Research Article

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Characteristics and purification of Himalayan salt by high temperature melting

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Abstract: Himalayan rock salt contains a variety of minerals and trace elements, which is conducive to human health. The solutions of black rock salt and rose salt are alkaline, and the content of water insoluble matter is 0.34 and 0.083%, respectively. The element composition of water insoluble matter in rock salt is determined and analyzed. It is found that the main component of two kinds of rock salt water insoluble matter is soil. Due to the presence of water insoluble matter in rock salt, according to the different specific gravity of molten sodium chloride and insoluble matter, rock salt was purified by high-temperature melting method. Rose salt is mainly studied during purification. The results showed that the content of insoluble matter in rose salt decreased from 0.083 to 0.0024% after holding at 950°C for 40 min; the contents of arsenic, barium, and lead decreased to 0.0032, 0.61, and 0.21 mg·kg $^{-1}$, respectively; the content of sodium increased to 39.24%, the contents of calcium, magnesium, and iron reached to 2,200, 855, and 1.31 mg·kg⁻¹, respectively.

Keywords: Himalayan salt, insoluble matter, purification, high temperature melting

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1 Introduction

Common salt is a necessity of human life, and NaCl is the indispensable material to maintain the normal physiological development of human body. At the same time, it is also the basic raw material of chemical industry. It is also widely used in agriculture and other industries [1,2]. The raw material sources of salt can be divided into four categories: sea salt, lake salt, well salt, and rock salt. Salt made from seawater is called "sea salt," salt produced by mining Modern Salt Lake minerals is called "lake salt," that produced by extracting shallow surface or underground natural brine by shaft sinking is called "well salt," and salt produced by mining ancient rock salt deposits is called "rock salt." Because rock salt deposits sometimes coexist with natural brine salt mines, and the advent of drilling water solution method for mining rock salt deposits, it is also known as "well salt" and "rock salt" - well mineral salt, or generally known as "mineral salt" [3-5].

Himalayan rock salt comes from Kwara, Karabakh, and other mines in about 300 km southwest of the Himalayas [6,7]. It has a unique pink or black color, while black salt is less common. Himalayan rock salt is rich in minerals. In addition to sodium and chlorine, there are other trace elements such as chromium, iron, and zinc. Therefore, it is not only used in high-grade dishes, but also often seen in some families, which is very popular [8,9].

The Himalayan rock salt is characterized by its unique pinkish and black color. Himalayan black rock salt has cooking and medicinal properties. In the western Himalayas of India, almost every household uses black rock salt as a folk prescription for gastrointestinal diseases in case of emergency. Black rock salt has amazing curative effects not only in folk medicine, but also in India's traditional medical system. This salt contains various trace and large elements, such as iron, potassium, and calcium. It is considered to have a good effect in fighting heartburn, abdominal distention, and flatulence [10–12]. Another salt of Himalayan rock salt is called rose salt or pink salt because its color is pink. It is a "treasure in salt," a "salt fossil" formed by the combination of

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underground minerals and sea salt after hundreds of millions of years of geological compression and underground high temperature. It is pure, pollution-free, and rich in a variety of minerals and trace elements. It is 2-3 times of ordinary sea salt and more than 10 times of ordinary table salt. Rose salt is widely used. It can be used to make salt lamps, take a bath, cook delicious food, and increase the flavor of food. Rose salt can reduce renal pressure. It is suitable for people with high blood pressure and mood swings. At the same time, ordinary salt metabolism needs a lot of water in the body, which is easy to lead to chronic dehydration of cells. However, rose salt has low salinity and faster metabolic time. In addition, the proportion of salt in the body is related to the clarity of muscle lines in healthy people. Too much salt in the body will lead to muscle water storage, but rose salt has low salinity, so it is more suitable for healthy people [13–15]. However, it contains a certain amount of insoluble matter, but no purification studies have been conducted.

Taking Himalayan rock salt as the research object, this work studies the characteristics of its salt solution and the content and composition of water insoluble substances. The high-temperature melting method is used to remove harmful impurities in rock salt and retain a variety of beneficial elements [16–18].

2 Experimental method

2.1 Samples

The Himalayan rock salt used in the experiment comes from the Himalayan rock salt vein on the Pakistani side. The rock salt is rich in iron, so it is pink, a natural color, while the black rock salt is black. Meanwhile, the rock salt has a faint sulfur taste.

2.2 Radioactivity detection of rock salt

Radioactivity of minerals generally refers to uranium (U), thorium (Th), radium (Ra), and other radioactive elements. These nuclides can spontaneously release particles or rays from the interior of the atomic nucleus and release energy at the same time. They are radioactive and can endanger human health. Himalayan rock salt belongs to a kind of mineral. The four radioactive elements ²³²Th, ²²⁶Ra, ⁴⁰K, and ²³⁸U in black rock salt and rose salt are tested. The radioactivity test results of the two salts are shown in Table 1.

Table 1: Radioactivity test results of rock salt

	Test items – C _(B) /Bq·kg ⁻¹					
	²³² Th	²²⁶ Ra	⁴⁰ K	²³⁸ U		
Black rock salt	_	-	80.3	_		
Rose salt	_	_	94.4	_		

Note: "-" indicates that it is not detected, Bq·kg $^{-1}$ indicates the activity concentration, and the activity concentration limit of 232 Th, 226 Ra, 40 K, and 238 U is: any nuclide in 232 Th, 226 Ra, and 238 U decay system ≤ 1 Bq·g $^{-1}$ and 40 K ≤ 10 Bq·g $^{-1}$.

According to Table 1, the radioactivity of ²³²Th, ²²⁶Ra, and ²³⁸U in black rock salt and rose salt cannot be detected as the salts do not contain these three radioactive elements, while the ⁴⁰K content in black rock salt and rose salt is 80.3 and 94.4 Bq·kg⁻¹, respectively, far lower than the activity concentration limit of 10 Bq·g⁻¹, indicating that the two salts are not radioactive and harmless to human body.

2.3 Thermogravimetric analysis of rock salt

The thermogravimetric analysis of rose rock salt is shown in Figure 1. It can be seen from Figure 1 that at about 800°C, rose salt absorbs a lot of heat, which is the melting point of the salt. Rock salt needs a lot of heat absorption to change it from solid state to molten state. It absorbs and releases heat repeatedly between 800 and 1,000°C, and slowly releases heat to stable state after absorbing heat at 950°C. The quality starts declining from 800°C. And there are small amount of bound water in black salt, which decomposes at 400–500°C in black salt.

2.4 Method

Because Himalayan rock salt contains water insoluble substances and other harmful components, dissolution and filtration methods and high temperature melting method are used for impurity removal and purification, and some detection methods are used to characterize the purified rock salt.

2.4.1 Purification of rock salt by dissolution and filtration methods

Weigh a certain amount of black rock salt and rose salt and dissolve them in normal temperature deionized

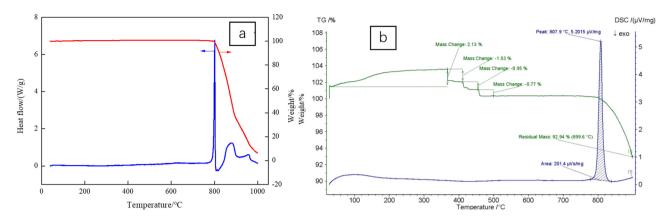


Figure 1: Thermogravimetric curve of rock salt samples: (a) rose salt and (b) black salt.

water 4–5 times the mass of rock salt, respectively, and stir the two salt solutions with a stirrer at the same time. The stirring time is 30 min and the rotating speed is 350-410 rpm, so that the rock salt is completely dissolved. The two completely dissolved rock salt solutions are allowed to stand and precipitate for 30-60 min, and then filtered with a $0.22\,\mu m$ filter membrane under a vacuum of 0.05-0.06 MPa to separate the water insoluble matter from the solution.

2.4.2 Purification of rock salt by high temperature melting method

Weigh a certain amount of black rock salt, put the rock salt into corundum crucible, then put it into muffle furnace, heat it from normal temperature to 850°C, the heating rate is 5–10°C·min⁻¹, and cool it naturally after holding for 20 min. According to the above steps, when the temperature is 850°C, set the holding time to 40 and 60 min, respectively, and compare the changes in rock salt in three different holding times at the same temperature. Then, select the holding time of 40 min and the temperatures of 900 and 950°C, respectively. Compare the changes in rock salt after baking at three different temperatures under the same holding time.

Put the rose salt into the corundum crucible and put it into the high-temperature crucible furnace, raise the temperature from normal temperature to 950°C, the heating rate is 5–10°C·min⁻¹, and keep the temperature at 950°C for 20, 40, and 60 min, respectively. After the heat preservation, close the electric furnace and cool naturally to normal temperature. Take out the cooled rose salt and separate the upper purified salt from the bottom insoluble matter to achieve the purpose of purification. The specific process is shown in Figure 2.

2.5 Detection and analysis

The insoluble matter in rose brine was detected by X-ray fluorescence (XRF) and X-ray diffraction (XRD), inductively coupled plasma mass spectrometry was used to detect and analyze lead, arsenic, cadmium, mercury, and barium in rose salt after high temperature melting purification, inductively coupled plasma optical emission spectrometry was used to detect the contents of sodium, calcium, magnesium, iron, and aluminum in purified rose salt.

3 Results and discussion

3.1 Characteristics of rock salt

3.1.1 Solution characteristics of rock salt

After dissolving the black rock salt and powder salt, the color of the solution is black and pink, respectively. There are sediments at the bottom of the solution. The black rock salt solution smells of rotten eggs. It can be seen from Tables 2 and 3 that in addition to sodium and chloride ions, the solutions of the two rock salts also contain a certain amount of potassium, aluminum, calcium, silicon, manganese, zinc, and other elements. The color and smell of the solutions are caused by the compounds composed of these ions. The filtered solution is colorless, so the color of the solution may be caused by the presence of water insoluble substances. The pH value, conductivity, and density of the two solutions before and after filtration are shown in Table 4. The contents of elements in the salt solution of black rock salt and powder salt are shown in Tables 3 and 4, respectively. It can be seen from Table 2 that the pH values of

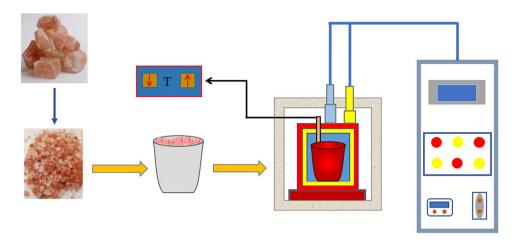


Figure 2: High temperature melting purification process of rock salt.

Table 2: Composition and content of elements in black rock salt solution (μg·mL⁻¹)

Na	Mg	Al	Cu	Fe	К	Mn	Ca	Si	Р	Ti	Cl-
8,940	<0.001	1.572	<0.001	<0.001	1,525	<0.001	1.009	1.328	1.73	<0.001	13,058
Ni	Zn	As	Rb	Ва	Sr	Ag	٧	Cd	Co	Li	SO ₄ ²⁻
<0.001	<0.001	<0.001	<0.001	0.0087	0.4887	<0.001	0.1543	<0.001	<0.001	<0.001	185

the two rock salts before filtration are 11.29 and 11.13, respectively, which are alkaline. After filtration, the pH value of the solution is reduced to 7.22 and 8.10 due to the separation of the solution from insoluble matter, indicating that the salt solution of rock salt itself is close to neutral, while the insoluble matter contains alkaline matter, which makes the salt solution before filtration alkaline.

3.1.2 Insoluble properties of rock salts

The content and composition of water insoluble matter after filtration of the two rock salt solutions are shown in Tables 5–7 and Figure 3, respectively.

It can be seen from Table 5 that the content of water insoluble matter in black rock salt and rose salt is 0.34

and 0.083%, respectively, and the content of water insoluble matter in black rock salt is more than that in rose salt. It can be seen from Table 6 and Figure 3 that the insoluble matter of black rock salt mainly contains elements such as oxygen, sodium, iron, chlorine, and calcium, and the compounds are mainly NaCl and CaCO₃, as well as a small amount of CaO, KFeO₂, Fe₂Si, etc. It can be seen from Table 7 and Figure 3 that the water insoluble matter of rose salt mainly contains elements such as oxygen, calcium, magnesium, chlorine, sodium, sulfur, silicon, aluminum, potassium, and iron, which is consistent with most elements in soil. The main components of insoluble matter are MgCO₃, SiO₂, and K₃VO₄, which are more in line with the composition characteristics of soil. It is judged that the main component of water insoluble matter is soil. The filtered rock salt solution is colorless,

Table 3: Composition and content of elements in rose salt solution ($\mu g \cdot mL^{-1}$)

Na	Mg	Al	Cu	Fe	K	Mn	Ca	Si	Р	Ti	Cl⁻
8044.5	111.3	0.103	0.0264	<0.001	1,839	4.978	653.2	0.8356	0.118	<0.001	11,658
Ni	Zn	As	Rb	Ва	Sr	Ag	٧	Cd	Со	Li	SO ₄ ²⁻
<0.001	63.63	<0.001	<0.001	0.0436	2.781	< 0.001	<0.001	< 0.001	< 0.001	< 0.001	171

Table 4: pH value, conductivity, and density of rock salt solution before and after filtration

		рН	Conductivity	Density (g⋅mL ⁻¹)
Black rock salt	Before filtration	11.29	-252.8	1.15
	After filtration	7.22	31.5	1.10
Rose salt	Before filtration	11.13	245.2	1.17
	After filtration	8.10	89.4	1.16

Table 5: Two kinds of insoluble salt content

-	Insoluble content (%)
Black rock salt	0.34
Rose salt	0.083

and the colors of insoluble matter are black and red, respectively, indicating that the color of rock salt is caused by water insoluble matter. This is because rock salt is formed underground in the Himalavas. Under the influence of geology, the salt dam is combined with a small amount of mud to form a salt block - Himalayan rock salt.

3.2 Separation and purification

3.2.1 Rose salt melting and impurity removal process

The Himalayan rose salt has pink characteristics, which makes people have an appetite and contains a variety of beneficial trace elements, while the black rock salt has a strong sulfur flavor, black appearance, and high impurity

content. Therefore, only the impurity removal of rose salt is studied when using high temperature melting purification.

The rose salt is melted at different temperatures and holding times, and the results are shown in Table 8. It can be seen from Table 8 that with the same temperature, the longer the holding time, the greater the mass loss rate of rock salt. In the three holding times of 20, 40, and 60 min, the mass loss rate is 0.25, 0.31, and 0.31% respectively. At 40 and 60 min, the mass loss rate remains unchanged, indicating that holding for 40 min is enough. At the same holding time of 40 min, the mass loss rate at 850, 900, and 950°C is 0.31, 0.38, and 0.38%, respectively, indicating that the increase in temperature will increase the mass loss rate. The impurity removal temperature can be 900 or 950°C.

3.2.2 Precipitation effect of insoluble matter in water during different heat preservation periods

The rose salt is melted and purified at 950°C, and the impurity removal process is shown in Figure 4. It can be seen from Figure 4 that the high-temperature melting method uses the different specific gravity of molten rock

Table 6: XRF detection of insoluble matter in black rock brine (%)

0	Ca	Cl	Na	Mg	S	Fe	Si	Р	Al	K
28.8408	18.0122	11.2702	11.1428	7.0060	6.5842	6.2072	2.4026	1.7937	0.8644	0.2888
Ti	٧	Mn	Ni	Cu	Zn	Rb	Sr	Ва	Pb	
0.1115	0.0256	0.1768	0.0218	0.0258	0.0655	0.0014	0.3004	0.042	22 0.01	103

Table 7: XRF detection of insoluble matter in rose brine (%)

0	Ca	Cl	Na	Mg	S	Fe	Si	Р	Al	K
41.6182	3.2991	4.0763	2.3235	16.3533	5.2194	1.4520	9.1819	0.0363	2.9107	4.2301
Ti	F	Cr	Ni	Cu	Zn	Rb	Sr	Ва	Pb	
0.2101	0.3900	0.0059	0.0218	0.0150	0.0116	0.0050	0.0288	0.0098	0.00	38

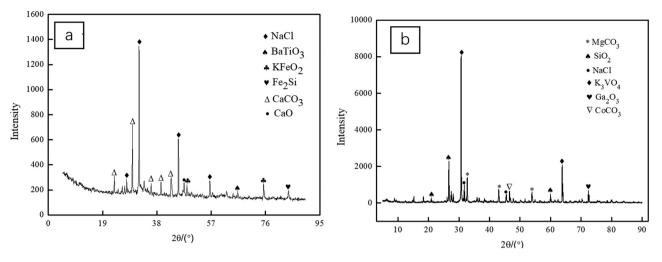


Figure 3: XRD patterns of water insoluble matter of (a) rose salt and (b) black salt.

Table 8: Variation in mass loss rate in the melting process of rose salt

	Temperature (°C)	Holding time (min)	Mass loss rate (%)
Black	850	20	0.25
rock salt	850	40	0.31
	850	60	0.31
	900	40	0.38
	950	40	0.38

salt and water insoluble matter to separate them from each other. Maintain the temperature for 20, 40, and 60 min, take out the recrystallized rose salt, measure the height of the upper purified salt and the bottom insoluble matter, respectively, and calculate the proportion of these two parts to the total height of the salt column. The results are shown in Table 9. Table 9 shows that at the same melting temperature, with the increase in time, the height proportion of the upper purified salt in the total salt column gradually decreases, while the height proportion of the bottom insoluble matter always increases, indicating that the insoluble matter precipitates more thoroughly with the extension of

Table 9: Proportion of purified salt and bottom insoluble matter to the total height of salt column under different holding time (%)

Insulation duration (min)	20	40	60
Upper purified salt	93.33	90.56	90.44
Bottom insoluble	6.67	9.44	9.56

holding time. The height ratio of the two parts is almost the same at 40 and 60 min, indicating that the insoluble matter has basically precipitated completely at the time of heat preservation for 40 min. Continuing to extend the heat preservation time will increase the process cost, but the effect is not obvious. Therefore, heat preservation for 40–60 min at 950°C can achieve a good purification effect for purifying Himalayan rose salt.

3.2.3 Changes in insoluble matter and element content of rose salt before and after purification

The rose salt is purified at 950°C and kept warm for 40 min. The insoluble matter and some element contents

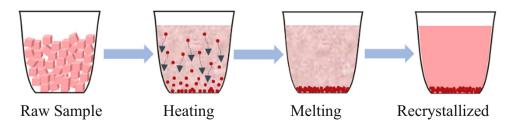


Figure 4: Rose salt melting process for impurity removal.

Table 10: Contents of insoluble matter in raw material and purified rose salt (%)

Rose salt raw material	Upper purified salt	Bottom insoluble		
0.083	0.0024	0.57		

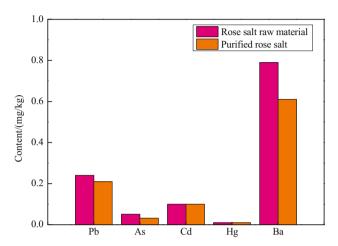


Figure 5: Changes in Pb, As, Cd, Hg, and Ba in rose salt before and after purification.

of the rose salt raw material and purified salt are shown in Table 10, Figure 5, Tables 11 and 12, respectively.

It can be seen from Table 10 that the content of insoluble matter in rose salt raw material is 0.083%, and the content of insoluble matter in purified rose salt is only 0.0024%. Most of the insoluble matter precipitates to the bottom during the purification process, so that the content of insoluble matter at the bottom is as high as 0.57%. After high temperature melting purification, the insoluble matter is distributed in two parts, most in the bottom and a small amount in the upper part, which is characterized by the different specific gravity of sodium chloride and insoluble matter. In the high temperature melting and baking process, when the rose salt becomes molten, the insoluble matter can be free in the salt. Due to the different specific gravity of the two, the specific gravity of the insoluble matter is greater than that of sodium chloride, so it will deposit at the bottom, thus sodium chloride and

Table 11: Content of lead, arsenic, mercury, cadmium, and barium in purified rose salt $(mg \cdot kg^{-1})$

	Pb	As	Cd	Hg	Ва
Rose salt raw material	0.24	0.051	0.081	0.0013	0.79
Purified rose salt	0.21	0.032	0.080	0.0012	0.61

Table 12: Changes in contents of Na, Ca, Mg, Fe, and Al in rose salt before and after purification $(mg\cdot kg^{-1})$

	Na	Ca	Mg	Fe	Al
Rose salt raw	3.871×10^5	2,600	1,048	2.21	0.272
Purified rose salt	3.924×10^5	2,200	855	1.31	0.268

insoluble matter can be separated and the effect of purification can be achieved. It can be seen from Figure 5 and Table 11 that after purification, the contents of lead, arsenic, and barium in rose salt are reduced, among which the effects of arsenic and barium are the most obvious, reduced to 0.032 and 0.61 mg·kg⁻¹, respectively, and lead is also reduced to 0.21 mg·kg⁻¹. The contents of cadmium and mercury are low. The content of cadmium before and after purification is less than 0.1 mg·kg⁻¹, while that of mercury is less than 0.01 mg·kg⁻¹. After high temperature melting purification, it has a certain removal effect on harmful elements in rose salt, which may be due to the precipitation of some harmful elements to the bottom with water insoluble substances in the process of high-temperature melting. Table 12 shows that the contents of calcium, magnesium, and iron reach 2,200, 855, and 1.31 mg·kg⁻¹ respectively, which are lower than the contents of these three elements in rose salt raw materials. High temperature melting purification has a certain impact on the contents of these three elements. After purification, the content of sodium increased from 3.871×10^5 to 3.924 \times 10⁵ mg·kg⁻¹, which may be due to the decrease in other trace elements and the increase in sodium content.

4 Conclusion

The salt solutions of black rock salt and rose salt are alkaline, with pH values of 11.29 and 11.13 respectively, and contain calcium, magnesium, potassium, zinc, iron, and other elements. The water insoluble content of the two rock salts is 0.34 and 0.083%, respectively. Water insoluble matter was detected by XRF and XRD. The results show that soil is the main component. The rock salt solution is colorless after filtration, indicating that the color of the solution is caused by insoluble substances. Impurities in Himalayan black rock salt and rose salt are basically insoluble in water. Solution filtration can separate water insoluble matter from solution.

Impurities can be removed from rose salt by melting at high temperature. When the holding time is 40 min at 950°C, the effect of impurity removal is better. The impurity

content in purified rose salt decreased from 0.083 to 0.0024%, which achieved good purification effect. High temperature purification has the greatest impact on arsenic and barium, and the contents are reduced to 0.032 and 0.61 mg·kg⁻¹, respectively, and lead is also reduced to 0.21 mg·kg⁻¹; the content of sodium increased to 39.24%, and the contents of calcium, magnesium, and iron reach 2,200, 855, and 1.31 mg·kg⁻¹, respectively. The effect of purifying Himalayan rock salt by high temperature melting method is good, and the process is simple. The purified rose salt contains a variety of beneficial components with low impurity content and pink characteristics.

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