# Nanosilica Extraction from Grumusol Soil

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***Abstract***
*Grumusol soil is a type of soil that contains significant amounts of silica. Silica content in grumusol soil can reach up to 90%. The aim of this research was to determine the characteristics of silica nanostructures in Grumusol soil. The stages of the research were carried out including the preparation of raw materials, silica extraction, and XRD analysis. The raw material preparation stage is carried out by cleaning the grumusol soil from dirt. The second stage was carried out by synthesizing silica with the base extraction method and followed by acid deposition. The final stage is X-Ray Diffracion (XRD) analysis. The results of the research that has been carried out is the FWHM value of 0.0900 (<0.1) which indicates that the crystals produced have very good crystals with a very regular structure. The crystal size which includes nanosilica is 39.5076-48.6633 nm for 3M NaOH with 1 and 3 hours extraction time and 5M NaOH with 1 hour extraction time.*

***Keywords*: *Extraction, Grumusol Soil, XRD.*

***Abstrak***
*Tanah grumusol merupakan salah satu jenis tanah yang mengandung silika dalam jumlah yang signifikan. Kandungan silika dalam tanah grumusol bisa mencapai 90%. Penelitian ini bertujuan untuk mengetahui karakteristik struktur nano silika pada tanah Grumusol. Tahapan penelitian yang dilakukan meliputi persiapan bahan baku, ekstraksi silika, dan analisis XRD. Tahap persiapan bahan baku dilakukan dengan membersihkan tanah grumusol dari kotoran. Tahap kedua dilakukan dengan mensintesis silika dengan metode ekstraksi basa dan dilanjutkan dengan pengendapan asam. Tahap terakhir adalah analisis X-Ray Diffracion (XRD). Hasil penelitian yang telah dilakukan adalah nilai FWHM sebesar 0,0900 (<0,1) yang menunjukkan bahwa kristal yang dihasilkan memiliki kristal yang sangat baik dengan struktur yang sangat teratur. Ukuran kristal yang termasuk nanosilika adalah 39,5076-48,6633 nm untuk 3M NaOH dengan waktu*

*ekstraksi 1 dan 3 jam dan 5M NaOH dengan waktu ekstraksi 1 jam.*

**Kata Kunci:** *Ekstraksi, Tanah Grumusol, XRD.*

## 1. Introduction

Silica, also known as silicon dioxide (SiO2), is a mineral compound that has wide applications in various industrial sectors. Silica is used in glass, ceramics (Anon, 1997), concrete (Paramitha et.al, 2019), electronics (Brinker and Scherer, 1990), cosmetics, pharmaceuticals, and many other industries. One of the interesting potential raw materials for silica production is grumusol soil.

Grumusol soil is a type of soil that contains significant amounts of silica. Silica content in grumusol soil can reach up to 90 percent in the form of amorphous crystals. This makes Grumusol soil a potential source rich in silica.

Utilization of Grumusol soil as a raw material for silica offers several significant advantages. First, its abundant availability. Grumusols can be found in many regions of the world, including Indonesia. This wide availability allows the use of grumusol soil as a relatively easily accessible source of silica.

In addition, the process of extracting silica from grumusol tends to be easier compared to other silica sources, such as quartz sand. Grumusol soil has a softer texture, making it easier to crush and separate silica. Its abundant existence also allows for the potential for using Grumusol soil on a larger industrial scale.

Utilization of grumusol soil as a raw material for silica also has environmental benefits. In the context of quartz sand mining, which is the main source of silica, the use of grumusol soil can reduce the negative impact on natural ecosystems. Quartz sand mining often causes significant environmental damage, while grumusol soil can be a more sustainable alternative.

However, although the potential for utilizing grumusol soil as a raw material for silica is quite promising, further research and development is needed to optimize the process of extracting and purifying silica from grumusol soil. Studies on the characteristics, quality and applications of the resulting silica also need to be carried out to ensure its suitability and sustainability in different industries.

Several previous studies have been carried out regarding silica, namely Ramadhan, et.al (2014) conducted research on the synthesis of SiO2 made from Bancar Tuban sand with the results of research on the 7M NaOH variable, the SiO2 formed is amorphous SiO2. Alimin (2016) researched that the sand at Losari Beach has a silica content of 63.76% and there is a mineral content of 20.7% cristobalite. Trianasari, et. al (2017) conducted research on the characterization of the silica content in pumice. the results of the research that has been carried out produce nanostructures of silica pumice measuring 5.790 ± 0.23nm. Fitri, et.al (2021) conducted research on making silica from vertisol soil which has a silica content of up to 77.7%. Hanawindy, et. al (2023) conducted research on the extraction of silica from napa soil minerals on the south coast with the results of his research stating that the yield of silica extraction increased with the increase in NaOH concentration.

Several studies that have been conducted have discussed the silica content produced by several types of soil/rock, but not much research has discussed the characteristics of silica nanostructures. Therefore, the aim of this research was to determine the characteristics of silica nanostructures in Grumusol soil.

## 2. Method

### 2.1 Materials and Tools

The research was conducted using Grumusol soil taken from Bringinbendo Village, Sidoarjo. The tools used in this study were glass, porcelain crucible, magnetic stirrer, analytical balance, vacuum pump, oven, mortar, sieve, electric stove, measuring cup, beaker glass, filter paper, volumetric flask, Erlenmeyer, stir bar, watch glass. While the supporting materials used in this study were HCl, NaOH, Whatman filter paper, and distilled water. The research variables used were NaOH concentration (3, 5, and 7M) and extraction time (1 and 3 hours).

### 2.2 Research Procedures

The stages of the research carried out included raw material preparation, silica extraction, and XRD analysis. The raw material preparation stage is carried out by cleaning the Grumusol soil from impurities by sieving. The second stage was carried out by synthesizing silica with the alkaline extraction method and followed by acid precipitation. The last stage is the analysis of X-Ray Diffracion (XRD).

### 2.3 Analysis

XRD is one of the analytical methods used to identify the crystal structure of a material. This technique uses X-rays to reflect these rays on the material being analyzed and produce diffraction patterns that can be used to identify the crystal structure of the material. In this research, XRD analysis was carried out to determine the size of the silica crystals resulting from the research results. XRD analysis was carried out at the Materials Laboratory of the Surabaya Sepuluh Nopember Institute of Technology using PANalytical.

## 3. Result and Discussion

The research results were carried out by conducting XRD tests on all research variables, namely NaOH concentration (3, 5, and 7M) and extraction time (1 and 3 hours). The XRD test is carried out at a short angle (5-60$^o$). The results obtained from the test can be seen in Table 1.

Table 1. XRD Test Result on Samples

| Sample | Pos. [°2Th] | FWHM Left [°2Th] |
|---|---|---|
| 3M 1 hour | 27.4732 | 0.0900 |
| 3M 3 hour | 27.6297 | 0.0900 |
| 5M 1 hour | 27.6301 | 0.0900 |
| 5M 3 hour | 27.7014 | 0.0900 |
| 7M 1 hour | 27.7023 | 0.0900 |
| 7M 3 hour | 27.7227 | 0.0900 |

Full Width at Half Maximum (FWHM) is a parameter used to calculate the distance between 2 points which has a value of half the maximum curve value (the width of the hill). Some of the common FWHM values used are FWHM < 0.1 indicating very good crystals with a very regular structure. FWHM between 0.1–0.5 indicates a crystal with good structure, but has little deformation or the presence of an amorphous phase. Meanwhile, FWHM > 0.5 indicates the presence of deformation or the presence of an amorphous phase in the crystal.

The data obtained from the XRD test results show that the FWHM value is <0.1 which indicates that the crystals produced have very good crystals with a very regular structure. This is in accordance with research conducted by Suryanarayana and Norton (1998) which states that the smaller the FWHM value, the better the crystal quality. In addition, the smaller the FWHM value, the easier it is to adjust the direction and bond length of adjacent atoms (Wahyuningsih, et.al, 2013).

Furthermore, the calculation of crystal size is carried out with reference to the main peaks of the difactogram using the Debye Scherrer Equation approach which is formulated as follows:

$$D = \frac{K.\lambda}{\beta \, cos\theta} \quad (1)$$

where:
D = Crystal size (nm)
K = Form factor of the crystal (0,9)
λ = Wavelength of X-rays (1,54056 Å)
β = Value of FWHM
Ɵ = Difraction angle

The results of calculating the size of silica using the Debye Scherrer Equation can be seen in Table 2.

Table 2. Silica Size Calculation Results

| Sample | Pos. [˚2Th] | D (nm) |
|---|---|---|
| 3M 1 hour | 27.4732 | 39.5076 |
| 3M 3 hour | 27.6297 | 48.6342 |
| 5M 1 hour | 27.6301 | 48.6633 |
| 5M 3 hour | 27.7014 | 54.5206 |
| 7M 1 hour | 27.7023 | 54.6040 |
| 7M 3 hour | 27.7227 | 56.5690 |

Based on the results of the research in Table 2, it can be seen that the greater the concentration of NaOH used, the larger the resulting crystal size. This is in accordance with research conducted by Zuwana, et.al (2020) which states that higher concentrations of NaOH produce higher yields of silica particles.

Research conducted by Hayati and Astuti (2015) states that the size of silica with the coprecipitation method ranges from 25-60 nm and research by Ardiansyah and Wahyuni (2015) in the size range of 13.36-50 nm using the sol-gel method. The size of the silica crystals produced in this study ranged from 39.5076-56.5690 nm. So from the results of this study it can be concluded that the sizes of silica crystals included in the nanosilika range using the sol-gel method are 3M NaOH with extraction times of 1 and 3 hours and 5M NaOH with 1 hour extraction time in the range 39.5076-48.6633 nm.

## 4. Conclusion

The conclusions from the results of the research that has been done are:

1. FWHM value is 0.0900 (<0.1) which indicates that the crystals produced have very good crystals with a very regular structure
2. The crystal size which includes nanosilika is variable 3M NaOH with extraction times of 1 and 3 hours and 5M NaOH with 1 hour extraction time in the range 39.5076-48.6633 nm.

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