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## New pretreatment method for high-tension electrical separation of zircon from quartz

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Abstract: Electrostatic separation is one of the mineral processing methods based on mineral conductivity. This method has some significant problems such as being sensitive to humidity, high middling product, and impurity of non-conductive minerals. In this study, a new pretreatment method was proposed for the separation of zircon from quartz before electrostatic separation to solve these disadvantages. In this regard, two stages of pretreatment were applied which involved using collector of sodium dodecyl sulfate (SDS) for adjusting wettability of the zircon surface and spraying electrolyte aqueous solution to increase conductivity of the quartz surface. The effects of important parameters including pH, collector concentration, conditioning time, and concentration and type of electrolyte on the process efficiency were evaluated. The results showed that the optimal conditions of high-tension electrical separation of  $1 \times 10^{-4}$  mol/L, conditioning time of 4 min and NaCl as an electrolyte with concentration of 4.27 mol/L. Separation efficiency of 95.12% was achieved in optimum conditions. This pretreatment method can be successfully used before high-tension electrical separation to separate the conductive or non-conductive minerals with various compositions.

Key words: zircon; quartz; collector; electrolyte aqueous solution; conductivity; high-tension electrical separation; surface treatment

### **1** Introduction

Zirconium is not found in nature as a native metal due to its intrinsic instability. The concentrations of zirconium in the earth's crust and sea water are about 130 mg/L and 0.026  $\mu$ g/L, respectively [1]. It can be mostly found in mines in the form of baddeleyite (ZrO<sub>2</sub>) and in beach sand in the form of zircon (ZrSiO<sub>4</sub>). Zircon is the most important and principal commercial source of zirconium. Zircon is a useful mineral and is used in many industries in a wide range of applications. It is mainly used as an opacifier in ceramic industries, refractory industries, foundry casting, TV glass and geochronological studies [2,3].

Processing of the heavy mineral contains a series of pre-concentration steps. In order to separate heavy minerals (such as zircon, rutile, magnetite, and ilmenite) from light minerals (such as quartz and clay), gravity separation methods are used. Subsequently, magnetic separation is used to remove all magnetic minerals (such as magnetite) from zircon and rutile. Then, zircon-rutile feed is filtered and dried to separate it by electrostatic separation [4].

High-tension electrical separation is a process that uses electrostatic charges to separate conductive minerals from non-conductive minerals. This method was initially applied in the 19th century in the agriculture industry to separate chaffs from seeds. The application of this method was generalized in the 20th century with progress in high voltage technology. Presently, it has been efficiently used in mining, agriculture and waste treatment [5].

Mineral conductivity is mostly influenced by feed humidity [6]. The separation efficiency is low in humid circumstances in high-tension electrical separation due to the formation of a conductive water layer on the surface of the minerals [7–9]. Furthermore, in some cases, low separation efficiency in high-tension electrical separation is due to misreporting of mineral properties, for instance, zircon with titanium coating can be considered as a non-conductive mineral during the process [10]. Also,

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there are always considerable middling products in high-tension electrical separation even under optimal conditions. Some factors such as feeding rate, distribution of space charge, non-uniform electric field and variant ambient conditions lead to an increase of meddling products [11]. Another problem in high-tension electrical separation is that it is very sensitive to the variation of particle size, feed grade and temperature [11]. VENTER and VERMAAK [12] evaluated the effect of acid treatment on the separation of zircon from rutile by high-tension electrical separation. They found that the surface conductivity of zircon was not sensitive to acid treatment at pH<8, while the surface conductivity of rutile showed sensitivity to acid treatment at a pH of 3-10. These results showed that selective separation can be achieved at lower pH with pretreatment. RAVISHANKAR and KOLLA [10] investigated the effect of chemical reagent adsorption on the rutile and zircon conductivity. They showed that electrostatic efficiency was improved by using Aero® EZ-2100 and Aero® EZ-1000 as a chemical reagent in the separation of rutile from zircon. In another study, RAVISHANKAR et al [13] investigated the influence of sea water on the high-tension electrical separation and effect of Aero® EZ-2100 reagent as a chemical enhancement on the selective recovery of rutile from zircon and removing impurity from the surface of the mineral. The results showed that sea water had an adverse effect on high-tension electrical separation, and the use of Aero® EZ-2100 reagent improved the recovery. KUMAR et al [14] studied the effect of temperature, roll speed and feed rate on the separation of conductive minerals in the beach sand sample. They showed that temperature has the greatest effect on the recovery of conductive minerals, while the feed roll is the most effective parameter on product grade; however, the roll speed has adverse effects on separation efficiency beyond its optimum value.

In this study, two stages of pretreatment were applied for purification of zircon before high-tension electrical separation. This pretreatment involved using collector for adjusting the surface wettability of zircon and spraying electrolyte aqueous solution to increase the surface conductivity of quartz. Separation efficiency was calculated to investigate the effects of pH, collector concentration, conditioning time, and concentration and type of electrolyte aqueous solution.

#### 2 Experimental

The experiments were performed on Oman's beach sand obtained from Chabahar beach, Sistan and Baluchestan Province, Iran. The sample was screened and a fraction of  $300-425 \ \mu m$  was used for the experiment. Sodium dodecyl sulfate (SDS) was used as an anionic collector (Merck), calcium dichloride (CaCl<sub>2</sub>) and sodium chloride (NaCl) as electrolytes and sodium hydroxide (NaOH) and hydrogen chloride (HCl) for adjusting the pH of the pulp. The Erize laboratory high-tension roll ionized field separator (England) was used for separation of zircon from quartz. In all experiments, the roll speed, voltage and feed rate were 260 r/min, 23 kV and 0.14 kg/s, respectively. X-ray fluorescence (XRF) analyzer and X-ray diffractometer (XRD) were used to determine the chemical composition and mineral phases in the sample. The results of XRD and XRF are presented in Fig. 1 and Table 1, respectively. The results show that quartz, zircon, and calcite are the main minerals in the test sample.



Fig. 1 XRD pattern of Oman's beach sand

 Table 1 Chemical composition of Oman's beach sand (wt.%)

CaO	TiO <sub>2</sub>	SiO <sub>2</sub>	$Al_2O_3$	$Fe_2O_3$	ZrO <sub>2</sub>	Others
4.19	0.8	68.06	2.42	1.27	19.50	3.76

#### 2.1 Pretreatment method

In order to modify the surface of the minerals, a novel method of pretreatment was used before applying the high-tension electrical separation. Figure 2 shows a schematic representation of this pretreatment method. Both minerals A and B in Fig. 2 are non-conductive. In the first stage, the sample containing minerals A and B is treated with the collector solution with a specific concentration. Collector ions can be selectively adsorbed on the desired mineral surface (mineral A) by adjusting the pH of pulp and zeta potential of the minerals. After drying the sample, in the second stage, the conductive film can be formed on mineral B by spraying the electrolyte aqueous solution on the sample. The conductive film cannot be created on the surface of mineral A due to its hydrophobic film. Therefore, mineral B behaves similar to the conductive mineral, while mineral A remains non-conductive. By using this



Fig. 2 Schematic diagram of novel pretreatment method

pretreatment, effective separation of high-tension electrical separators can be achieved even for the sample with humidity. Also, high-tension electrical separation can be used for effective separation of minerals, which have the same behavior in the electrical field (conductive or non-conductive) and are difficult to separate by other separation methods.

#### 2.2 High-tension electrical separation

In order to ensure a good representative feed sample, a great amount of the test samples passed through a riffle. Then, two stages of modification were carried out to increase the surface conductivity of quartz. Figure 3 shows a simplified flow diagram of processing. In each experiment, 1 kg of the sample was put into 1 L of water and the pH was adjusted. Subsequently, collector solution with a specific concentration was added to the pulp in order to adjust the surface wettability of zircon. After conditioning with collector solution for various time, the pulp was filtered and dried by using a vacuum desiccator. Then, the electrolyte aqueous solution was sprayed on the dried sample in order to form the conductive film on the surface of quartz. The conductive film did not form on the surface



Fig. 3 Simplified flow diagram of processing sample

of zircon due to its hydrophobic film. After spraying electrolyte aqueous solution, the sample was used for high-tension electrical separation. In this study, the separation of zircon from quartz using the electrostatic method was evaluated based on separation efficiency. In this regard, the effects of important factors such as collector concentration, pH, conditioning time, electrolyte concentration, and type of electrolyte were evaluated by considering the separation efficiency.

Separation efficiency  $(\eta_{\rm SE})$  was calculated as follows:

$$\eta_{\rm SE} = \frac{m(c-f)(f-t)}{f(m-f)(c-t)} \times 100\%$$
(1)

where *m* is theoretical  $ZrO_2$  content of zircon, *f* is  $ZrO_2$  grade of zircon in the feed ore sample, *c* is  $ZrO_2$  grade of zircon in concentrate and *t* is  $ZrO_2$  grade in tailing.

#### **3 Results and discussion**

#### 3.1 Effect of pH

To determine the effect of pH on the separation efficiency, high-tension electrical separation experiments were carried out as a function of pH at fixed SDS concentration, conditioning time and electrolyte concentration. The results are shown in Fig. 4. The separation efficiencies of 74.15%, 80.09%, 88.01%, 75.08%, and 70.11% were obtained at pH of 2, 3, 4, 5 and 6, respectively. The maximum separation efficiency was obtained at pH of 4. The isoelectric point (IEP) and zeta potential are very effective factors for the collector adsorption on oxide minerals in the flotation process. Many researchers indicated that the adsorption of the collector on oxide minerals is physical adsorption which makes a high recovery during the process [15,16]. In study, the separation efficiency this decreases significantly when the pH of pulp is higher than 4. The IEP values of zircon and quartz are at pH of 4.5 and 2.7, respectively [17,18]. The IEP of zircon is higher than that

of quartz. The zeta potential of the minerals is positive when the pH of the pulp is lower than IEP, and with an increase in pH from the isoelectric point, surface charge becomes more negative. Therefore, when the pH of pulp is higher than the IEP of zircon (pH=4.5), the surface charge of zircon is negative, and the adsorption density of anionic collector ions on the surface of zircon is decreased, which leads to a decrease in separation efficiency. When the pH of pulp is lower than the IEP of quartz (pH=2.7), the zeta potential of quartz is positive, which leads to an increase in the adsorption density of collector ions on the surface of quartz. Therefore, when the pH of pulp is lower than 2.7, the collector ions are adsorbed on both zircon and quartz surfaces. Thus, selective adsorption of collector ions is decreased, which leads to a decrease in separation efficiency.



**Fig. 4** Separation efficiency versus pH (SDS concentration  $1 \times 10^{-4}$  mol/L, conditioning time 3 min and electrolyte concentration (NaCl) 1.71 mol/L)

#### 3.2 Effect of collector concentration

Separation efficiency at different SDS concentrations was studied at constant pH, conditioning time and electrolyte concentration. The results are shown in Fig. 5. The separation efficiencies of 80.53%, 87.04%, 85.22%, 68.49% and 31.12% are achieved at SDS concentrations of  $1 \times 10^{-5}$ ,  $1 \times 10^{-4}$ ,  $1 \times 10^{-3}$ ,  $1 \times 10^{-2}$  and  $1 \times 10^{-1}$  mol/L, respectively. The maximum separation efficiency is obtained at SDS concentration of  $1 \times 10^{-4}$ mol/L. At SDS concentration lower than  $1 \times 10^{-3}$ , a decrease in collector concentration leads to a decrease in probability interaction between the surfaces of zircon and collector ions. Therefore, the surface wettability of zircon is increased, which leads to a decrease in separation efficiency. The separation efficiency decreases at SDS concentrations higher than  $1 \times 10^{-3}$  mol/L. Critical micelle concentration (CMC) of SDS at 25 °C is  $8.2 \times 10^{-3}$  mol/L [19,20]. At SDS concentrations lower than the CMC, SDS molecules associate into hemimicelle on the surface and cause the zircon particles

to become hydrophobic. At SDS concentrations higher than the CMC, the particles regain their hydrophilicity due to the micelle formation on the surface, and therefore separation efficiency is decreased. The adsorption of SDS ions on the surface of zircon accrues through electrostatic interaction between the collector and mineral, because at pH of 4 (optimal pH), the zeta potential of zircon is positive, while the surface charge of quartz is negative.



**Fig. 5** Separation efficiency versus SDS concentration (pH 4, conditioning time 3 min and electrolyte concentration (NaCl) 1.71 mol/L)

#### 3.3 Effect of conditioning time

The effect of conditioning time on the separation efficiency was studied in a range of 3-6 min. The results are shown in Fig. 6. The separation efficiencies of 76.21%, 82.13%, 88.65%, 81.30%, 76.65%, 72.45% and 69.68% were obtained for the conditioning time of 3, 3.5, 4, 4.5, 5, 5.5 and 6 min, respectively. Separation efficiency is initially increased with an increase in conditioning time, and reaches its highest value in a conditioning time of 4 min. In shorter conditioning time, collector ions do not have sufficient time for adsorption on the zircon surface. Therefore, the adsorption density of collector ions on the surface of zircon is decreased, which leads to a decrease in separation efficiency. The decrease in separation efficiency at high conditioning time can be due to the dissolving of calcite and an increase in Ca2+ concentration, which leads to the activation of quartz. Figure 7 shows the distribution of elements of calcite at different pH values [21]. It is shown that Ca<sup>2+</sup> activity increases with a decrease in pH. At pH of 4 (test condition), quartz has negative surface charge. Therefore, the adsorption of Ca<sup>2+</sup> on the surface of quartz at pH of 4 leads to an increase of the adsorption density of the collector on the surface of the quartz. Thus, separation efficiency is decreased at a conditioning time higher than 4 min.



**Fig. 6** Separation efficiency versus conditioning time (SDS concentration  $1 \times 10^{-4}$  mol/L, pH 4 and electrolyte concentration (NaCl) 1.71 mol/L)



Fig. 7 Distribution of species for calcite [21] (a stands for activity)

#### 3.4 Effect of concentration and type of electrolyte

To evaluate the effect of concentration and type of electrolyte on the separation efficiency, high-tension electrical separation experiments were carried out as a function of electrolyte concentration and type of concentration at fixed pH, SDS concentration and conditioning time. The results are shown in Fig. 8. The separation efficiencies of 83.04%, 89.12%, 90.62%, 92.30% and 95.12% are obtained at concentrations of aqueous NaCl solution of 0.85, 1.71, 2.56, 3.41 and 4.27 mol/L, respectively. The separation efficiency increases with an increase in the concentration of NaCl solution. Figure 9 illustrates the relationship between conductivity and concentration of aqueous NaCl and CaCl<sub>2</sub> solutions at 25 °C. It shows that an increase in the concentration of NaCl solution increases the solution conductivity. The adsorption of electrolyte solution on the surface of quartz leads to an increase in the conductivity of quartz. Therefore, the increase in the conductivity of NaCl solution increases the surface

conductivity of quartz. The difference in conductivity between the surface of quartz and zircon leads to an increase in the separation efficiency. Electrolyte aqueous solution changes the surface tension of the water–air interface and influences the solid–water contact angle. Contact angle increases with an increase in the NaCl concentration on the hydrophilic surfaces [22–26]. Also, the stern layer becomes thinner with an increase in NaCl concentration, which affects the process efficiency [26].



Fig. 8 Separation efficiency versus electrolyte concentration (SDS concentration  $1 \times 10^{-4}$  mol/L, pH 4 and conditioning time 4 min)



Fig. 9 Conductivity versus concentrations of aqueous NaCl and CaCl<sub>2</sub> solutions

The results show that the separation efficiency is increased in the range of 0.45-2.25 mol/L of  $CaCl_2$ solution due to an increase in the conductivity of the solution. On the other hand, a decrease in conductivity of aqueous  $CaCl_2$  solution at a  $CaCl_2$  concentration higher than 2.25 mol/L (Fig. 8) leads to a decrease in the conductivity of the quartz surface and difference in conductivity between the surface of quartz and zircon. Therefore, a concentration of  $CaCl_2$  higher than 2.25 mol/L decreases the separation efficiency. The results show that the conductivity of aqueous solution decreases at  $CaCl_2$  concentrations higher than 2.25 mol/L. At high concentrations of  $CaCl_2$ , conductivity of aqueous solution depends on the viscosity change of the solution and hydration ion. Thus, the conductivity of aqueous solution decreases due to an increase in viscosity of solution at high concentrations of  $CaCl_2$ [26].

#### **4** Conclusions

(1) Optimal conditions of high-tension electrical separation of zircon and quartz were a pH of 4, collector concentration of  $1 \times 10^{-4}$  mol/L, conditioning time of 4 min and electrolyte (NaCl) concentration of 4.27 mol/L. A separation efficiency of 95.12% was obtained in optimum conditions.

(2) Using NaCl as electrolyte indicates higher separation efficiency than using CaCl<sub>2</sub> as electrolyte.

(3) High-tension electrical separation method can be used effectively for the separation of quartz and zircon using the appropriate pretreatment stage.

(4) Effective separation can be achieved even in humid circumstances in high-tension electrical separation. This pretreatment can be also used for conductive or non-conductive minerals before high-tension electrical separation to achieve high separation efficiency.

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# 高压电选法从石英中分离锆石的新预处理方法

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摘 要:静电选矿是一种基于矿物导电性的矿物加工方法。但该方法存在一些严重问题,如对湿度敏感,中矿产 率高和含有非导电矿物杂质。为克服这些缺点,本文作者提出一种新的预处理方法,用于在静电选矿前从石英中 分离锆石。预处理包括两个阶段:一是使用十二烷基硫酸钠(SDS)作捕集剂调节锆石表面的润湿性,二是喷洒电 解质水溶液以提高石英表面的导电性。考察 pH 值、捕集剂浓度、调节时间、电解质浓度和种类等重要参数对分 选效率的影响。研究表明,高压电选的最佳条件如下: pH 值为 4, SDS 浓度为 1×10<sup>-4</sup> mol/L,调节时间为 4 min, NaCl 为电解质,其浓度为 4.27 mol/L,最佳条件下的分选效率为 95.12%。该预处理方法可成功地应用于高压电 选前各种组成的导电性或非导电性矿物的分选。

关键词: 锆石; 石英; 捕集剂; 电解质水溶液; 导电性; 高压电选; 表面处理

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