



A novel “green” solvent to deeply purify quartz sand with high yields: A case study



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ABSTRACT

A novel “green” solvent for removing iron and aluminum impurities simultaneously from quartz sand was presented to replace the outdated industrial strong acids. The solvent is a “green” mixture comprised nontoxic, renewable acids and a very diluted hydrochloric acid. The new solvent can significantly reduce the pollution caused by residual acids and remain the high efficiency in removing impurities and is harmless to the target product as well. The final optimum removing rate was up to 79.1% and 42.3% respectively if its leached residue was washed, then leached again in a 4 g/l Na₂CO₃ solution under ultrasound for 25 min.

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Introduction

Direct discharge of chemical industrial wastewater and wastes of the chemical laboratory often leads to deterioration of the environment and soil acidification [1–4], which will consequently cause crops reduction [5,6], and ultimately endangers human's health [7]. Therefore, many environmental scientists have to spend a lot of time and money to remedy the contaminated objects [4,8]. At the same time, highly pure quartz is so important to acquire clean energy, make advanced materials and protect environment [9–12], it has to be widely produced and thereby to bring the pollution of acidic wastes as well as the possible second pollution of unexpected heavy metals. Therefore, market appeals for “green” technology urgently.

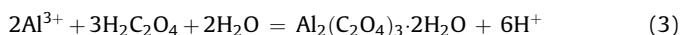
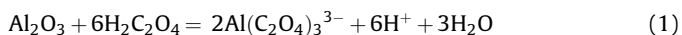
Conventionally, to remove impurity from quartz sand, the most effective way was the leach methods by a mixture of hydrofluoric acid (HF) with other different proportions of industrial acids such as hydrochloric acid (HCl), sulfuric acid (H₂SO₄) [13,14]. However, because the reaction between HF and target product SiO₂ will lead to a loss of the target product, so this method is currently declining. The other industrial strong acids [15] were also used to purify processes, however, since quartz sand is hard, the concentrations of these industrial strong acids have to be very high, the typical concentrations of the acids were ranged of 20–30% in many cases

[13,16]. Consequently, the high acidic concentration is not only more corrosive to leaching equipments, but also leads to the large risk of environment pollution due to the high residual acids and possible heavy metals, so the weaker acid was also tried to reduce the contamination as much as possible [17]. Recent years, weak organic acids, such as oxalic, citric, ascorbic, have been proposed to displace the industry strong acids due to technical and environmental reasons. Of these organic acids, oxalic acid (OA) [18] and ascorbic acid (AA) [19] are the most promising solvents because of their higher efficiency in leaching processes, milder acidity to environment, better complexation ability and higher reducing power to the impurity ions. The combination of these weaker acids is sometimes also used to raise leaching efficiency [20,21]. For example, Taxiarchou et al. [18] removed iron from silica sand by OA, and obtained 40% of iron removal at 90–100 °C, the sample size was 265 μm; Vegliò and his helpers [22] obtained 20–30% iron removal and less than 5% aluminum removal in the conditions of 3 g/l OA, 80 °C, and average size of the quartz sand was 71 μm. Though these percentages of iron removal were obtained at different sample sizes, temperatures and concentrations of oxalic acids, to take a whole, the efficiency of impurity removal was unsatisfactory. Instead of starting from industrial quartz sand purchased on market, Jin et al. [23] removed impurities from very beginning of quartz rock, they firstly roasted the mineral rock to 800–850 °C, then quenched into a loose state by cold water, after that, the cracked rocks were ground to 200 μm, and then the quartz sands were leached by H₂SO₄ at 250 °C for 40 min, they finally obtained 94% of iron removal and 95% of aluminum

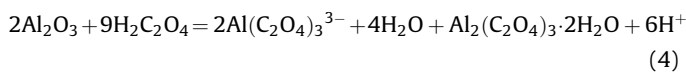
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removal. Though these results look good, the flow-sheet was too tediously long and the benefit of existing commercial quartz sand cannot be fully utilized. Additionally, the exclusive equipments have also to be re-desired for this innovative method. Ubaldini et al. [24] removed 46.1% of impure iron in 16 g/l OA solution, at 80 °C for 8 days with the mean size of 221 μm; again for raising leaching efficiency, ultrasound was also occasionally used in the leaching processes [21,24–26]. Du et al. [25] obtained 75.4% of iron removal by 4 g/l OA for 30 min, at 95 °C, the mean size of the samples was 71 μm intensified by both 500 rpm and 150 W ultrasound. In consideration of quartz sand being so hard to leach, these rates of iron removal were reasonably high compared with the over 95% leaching rate of non-ferrous metallic concentration (e.g. concentration of nickel oxide leached by dilute H₂SO₄), however, these investigations were exclusively designed for only iron removal, to remove the other impurities, investigators have to change the operating parameters and leaching circumstances, thereby the production cost will be increased remarkably and the flow-sheet will be definitely lengthy, this is a big shortcoming for these methods. Like OA, ascorbic acid (AA) is another leaching reagent for its stronger reductive ability, it is entirely nontoxic to the environment and harmless to the target product. However, differing from the characteristic of OA, when AA was used alone, its functions of removing iron and aluminum were negligible. However, we found that the ability of removing impurity will have a remarkable increase if it is added to a mixture of OA and much diluted HCl acid. Possible chemical reactions for OA removing aluminum (that is the more inefficient to be removed than iron) might be given as follows [19]:



Total reaction may be:



Besides these projects mentioned above, some other green and high efficiency processes [27,28] for various aims are also in progress.

Therefore, the aim of this project is to search for a novel and efficient environment-friendly solvent to extract impurities iron and aluminum simultaneously without hurting the target product and leaching equipments. For this aim, the first part of this work is mainly to search for a “green” solvent with efficiency as high as possible, and the second part is to raise the leaching yields as many as possible. Meanwhile, we explain the reasons behind our observations as possible as we can, however, because this is a case study, some complicated factors behind the observations have to be left for further study.

Materials and methods

Materials

The samples of white silica sand were purchased from Mingwang Quarry Co. Ltd.; it locates in Xinyi City of Jiangsu Province, China. The samples were dried before use.

The chemical compositions of the dry silica sand are reported in Table 1.

Table 1
Chemical composition of the dry silica sands.^a

Components	Amount (%)
SiO ₂	99.391
Al ₂ O ₃	0.249
Na ₂ O	0.081
K ₂ O	0.073
CaO	0.069
Fe ₂ O ₃	0.060
TiO ₂	0.014
Sum	99.937

^a The oxides of less than 0.014% are not shown.

The average particle size of the samples was determined as 71.5 μm by laser light scattering method (Mastersizer 2000, Malvern, UK). Other data of the original silica sands were measured by a standard method [29]. The chemicals are of guaranteed-grade from Sinopharm Chemical Reagent Co. Ltd. China, and deionized and distilled water was used throughout the work.

Methods

For the first part of this work, the mixed leaching solution was prepared by dissolving known quantities of acids in deionized and distilled water. For each run, 100 ml volumes of the prepared solution were poured into a 250 ml triangles frosted-mouth flask, which placed on a reinforcement motor stirrer (JJ-1, Jintan, China) with a controllable thermostat (DF-101S, Yuhua, China). Cooling water was circulated in a water bath to maintain the solution at a required temperature. While the required temperature was reached, 10.0 g of dry silica sand was added into the triangle frosted-mouth flask. The mouth of triangle frosted-mouth flask was covered to prevent evaporation of the solution. After reactions for appropriate time, the leached samples were filtrated, and then the filtrated residue was dried and weighted, finally the impurity content both in the residue and mother liquor was determined with ICP-OES (Vista-MPX, Varian, USA) for consistency.

Sonicator used in the second part of this work is JY88-II, made in Xinzhi, China, which has a probe with a tip radius of 6 mm and an adjustable power in the range of 0–500 W at frequency 20 kHz. The sonicator can be driven by either continuous or intermittent way. The sonicator was aligned its horn tip pointing to the vertical center of the container so that there was ultrasonic streaming sufficient to guarantee the solution being mixed perfectly. The content of the beaker was stirred at a pre-set speed and irradiated at a required power simultaneously (the experimental set-up can refer to Refs. [17] and [25]). Besides the sonicator irradiation and Na₂CO₃ leaching, the treatment and leach method for the samples was at the same as in the first part. All the experiments were conducted in duplicate and repeated again if values were too scattered, and the mean values have been reported. Analytical methods are accorded with a popular handbook on the property measurement of physical chemistry and composition analysis for non-metallic minerals [30].

Results and discussion

Effects of temperature on leaching rates of the impurities by HCl

Effects of temperature on the removing rates of iron and aluminum at different concentrations of HCl acid are given in Fig. 1. The percentages of iron removal rose remarkably as temperature increasing. For instance, the percentage of removal iron in 1.0% HCl acid was 16.03% at 35 °C, and rose to 36.62% at 80 °C, showing the leaching percentage was doubled as the temperature went up. The same trend was remained for aluminum

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