Progress of joint process research on purification of high purity quartz sand

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Abstract. High-purity quartz sand is an important raw material for semiconductors, photovoltaics, aerospace and other cutting-edge fields, high-quality natural crystals can no longer meet the urgent demand for high-purity quartz in the domestic market, and it is imperative to accelerate the realisation of high-quality quartz purification technology from non-quality mineral sources. This paper summarises the joint process flow in the unused scenario, and outlines the main methods for the purification of quartz sand in China at present, including pretreatment, ultrasound-assisted leaching, chlorination roasting, etc., from the point of view of the joint process, and puts forward the problems that exist in these methods, so as to provide certain references for the research on the purification process of high-purity quartz sand in China.

Keywords: High-purityquartz sand, Purification, Combined process, Pretreatment, Chlor ination roasting.

1. Introduction

High-purity quartz sand refers to solid particles with SiO_2 content above 99.9%, which has the advantages of high hardness, low expansion coefficient, corrosion resistance, high temperature resistance, etc., and has a wide range of applications in high-tech industries such as semiconductors, solar photovoltaics, aerospace, precision optics, etc. [1, 2]. since the 1960s, along with the development of new materials, computers and other emerging technologies, there are higher requirements for quartz purity. With the development of new materials, computers and other emerging technologies since the 1960s, the purity of quartz has higher requirements, and the concept of high-purity quartz has been put forward. China's preparation of high purity quartz began in the 1980s, and the raw materials for the early production of high purity quartz mainly came from natural high-quality crystals, and the natural crystals were crushed, magnetically selected, flotation, acid leaching, drying, roasting, and other simple processes to get the finished quartz sand [3, 4]. In recent years, the demand for high purity quartz in China's market has gradually increased, and the shortage of high-quality quartz raw ore resources has become increasingly obvious. At the same time, due to the late start of the purification technology of high purity quartz sand in China, it further aggravates the unbalanced development of the supply and demand of high quality quartz sand [5]. Therefore, with the gradual depletion of natural crystals and high-quality quartz ore sources, in order to meet the domestic demand for high-purity quartz market, the study of quartz sand purification technology is of great significance [6].

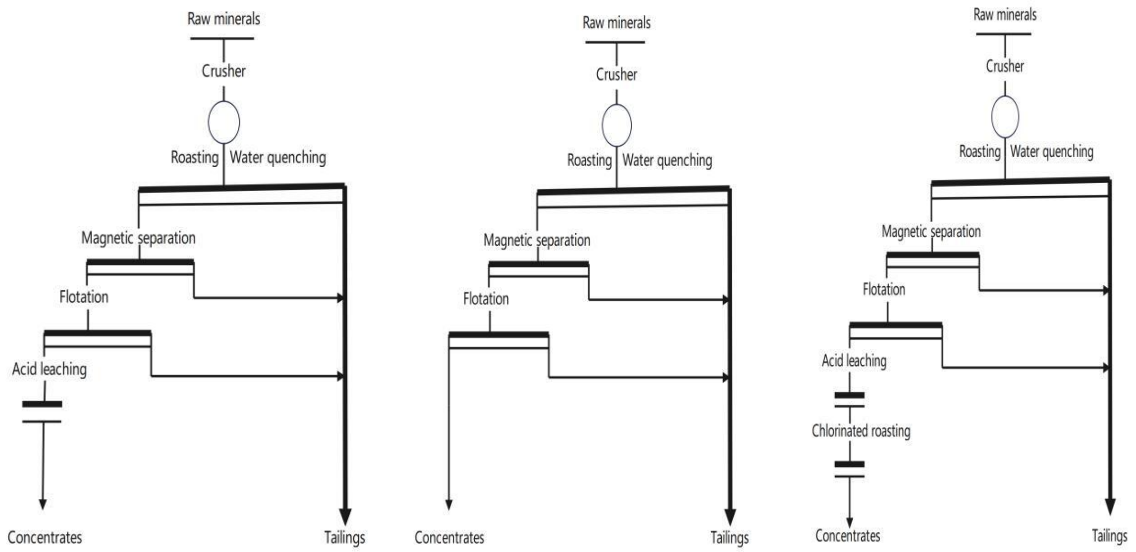
2. Joint Process

High purity quartz sand needs to be prepared through a combination of processes. Combined processes generally include: pretreatment-magnetic separation-flotation-acid leaching-chlorination roasting, etc. The above purification processes can be matched as needed, using different combinations of processes obtained by the purity of the quartz sand has a great deal of difference.

For example, the process of crushing and sieving-roasting and water quenching-magnetic separation and flotation can be applied to the recycling of quartz tailings and the preparation of low-end quartz products. Wang et al [7] used the process of crushing, screening combined with magnetic separation and flotation to recover and purify quartz from gold mine tailings, and the SiO_2 content of the resulting product was 99.91%. In addition, Zhao et al [8] processed wolfram tailings by grinding-strong magnetic separation-anyway flotation, and the mass fractions of Al_2O_3 and Fe_2O_3 in the obtained concentrates were 0.61% and 0.09% respectively, which can meet the requirements of raw materials for quartz plates. The principle process flow is shown in Fig 1(a). The quartz sand obtained by this method can only be used as low-end quartz raw material without acid leaching treatment, which is far from meeting the requirement of high-quality quartz.

The purity of quartz is further improved by adding the acid leaching purification process on the basis of the above process, and the effect of acid leaching purification under different external environments is slightly different. Du [9]purified quartz from a region in Gannan, which was leached by ammonium sulfate activated roasting - hydrofluoric acid mixed acid at atmospheric pressure, and the SiO_2 content of the resulting sample was 99.99wt%. Li et al [10] increased the SiO_2 purity from 99.917% to 99.994% by hot-pressure leaching of roasted quartz sand for 6 h through the roasting-water quenching-hot-pressure mixed acid leaching process.Li et al [11] removed more than 99.8% of Fe impurities in quartz through microwave heating and ultrasound-assisted acid leaching treatment by combining microwave treatment for 30 min at 400℃. The sample obtained was 99.99wt% of Fe impurities in quartz. Yang et al [12] carried out advanced purification of industrial quartz by calcination pretreatment combined with ultrasound-assisted leaching, and the SiO_2 content increased from 99.6828% to 99.9047%. The purification effect of quartz after combining with acid leaching was significantly improved, and the acid leaching effect under hot-pressure environment was slightly better than that under atmospheric pressure, and the acid leaching effect under ultrasonic environment was affected by frequency and time to be further studied. The principle process flow is shown in Fig. 1(b).

The impurities in the quartz lattice could not be effectively removed using traditional purification methods, and chlorine roasting of the acid-impregnated quartz sand could remove the impurities in the lattice to a large extent. Wu [13] optimized the traditional process of pretreatment, flotation, acid leaching and studied the effect of chlorination roasting with different chlorinating agents, and determined NH_4Cl as the chlorinating agent, the chlorination temperature of 600 ℃, and 2h as the optimal experimental conditions, and the main impurity content of the resulting quartz product was only 3.107×10^{-7}. Song [14] proposed a simple method for rapid removal of aluminum impurities in quartz by microwave treatment combined with chlorinated calcination, and the removal rate of aluminum impurities in quartz could reach 97.58% by chlorinated calcination at 900℃ for 2h in microwave.The combination of traditional purification technology and chlorine roasting combined process makes the purity of quartz sand to another level, the amorphous lattice impurities and lattice impurities in the quartz sand are effectively removed, and this type of combined process provides a direction for the preparation of ultra-high purity quartz sand. The principle process flow is shown in Fig 1(c).



(a) (b) (c)

Fig.1 Combined process flow of quartz sand purification

3. Preprocessing

Quartz sand purification process, its pretreatment is essential. The main purpose of pretreatment is to carry out preliminary screening of quartz: quartz ore crushed to a certain size; will be attached to the surface of the quartz clay and other impurities to remove; so that the gas-liquid inclusions in the quartz rupture, the impurities outside. Pre-treatment generally includes crushing, screening, scrubbing, roasting, water quenching steps.

3.1 Crushing-Screening-Scrubbing.

Crushing refers to obtaining quartz particles with appropriate particle size under the action of mechanical external force, while scrubbing is to remove clay impurities, rust, film, etc. on the surface of quartz on the basis of crushing. Niu [15] obtained a concentrate with 99.81% SiO_2, 0.023% Fe_2O_3, 0.051% Al_2O_3, 0.021% TiO_2 by using ball milling and scrubbing, and grading and desliming. Tian [16] obtained quartz samples with 80-140 mesh, 99.14% SiO_2 grade and 0.067% Fe_2O_3 content by using a crusher for coarse crushing of the raw ore and a vibrating sieve machine after placing the screened samples into a roller machine for fine crushing treatment. The quartz sand produced by the above traditional crushing and scrubbing methods tends to introduce mechanical impurities in the crusher and the dissociation degree is not high, the use of new equipment for crushing the raw ore can effectively solve such problems. Zhang [17] used high voltage pulse discharge equipment for magnetite quartz ore crushing, compared with the traditional mechanical crushing methods, high voltage pulse discharge crushing in 100 pulses, the same crushing 3min in the case of particle size -0.074+0.045mm monomer dissociation degree is higher than the traditional mechanical crushing method of 24.88%. Qin et al. [18] on the magnetite quartzite by high-voltage pulse discharge produced a new understanding of the fracture mechanism. The release rate of mill products increased by 20.93% for a pulse number of 300 at a milling time of 3 min. High-voltage pulse discharge significantly increased the release rate of magnetite quartzite. The use of high voltage pulse discharge equipment can improve the efficiency and quality of crushing, the disadvantage is that the equipment is expensive, high maintenance cost and high energy consumption. Comparison of the effects of conventional crushing of quartz and high voltage pulsed discharge crushing of quartz is shown in Table 1.

3.2 Roasting-Water Quenching.

Roasting is the use of professional roasters to roast the quartz raw ore particles at 300℃-1500℃ for 2-5 hours, while water quenching is based on roasting by placing the roasted quartz particles in cold water for rapid cooling to remove the vapor bubbles, water streaks, and some encapsulated impurities inside the minerals. Zhang [19] studied the effect of changing the temperature and time of roasting-quenching on the structure and appearance of quartz crystals as well as on the removal of Al and Fe. The results showed that the crystals of quartz were microwave roasted at 900℃ for 1h could induce the outward migration of the impurity Fe and enrich it, which could greatly improve the removal of the impurity Fe. Guo et al [20] used the method of vacuum roasting to make the quartz substrate produce cracks , inclusions were opened, providing a direct channel for the escape of water from the inclusions, reducing the water content of quartz from 226.8 $\mu g \cdot g^{-1}$ to 88.2 $\mu g \cdot g^{-1}$, comparable to that of first-order crystals.Prasetyo et al [21], by roasting quartz sand with the addition of sodium carbonate, determined that a readily extractable sodium silicate mucilage could be formed at a mixed roasting temperature of 1200℃, and that then, after a variety of subsequent processes The silica precipitates obtained contained up to 99.99% SiO_2. The gas-liquid inclusions in the quartz crystals after roasting-water quenching treatment rupture and expose the impurities, which lays the foundation for the further purification of quartz.

Table 1 Dissociation degree of monomer of each particle size for 3min with different crushing methods[17]

	Mechanical crushing		100 pulses	
Particle size	Grain yield/%	Monomer dissociation/%	Grain yield/%	Monomer dissociation/%
+0.074	26.68	48.36	26.86	73.24
-0.074+0.045	15.33	55.69	14.31	65.36
-0.045+0.037	51.63	59.61	53.26	56.32
-0.037	6.36	49.63	5.57	48.32
Average monomer dissociation	55.37		61.71	

From the above literature can be summarized that: 1. The use of high-voltage pulse discharge equipment on the crushing effect of quartz ore compared with the traditional mill has the advantages of high degree of dissociation of the monomer, good quality, etc., the disadvantage is that the equipment is expensive and not easy to maintain. 2. Microwave roasting method can make a large number of Fe impurities enriched, and the vacuum roasting can effectively reduce the water content of quartz in the water content of the quartz. 3. After pretreatment of the surface layer of the quartz impurity is basically removed, the existence of the The impurities in the gas-liquid inclusions are externally visible to facilitate the subsequent purification process.

4. Magnetic Separation

After the crushing and scrubbing process, there is still a certain amount of iron and aluminum impurities in the quartz sand, in which the main source of iron impurities from the embedded in the quartz particles of iron-containing minerals and crushing and processing process of mechanical iron, and aluminum impurities are mainly from the quartz sand in the mica, feldspar [22]. Magnetic separation can effectively remove the ferromagnetic impurities in quartz by using the different magnetic permeability of the substance to screen the substance in the magnetic separation equipment.Li et al [23] used superconducting high-gradient magnetic separation technology to separate and extract quartz with SiO_2 content of 98.56±0.13% from the tailings of high-silica iron ore, and determined the optimal conditions for the magnetic separation process: slurry flow rate of 0.12m/s, solids concentration of 4%. Li et al [24] carried out magnetic separation of quartz ore pieces with a particle size of >200mm in Gansu, and experimentally determined that the ferromagnetic impurities in the quartz could be basically removed after three sections of 1.4 T magnetic separation, and it was not significant to continue to increase the number of magnetic separation sections. In addition, Yang et al. [25] increased the SiO_2 content from 93.35% to 99.92% by magnetic separation of quartz ore rocks in Anhui region under the conditions of 0.4T and 1.3T in turn, combined with the subsequent process. The magnetic impurities on the surface of quartz particles can be removed by multi-stage magnetic separation with different gradients, and superconducting high-gradient magnetic separation has the advantages of saving electric energy and equipment materials compared with ordinary magnetic separation.

5. Flotation Process

Flotation removes non-magnetic associated impurity minerals from quartz by selecting different trapping agents and inhibitors [26].Larsen et al [27] carried out flotation on quartz-feldspar mixtures, using a bubbler to selectively flotation the quartz from feldspars in dilute solutions of hydrofluoric acid (HF) to produce feldspar concentrates with yields of 99.9% and recoveries of 95.6%. Li [28] determined that the selection of sodium oleate and soluble starch as the collector and inhibitor, respectively, can realize the effective separation of quartz and magnetite by selective flotation separation. Lei et al [29] investigated the types and ratios of traps and determined that 80 g/t of propylenediamine was used in rough flotation, and sodium petroleum sulfonate and octyl hydroxamic acid in the ratio of 4:1 was used as a trapping agent in selection for the best effect of purification of vein quartz, and a sample with a total impurity element of 99.01 μg/g was obtained. In addition, Cao et al [30] explored the response of temperature to quartz flotation through microflotation experiments, and the results confirmed that the flotation effect of quartz was strengthened with the increase in temperature, and proposed an adsorbable model, and the results of quartz flotation at different temperatures are shown in Fig. 2. The selection and proportioning of the capture agent and inhibitor in the flotation process is extremely important, which greatly affects the flotation effect of quartz.

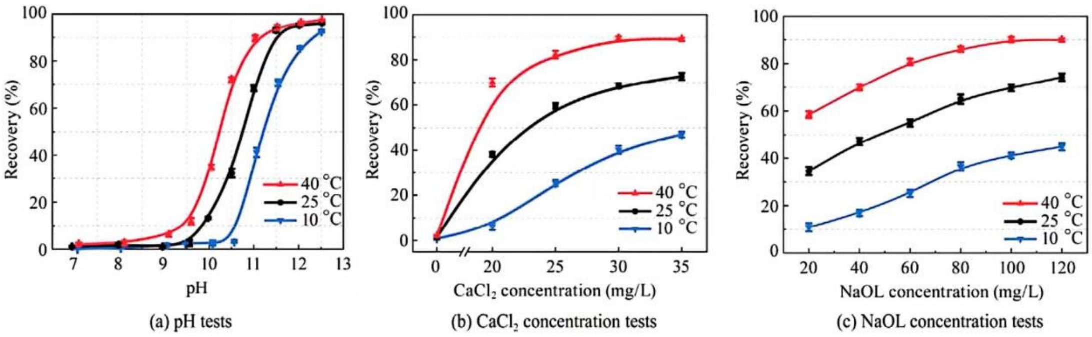


Fig. 2 Flotation results of quartz at different temperatures: pH tests with 30 mg/L $CaCl_2$ and 100 mg/L NaOL; $CaCl_2$ concentration tests with 100 mg/L NaOL at pH 11; and NaOL concentration tests with 30 mg/L CaCl2 at pH 11[30]

6. Acid Leaching

Because the magnetic properties, surface potential, dielectric properties of quartz and mica and feldspar are very similar, so the effect of quartz purification by magnetic separation, electric separation and flotation is not satisfactory enough, so it needs to be further purified, and at present the most widely used is the use of quartz, mica and feldspar in the differences in the solubility of acid in the acid leaching treatment [31].

6.1 Atmospheric Pressure Leaching.

In quartz acid leaching experiments, quartz is usually acid leached using different mixtures of acids at atmospheric pressure. Liu [32] used a mixture of acids with the volume ratio of

H_2SO_4:HCl:HNO_3:HF=10:5:3:1 to investigate the leaching efficiency at different temperatures under atmospheric pressure, and the study showed that the leaching time of 2h at 70°C was the best for the removal of impurities, and obtained a high purity quartz sand with a total content of 37.2 μg/g of impurities. Wang [33] used a solid-liquid mass ratio of 1:5, a mixture of hydrochloric acid, nitric acid and oxalic acid in a stirring reaction at 60 °C for 120 min on the acid leaching treatment of granite pegmatite from the Jiangxi area, and obtained a purified product with a total content of impurity elements of 89.79×10^{-6}. Zhong et al [34] used a quartz purification process by acid leaching and ethanol deionized water cleaning, and the resulting purity of silica in the refined silica sand was higher than 99.997%, and the total content of impurities was higher than 99.997%. The purity of the resulting refined silica sand was higher than 99.997%, and the concentrations of K and Na impurities were 1 ppm and 12 ppm, respectively.Atmospheric pressure acid leaching can effectively remove the oxide film on the surface of the quartz, and dilute acids such as oxalic acid have a better effect on the removal of Fe and Al. Strong acids are used for the removal of Cr and Ti, and in addition, the selection of the acid leaching temperature is extremely important.

6.2 Ultrasound-assisted Leaching.

Ultrasound-assisted leaching has an important role in quartz purification by accelerating the leaching reaction, enhancing the mass transfer process, improving the leaching selectivity, reducing the energy consumption, and facilitating the stripping of the impurity particles.Li et al [35] achieved 52.5%/53% of aluminum removal with very dilute acid mixture at 30°C/80°C in an ultrasonic unit at 17.4 ppm/17.7 ppm, respectively.This result is lower than the 4.4 ppm/4.7 ppm lower than the results provided by the world famous quartz sand suppliers.Yang et al [36] investigated the kinetic effects of ultrasonic power, temperature and other conditions on the removal of iron from quartz, and found that the activation energy of the reaction in the ultrasound-assisted leaching process was higher than that in the conventional leaching, which had the advantages of shorter reaction time and higher iron removal rate. In addition, Li et al [37] recovered high-purity SiO_2 by acid leaching of quartz from iron ore tailings using ultrasound-assisted fluorine-free acid solution, and the purity of SiO_2 reached 99.93% after three-stage leaching, and the removal rate of Mg, Al, and Fe reached more than 97%.

Summarize the above literature can be obtained the following conclusions: 1. at atmospheric pressure by changing the ratio of strong acid weak acid combination of mixed acid, as well as temperature, time and other conditions for acid leaching experiments can achieve better purification results, the ideal leaching temperature and time is generally in the 60-70°C, 2h. 2. ultrasound-assisted leaching can effectively increase the efficiency of the acid leaching, the activation energy of the acid leaching process reaction is higher than that of the atmospheric pressure leaching. 3. acid leaching of the quartz sand The purity of quartz sand after acid leaching can meet the demand of most industrial sand, but there is still a long way to go for the requirement of cutting-edge field.

7. Chlorinated Roasting

As the impurities in quartz are divided into lattice impurities and non-lattice impurities, the lattice impurities are mainly by his class of atoms in a qualitatively isomorphic way instead of silicon atoms in the silica-oxygen tetrahedron, and the main impurity elements are Al^{3+}, Ti^{4+}, Ge^{4+} and so on [6], the effect of removing the impurities in the quartz lattice using the above purification methods is not very satisfactory, so it is necessary to use the chlorination baking methods can be achieved by controlling the chlorination temperature and chlorine oxygen ratio value. By controlling a certain chlorination temperature and chlorine-oxygen ratio, some metal oxides can be converted into chlorides, thus achieving the effect of removing impurities from the crystal lattice.

Pan [38] carried out NH_4Cl solid and Cl_2 gaseous chlorination roasting experiments, compared with the ordinary processing method impurity content decreased by 28.2 ug/g and 6.1 ug/g, but these two methods for the removal of alkali metal elements is not effective. Lin [39] compared the effect of leaching quartz samples under two different systems, NH_4Cl-HCl and NH_4Cl-H_2SO_4, after chlorine roasting at 900°C for 5 h. The SiO_2 contents of the samples obtained were 99.991wt% and 99.990wt%, respectively. Lou et al [40] performed high temperature chlorination roasting of three quartz samples through comparative experiments, and finally determined the optimal experimental conditions as 300g HCl, roasting temperature 1000°C, 2h, and concluded that the removal of Al impurities by this method was ineffective. In addition, Zhang [41] used the same process for five samples through the Cl_2 roasting treatment for 1h, the final five samples of quartz purity have reached more than 4N8 standard, one of them even reached the 5N level standard. Chlorination roasting plays an important role in removing impurities in the quartz lattice and is an extremely critical step in the process of preparing ultra-high purity quartz sand, but it is easy to produce chloride gas in the preparation process causing environmental pollution, and the removal of Al elements or alkali metal elements is less than ideal.

8. Conclusion

At present, high-purity quartz has become a national strategic resources, in the new energy photovoltaic, aerospace, semiconductor field has a wide range of applications, the domestic market demand is huge, the use of joint processes from non-quality quartz ore sources to produce high-purity quartz sand is the challenge that our country is facing. In the quartz sand purification process, the pretreatment part of the quartz ore to avoid the introduction of mechanical impurities in the process of crushing and obtain high-quality particle size quartz particles can be selected high-voltage pulsed discharge crushing, but high-voltage pulsed discharge equipment is expensive, high energy consumption, the research and development of inexpensive and crushing quality of the equipment is particularly important, the use of vacuum roasting or microwave roasting to effectively improve the exposure rate of impurities; Magnetic Separation The choice of magnetic field strength and number of segments in the process has a great impact on the removal of magnetic impurities, the use of superconducting high-gradient magnetic separation equipment for magnetic separation can effectively reduce the consumption of materials and high efficiency of magnetic separation; flotation is extremely important for the selection and proportion of the capture agent, inhibitor; acid leaching process using ultrasound-assisted acid leaching can increase the activation energy of the reaction to improve the efficiency of the acid leaching; chlorine baking although effective in removing some of the quartz lattice of metal oxides Although chlorine roasting can effectively remove some metal oxides in the quartz lattice impurities, combined with pretreatment-magnetic separation-flotation-acid leaching process can produce 5N grade ultrahigh-purity quartz, but currently only in the experimental stage, the removal of its alkali metal elements is the lack of good effect is the current constraints on the preparation of ultrahigh-purity quartz sand is extremely critical, how to remove alkali metals in the quartz lattice of the quartz elements is the future purification of quartz sand is one of the main directions of the quartz sand purification process.

Acknowledgements

This research was supported by the Open Projects Foundation (No. SKLD 2205) of State Key Laboratory of Optical Fiber and Cable Manufacture Technology (YOFC).

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