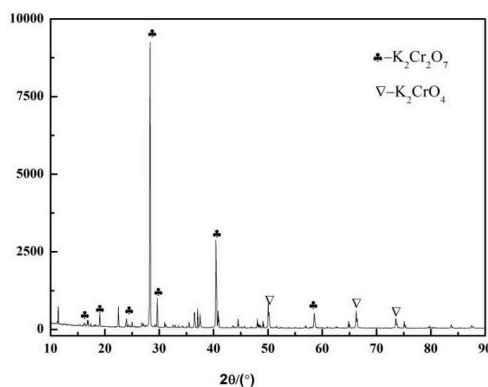

Figure 11. XRD pattern of the final product.

3.3.3. XRF Analysis

Table 2 represents the brief chemical component of products after microwave roasted and Table 3 represents brief chemical components of slag after ultrasonic extraction. After microwave roasted O, Cr, Na elements are present in large quantities. Comparison of both tables indicates that after ultrasonic extraction other elements such as Fe, Mg, Al, Si and Ca are all more than roasted, which shows in the process of ultrasonic extraction is beneficial. However, in this process, Cr has not been completely converted. By XRF analysis, a high conversion rate of Cr is 94.57%, which is reasonable and after ultrasonic extraction, the conversion rate of Cr is 82.72%, this is an acceptable number to be optimistic.

Table 2. Brief chemical components of the products after microwave roasted.

O	Cr	Na	Fe	C	Mg	Al	Si	Ca
65.71	33.27	45.95	6.20	5.11	2.42	1.89	0.83	0.29

Table 3. Brief chemical components of the slag after ultrasonic extraction.

O	Cr	Na	Fe	C	Mg	Al	Si	Ca
46.48	11.18	9.52	17.67	2.82	7.04	4.12	3.11	0.74

3.4. The Purity of the Product

According to the formula of (2)–(4), the purity of final $K_2Cr_2O_7$ product was calculated. After microwave roasted and ultrasonic extraction, the purity of the product is up to 67.4%, compared with the conversion rate of Cr after microwave roasted that is a little bit down. Since the acidity has not been controlled ideally, the Na_2CrO_4 having not been completely transformed into $Na_2Cr_2O_7$. Therefore, the final product $K_2Cr_2O_7$ includes small amount of K_2CrO_4.

4. Conclusions

Using chromite and sodium carbonate for the preparation of potassium dichromate crystals in the microwave field is reasonable because chromite and sodium carbonate having good wave absorbing properties, and it takes only ten minutes in the heating process. The conversion rate of Cr is 94.57% after microwave roasted. The Ultrasonic extraction can strengthen the extraction efficiency and has a good effect on the conversion rate of Cr. Finally, the product obtained shows that the purity of product is up to 67.7% of microwave heating and ultrasonic extraction. The present work suggests that the microwave field is ideal for the preparation of potassium dichromate crystals. However, controlling the reaction parameters such as pH influence the final product purity.

Author Contributions: Linqing Dai and Shenghui Guo conceived and designed the experiments; Hua Liu performed the experiments and wrote the paper; Yu Duan analyzed the data; Jinhui Peng and Libo Zhang contributed reagents and materials and analysis tools.

Conflicts of Interest: The authors declare no conflict of interest.

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