Identification of Quartz Sand From the Hills of Gunung Walat at Sukabumi Regency for Raw Materials of Nano Silica Precipitate

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Abstract. Gunung Walat Hills Formation, Sukabumi Regency is an area that has abundant natural quartz sand resource potential. Quartz sand from the Gunung Walat hills is partly in the form of Quartz Wacke which is composed of monochrystaline and polychristaline quartz minerals associated with clay minerals so it has high Al²O₃ and Fe²O₃ impurities. Until now the use of quartz sand from the hills of Gunung Walat has only been used as building material so that it has a low sale value. In this paper, we will examine the potential of quartz sand from Gunung Walat hills for the raw material of nano silica precipitates so that it can increase the added value of natural resources of quartz sand. In this paper the results of identification are described by taking samples from unprocessed quartz sand, the results of washing by mining companies and dissolving with sulfuric acid. From the results of the X-Ray Fluorescent analysis it is seen that quartz sand from mining has a silica composition of 85.869% wt, water leaching results by the mining company 93.702% and sulfuric acid leaching results obtained levels of 95.672% wt. The results of SEM-EDX analysis show that impurities Al²O₃ and Fe²O₃ are in the mineral sludge, not in one complex compound bond on the same grain so it is relatively easier to separate. From the identification results it can be seen that quartz sand from the Gunung Walat hills can be used as a raw material for making nano silica precipitates.

1. Introduction
One of the main producers of quartz sand from Sukabumi comes from the Gunung Walat hill formation in the western area of Sukabumi City. Its location adjacent to the highway and business center makes quartz sand from the hills of Mount Walat much mined. In general, the mining of quartz sand from the hills of Gunung Walat is used as a raw material for the building construction industry so that the added value of quartz sand is very low. Based on the results of a report from the Department of Mining and the Environment Sukabumi Regency in 2003 to 2014, it is recorded that the production of quartz sand reached 100,000 tons to 250,000 tons [1]. Quartz sand reserves in Sukabumi Regency reach 2,990 billion tons, is the third largest mineral reserves after mountain rocks and limestone [1].

The silica content of quartz sand from Sukabumi Regency is quite low below 90%, for example the silica sand content in Cibadak is around 89%, Ciemas is around 49.8% and Cisaat is around 93.33% [1]. Quartz sand that is in the mountains of walat is mostly of Quartz Sand Quartz Wacke which is composed of monochrystaline and polychristaline quartz minerals associated with clay minerals so it...
has high Al$_2$O$_3$ and Fe$_2$O$_3$ impurities [2]. Alumina impurity is derived from hallosite minerals and brusal iron oxide impurities from geotite minerals [2]. Quartz sand from the hills of Gunung Walat is composed of alternations between sandstone and clay stone in the form of brownish white solids, clastic texture, lamination and normal gradation [2]. Quartz sand that is in the mining area is in the form of lumps like rock but is easily crushed into coarse sand grains, this can be seen from the picture below [2]:

Figure 1. Anita Yulianti et al survey results of the Gunung Walat quartz sandstone outcrop both natural and artificial outcrops [2].

Walat Formation in the southern area of Sukabumi Regency is generally a distinction between sandstone, conglomerate and clay stone containing a small amount of coal [3]. The Walat Formation is composed of paleogene-aged or late Eocene Early Oligocene sedimentary rock, equivalent to the Bayah formation of Oligocene age [3].

Based on the above data, in order to increase the added value of quartz sand from Gunung Walat, a washing process is carried out to separate it from the impurities [2]. Removal of impurities such as iron in quartz sand can increase the value added by tens of times. This is due to an increase in the function of quartz sand for example quartz sand with Fe$_2$O$_3$ content of 0.2% for refractory and glass materials, Fe$_2$O$_3$ content of 0.02% for special glass materials, Fe$_2$O$_3$ content below 100 ppm for premium ceramics, Fe$_2$O$_3$ content below 10 ppm for optical glasses and Fe$_2$O$_3$ levels below 1 ppm for optical fibers [4].

To improve the quality of quartz sand through the removal of impurities, many efforts have been made, among others through the process of washing with water which is assisted with a magnetic separator, flotation and ultrasonic cleaner [5-7]. Washing process with water is the cheapest, simplest and most environmentally friendly, because it does not use chemicals. However the washing process with water cannot increase high purity, in general it is only able to eliminate alumina and potassium compound compounds [7]. Washing process with water though less effective but able to increase the level of quartz sand from 96% to 97.75% in quartz sand obtained from mining results [8]. The process of washing water with the help of ultrasonic waves is able to take heavy metal impurities by adding sodium carbonate solution [9]. The process of washing with water does not produce a higher level of purity so it needs to be followed by an acid leaching process, such as sulfuric acid and hydrochloric acid. This acid leaching process is usually for the removal of impurities from the element groups of aluminum, iron, calcium, magnesium and other elements [10]. The results of the acid washing process cannot be directly used because of the new bond between the impurities and acidic materials such as sulfur and chloride, therefore the acid washing results must be washed immediately with water bersih [10]. The acid leaching process has evolved using oxalic acid which is able to take iron impurities by forming iron oxalate bonds so that iron impurities dissolve easily [8, 11, 12, 13]. The effectiveness of the use of oxalic acid is far better than the use of sulfuric acid and hydrochloric acid, where with oxalic acid obtained quartz sand with a high level of purity. The results of the washing process of oxalic acid in quartz sand from Mojosari with silica content of 95% can be increased to 97.79% [13]. Then with the help of ultrasonic waves, the washing process with oxalic acid can increase silica levels from 97.75% to 99.37% [8].
2. Experimental
2.1. Raw Materials
The object of the study was quartz sand from the hills of Gunung Walat, Sukabumi Regency. In this activity, samples of quartz sand were taken from a quartz sand mining company that is under the hills of Gunung Walat. The samples come from two different parts, namely the samples in the reservoir that have not been washed with water and the samples that have been washed one time with river water and then rinsed again with clean water. The quartz sand mining company that we visited processes quartz sand mixed with clay with water to obtain clean quartz sand that is ready for sale for construction materials such as lightweight concrete and brick.

![Figure 2](image1.png)

**Figure 2.** The location of sampling is the quartz sand mining company, which is the quartz sand pile from the mining and the location of the quartz sand washing process.

2.2. Characterizations
Quartz sand samples taken from the quartz sand mining company are then carried out the characterization process and the washing process with sulfuric acid and washing again with water to remove the acid in the quartz sand. The quartz sand obtained was divided into three samples, namely samples from mining products, samples from the first washing and the results of the second washing. Then the results of the second washing were washed with sulfuric acid with a concentration of 10 N and 5 hours with a ratio of dissolving 50 g per 100 mL solution. After the four samples were obtained the characterization process was carried out using X-Ray-Fluorescent (XRF), X-Ray-Diffraction and SEM-EDX.

3. Result and Discussion
3.1. Analysis of Silica Content with X-Ray Fluorescent (XRF)
Silica content analysis of the quartz sand from Gunung Walat hills was carried out using the X-Ray Fluorescent method. The results of the analysis conducted at the LIPI Metallurgical and Material Research Center are as follows:
Table 1. Silica content analysis of the quartz sand from Gunung Walat hills

| Component | Natural Quartz Sand (% wt) | Water Dissolution Process I (% wt) | Water Dissolution Process II (% wt) | Sulfuric Acid Dissolution 10 N (% wt) |
|-----------|--------------------------|----------------------------------|-----------------------------------|--------------------------------------|
| SiO$_2$   | 85.869                   | 89.753                           | 93.702                            | 95.672                               |
| Al$_2$O$_3$ | 11.706                  | 7.769                            | 4.691                             | 2.877                                |
| K$_2$O    | 1.068                    | 0.625                            | 0.414                             | 0.369                                |
| Fe$_2$O$_3$ | 0.505                  | 1.098                            | 0.641                             | 0.166                                |
| MgO       | 0.468                    | 0.376                            | 0.273                             | 0.192                                |
| TiO$_2$   | 0.189                    | 0.138                            | 0.091                             | 0.252                                |
| LOI       | 0.049                    | 0.036                            | 0.091                             | 0.472                                |

From the XRF test results, it appears that quartz sand from the hills of Gunung Walat, Sukabumi Regency has a low quality. It is indicated by silica levels below 90% and there are many impurities. Low levels of quartz sand due to the large number of impurities makes the selling value of quartz sand from the hills of Gunung Walat very low. By business actors, the quartz sand produced by mining is washed with water from the river in the first washing step and then with clean water in the second washing step. From the results of washing with water, it appears that an increase in silica levels is quite high from 85.869% by weight to 93.702% by weight in the second stage of washing. The impurity element that has decreased during the water washing process is Al$_2$O$_3$, while the Fe$_2$O$_3$ content has not decreased. This shows that most of the alumina is in the clay mineral group that is easily suspended in water. While Fe$_2$O$_3$ is a goethite mineral with has the same density as silica. In the dissolution process with 10 N of sulfuric acid, silica levels increases to 95.672%, a decrease in alumina content of 50% to 2.877% wt and a decrease in Fe$_2$O$_3$ levels by 80% to 0.166%. After washing with sulfuric acid, it is seen that quartz sand from the hills of Gunung Walat is suitable as a silica precipitate with a slightly reduced Al$_2$O$_3$ content to below 2% wt and Fe$_2$O$_3$ levels below 0.1% wt [14]. Therefore it is necessary to wash using other types of acids such as hydrochloric acid and oxalic acid or by using magnetic separator, flotation and ultrasonic waves.

3.2. Analysis result with XRD
From the results of tests with X-Ray Diffraction on quartz sand from the hills of Mount Walat shows that the peak formed is only the peak of the Quartz compound (SiO$_2$) in all the peaks obtained and other peaks do not exist. From the results of this analysis showed that there is no complex compound between SiO$_2$ and Al$_2$O$_3$ even though Al$_2$O$_3$ content are quite high at around 11% wt. This gives an indication that the impurities in quartz sand can be destroyed by physical processes such as washing, dissolving, flotation, magnetization and ultrasonic processes.
3.3. Analysis result with SEM-EDX

SEM analysis was performed to see the appearance of the granules and the location of the main elements of silica and the concentration of impurities present in the granules. Then the mapping analysis of the elements is taken on three grains that show different appearance. Images of grain appearance can be seen in the image below.

By looking at the shape of the grains, it is seen that quartz sand grains are a mixture of various types of rock even though the grains are dominated by quartz. In SEM images, there are three different forms of buitran, after an EDX analysis it is seen that each granule has a different elemental content. Granule (1) is dominated by zircon elements with 38% content, granule (2) is dominated by silica with 26% content and granule (3) is dominated by iron with 30% content. By looking at this result, it show that impurities do not bind fully with silica so that it is more easily separated physically.
Figure 5. The results of EDX analysis of natural quartz sand granules

The results of SEM analysis of quartz sand that has been washed with water twice shows the same thing in granules as before washing process. Results of grain appearance can be seen in the picture below:

Figure 6. The results of the analysis of the appearance of grains on quartz sand that has been washed 2 times with water.

In grain (1) shows a mixture of silica and alumina with levels of 21.9% and 10.8%. Then in grain (2) shows a mixture of 30% iron and 18% silica. In item (3) it shows that silica is 34% and Carbon is about 6%. From these results it appears that impurities are mostly on smaller grains and silica is on grains with relatively larger sizes.

Figure 7. EDX analysis results per grain from the SEM image of quartz sand from the water washing process twice.

4. Conclusion
The results of the study indicate that quartz sand from the area has the potential as a raw material for the manufacture of nano silica precipitates. With a number of treatments, namely washing water twice and followed by washing using sulfuric acid, quartz sand has not been obtained that meets the requirements for raw materials of nano silica precipitate. However, quartz sand from the hills of Mount Walat has the potential to produce better quartz sand because there is no bond between impurities and silica that forms complex bonds. The requirement for raw material of nano silica precipitate is to be able to be processed into sodium silicate which has requirement of Al₂O₃ content.
under 1.5% and Fe$_2$O$_3$ content below 0.1%. Therefore it is necessary to do a better experiment using oxalic acid and using the help of physical processes such as flotation, magnetization and ultrasonic..

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