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Double Acid-Base Extraction of Silicic Acid from Quartz Sand

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Abstract

Silica is becoming more attractive as plant nutrient for non-graminae crops particularly in relation with drought-stress tolerant. Many efforts have been conducted to obtain an efficient technique to produce silica fertilizer worldwide, but the results are varying considerably due to various factors including raw material and extraction technique. This study was carried out to develop an efficient extraction technique for ortho-silicic acid (OSA-H_4SiO_4) from a Bangka-Belitung quartz sand by employing acid-base dissolution method. A 325-mesh size quartz sand was boiled in HCl solution at various concentrations. The optimum concentration was then used in the following experiment at several different volumes of solution. The sand obtained from optimum concentration and volume of HCl solution was then reacted with different amounts of NaOH (s), and heated until a wet mixture was obtained. As a reference the best extraction conditions were applied to a natural zeolite sample. All OSA analyses were done in triplicates with spectrophotometric method. Supporting evidences were collected from x-ray diffraction and scanning-electron-microscopy analyses of the treated samples. The yield of quartz sand-originated OSA was 183 $g \cdot kg^{-1}$ and significantly increased linearly with increasing weight of NaOH ($R^2 = 0.99^{**}$), whereas that from zeolite was only 104.2 $g \cdot kg^{-1}$ at 80 g NaOH. XRD and SEM data confirmed the evidences that the acid-base extraction disrupted the quartz mineral structure and as a consequence releasing more water soluble OSA.

Keywords

Ortho-Silicic Acid, Bangka-Belitung Quartz Sand, Acid-Base Dissolution, Pre-Washed

1. Introduction

Silicon is abundant in soil, but most of its sources are not available for plant uptake due to low solubility of Si compounds in soil [1] [2]. Although it is not accepted as one of essential plant nutrients, many evidences have shown strategic function of silica (Si) in crop cultivation *i.e.* stimulate nutrient uptake and plant photosynthesis, decrease susceptibility to disease and insect damage, alleviate water and various mineral stresses and decrease the toxic effects of aluminum [3] [4] [5]. Reference [6] stated that plants absorb Si as much as some other macro essential nutrients. Some evidences indicate that Si application to soil can improve phosphate (P) uptake by plants as Si will compete to get aluminum (Al) and/or iron (Fe) and releasing P as a result. These are some reasons why the use of Si will become highly potential to be a fundamental element in supporting sustainable agriculture, biological/organic production, and environment protection [7]. Silicon in the soil solution usually in different forms and occurs primarily as monomeric *i.e.* ortho-silicic acid (OSA). This monomer is up taken by plant [8] and mostly resides in the cell walls. This will then, to some extent, be considered to be responsible in controlling stomatal activities especially when the plant face drought stress [9] [10]. Reference [11] observed that Si addition to soils will not only improve chlorophyll content but also chlorophyll a/b ratio. There was a linier relationship between chlorophyll a/b ratio and photosynthesis impedance.

Si is the second abundant element (27.7% w/w) after oxygen (47% w/w) in soils [12]. Si compounds mainly present as SiO_2, about 50% - 70% of the soil mass, and in various aluminosilicate forms [13]. However, most of the Si in soils is found in insoluble forms. It is well known that Si absorption could be insoluble form *i.e.* OSA (H_4SiO_4). Its solubility was reported depending on particle size of the bearing mineral [14]. In soil, silicon is generally grouped into three different fractions such as the solid phase, the liquid phase and the adsorbed phase [15]. The crystalline form consisted only of the primary and the secondary crystalline silicates, which are abundant in mineral soils that developed from rocks and sediments [16]. The silica materials consist primarily of quartz and disordered silica. The amorphous and poorly crystalline and microcrystalline forms are also components of the silicon fractions in the solid phase [17]. The components of silicon in the liquid and the adsorbed phases are similar, with exception that those in liquid phase are dissolved in the soil solution, whereas those that are adsorbed are held onto soil particles and the Fe and Al oxides/hydroxides.

In soil, Si compounds exist in two forms viz. liquid and solid. The solid form mainly consists of crystalline, poorly crystalline, and amorphous silica (ASi) [15]. The liquid form generally consists of mono- and polysilicic acids, complexed with organic and inorganic compounds, etc. [18]. Wollastonite is a naturally occurring mined $CaSiO_3$ and can be a useful Si source when finely ground. However, this mineral is not found in Indonesia. Bangka-Belitung province is the largest deposit and has best quality of quartz in Indonesia, therefore can be used as locally sources for silicate fertilizer. Quartz is the second most abundant mineral in Earth's continental crust, after feldspar. Quartz is a compound of one-part silicon and two parts of oxygen, silicon dioxide, SiO_2. The specific objective of this study was to justify the assumption that a locally–abundant quartz sand is suitable as an efficient raw material to produce Si fertilizer by using a double HCl-NaOH extraction technique in Indonesia.

2. Material and Method

2.1. Silica Sources

A 325-mesh quartz sand commercial sample was originated from Bangka-Belitung province, Indonesia (**Figure 1**). The area has highly unique geological landscape as it was derived from metamorphic rocks with granitic intrusion including tin-consisting granite and granodiorite [19]. Other geological formations are also found consisting of metamorphic alluvial sandstone, mud deposit with sandstone lenses, and quartzite [20]. Biotite Granites rock found are rich in silica (average 717.8 $g \cdot kg^{-1}$ SiO_2), coarse grained, commonly porphyrite and sometimes foliated. Hornblende-Biotite Granodiorites are, medium to coarse grained, sometimes porphyritic and usually non-foliated. The Middle Triassic Tanjung Pandan batholith on Belitung Island is associated with major alluvial tin deposits and with minor primary tin mineralization of greisen type (Tikus Mine). Sub economic quartz-tourmaline-cassiterite veinlets and stock works are locally abundant. The batholith consists of two petrogenetically different, ilmenite-series rock suites with about the same age of 215 ± 3 Ma [21], and a really dominating biotite granite suite and a quartz syenite suite of more restricted extent. The granite suite is composed of three subunits which are, from oldest to youngest: K-feldspar mega crystic medium- to coarse-grained biotite granite (main phase); mega crystic biotite microgranite with medium- to coarse grained porphyroclasts of plagioclase, K-feldspar, quartz and biotite (first sub intrusion); non-mega crystic biotite microgranite (second sub intrusion). The quartz syenite suite covers a large compositional spectrum from gabbroic cumulate rocks to hornblende-biotite quartz syenite (main phase) to alkali feldspar-hornblende granite pegmatite.

Figure 1. The geographical position of Bangka-Belitung Island, Indonesia.

2.2. Silica Extractions

A 150 g of 325-mesh quartz sand sample was boiled in 100 mL HCl at various concentrations, *i.e.* 1, 3, 5, 7, and 9 N until almost all of solution evaporated to dissolve any contaminant elements present. The treated samples were then washed out with tap water several times to eliminate the contaminants and the rest of HCl solution. Wet samples were transferred on a sheet of paper and dried out at 100˚C in an oven until completely dry. A 60 g washed sample was then mixed with 80 g NaOH (s) in a stainless pan and heated on stove at 330˚C while stirred manually until melted. The melted mixture was kept stirred until it dried out. After cooling at room temperature, a 60 g pre-treated quartz sand was dissolved in 400 mL distilled water. The liquid obtained was the soluble silica (H_4SiO_4). Silica concentration was determined by spectrophotometer. A series of similar experiment was also conducted by varying the volume of HCl with optimum concentration obtained previously, *i.e.* 100, 200, 300, and 400 mL.

The HCl concentration and volume yielding the highest soluble Si was then used in the next experiment to determine the optimum volume of NaOH (s). A 150 g of 325-mesh quartz sand sample was boiled in HCl with optimum concentration and volume obtained previously following the above-described method and then a 60 g pretreated sand was mixed with different volume of NaOH (s), e.g. 20, 40, 60, 80, and 100 g. As a comparison, the same experiment was carried out by replacing the quartz sand with other Si-bearing mineral, *i.e.* a 150-mesh natural zeolite originated from Bayah, West Java. All solid samples were subjected to XRD and SEM analyses.

2.3. Chemical and Mineralogical Characterization

2.3.1. Chemical Analysis

The mineral used was quartz type collected from Bangka-Belitung province. Quartz samples were air dried and passed through 325 mesh sieves and analyzed for the following: pH, total carbon (spectrophotometer), nitrogen (Kjeldahl), phosphorus (spectrophotometer), potassium (Atomic Absorption Spectrophotometer, AAS), magnesium (AAS), calcium (AAS), total SiO_2 (gravimetry), soluble Si (spectrophotometer), Sulphur, Zinc, aluminum trioxide, iron trioxide, manganese dioxide, and cation exchange capacity (CEC) by using [22] standard method.

2.3.2. X-Ray Diffraction

Random-oriented samples were analyzed by XRD using Cu Kα radiation at 40 kV and 30.0 mA from XRD-6000 Shimadzu equipped with 1ow divergence and receiving slits and a graphite monochromator. Minerals present in the sand fraction sample were identified from random powder diffraction patterns following the procedures given by [23]. A continuous scan was applied in a horizontal scale ranging from 3 to 90˚2θ at a scan speed of 3˚2θ $\min^{-1}$ and a sampling pitch of $0.02 - 2\theta$. Calibration was carried out using a 325-mesh Silicon powder standard of Shimadzu containing 990.0 $g \cdot kg^{-1}$ SiO_2.

2.3.3. Electron Microscopy

All solid material both treated and un-treated samples were examined with a Scanning Electron Microscope (SEM). The electron beam is accelerated through a high voltage 20 kV and pass through a system of apertures and electromagnetic lenses to produce a thin beam of electrons [24]. In the early stages a material sample leveled with a special tool. After sputter coating the cast with 35 nm of gold-palladium (Au-Pd), electron micrographs were generated using a Jeol JSM-5310LV SEM.

3. Result and Discussion

3.1. Chemical Characteristics of Bangka-Belitung Quartz Sand

The Bangka-Belitung province and the adjacent area have been known since a long time ago as a main quart's sand-producing area in Indonesia. Selected chemical analyses in this research show that the quartz sand has alkaline reaction (pH H_2O-8.2) with the 1.2 $g\cdot kg^{-1}$ C-org, 0.3 $g\cdot kg^{-1}$ total N, 30.1 $mg\cdot kg^{-1}$ P_2O_5, 6.1 $mg\cdot kg^{-1}$ K_2O, 0.05 $mg\cdot kg^{-1}$ CaO, <0.01 $mg\cdot kg^{-1}$ MgO, 971 - 991 $g\cdot kg^{-1}$ total SiO_2, 0.90 $mg\cdot kg^{-1}$ H_4SiO_4, 86.0 $mg\cdot kg^{-1}$ S, 53.2 $mg\cdot kg^{-1}$ Zn, 1.7 $g\cdot kg^{-1}$ Al_2O_3, 0.6 $g\cdot kg^{-1}$ Fe_2O_3, <0.1 $g\cdot kg^{-1}$ MnO_2, and CEC 1.98 $cmol^+\cdot kg^{-1}$. The results of the analysis of the levels of silica in this study support the analysis by [25]. Reference to [23], based on the diffractogram silica sand samples analysis is dominated by the initial mineral quartz (SiO_2) and this is supported by the XRF data showing that silica sand obtained a good quality with a cadre of over 980 $g\cdot kg^{-1}$ total SiO_2. The result of this analysis show that impurities usually present in the silica sand are free and coated iron oxides, aluminum, and smaller amounts of magnesium, potassium and calcium minerals. The iron, being the most detrimental impurity, can be reduced by a number of physical, physico-chemical or chemical methods, the most appropriate method depends on the mineralogical forms and distribution of iron in the ore [26] [27]. According to [28] [29] [30], upgrading of silica sand requires partial removal of iron, and other minerals which are detrimental to its end use. While much of the liberated impurities can be reduced or removed by physical operations such as size separation (screening), gravity separation (spiral concentration), magnetic separation etc. [31] [32] [33] [34]. Sometimes, physico-chemical (flotation) or even chemical methods (leaching etc.) are to be adopted for the effective removal of iron which may be in intimate association with the mineral quite often superficially [35] [36].

3.2. Optimization of Silica Extraction Condition

In this study water-soluble Si (OSA) was obtained by washing the quartz sand with HCl prior to NaOH extraction. To determine the most optimum conditions for extraction, a series of concentration and volume of HCl was tested to obtain OSA. Data in **Figure 2** indicate that HCl concentrations are quadratically related with OSA obtained ($R^2 = 0.72^*$), with optimum concentration at 3.4 N. On the other experiment, the volume of HCl 5 N was also found quadratically correlated

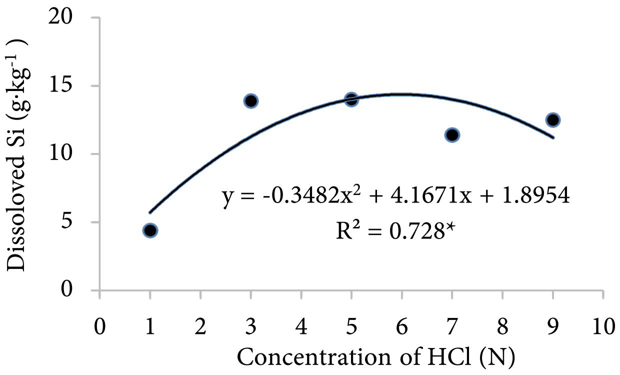


Figure 2. Correlation between HCl concentration with OSA extracted from acid-pre-washed quartz sand.

with OSA extracted ($R^2 = 0.56^*$) reaching the optimum volume at 300 mL (**Figure 3**). By employing both optimum values of HCl solution on NaOH (s) extraction experiment, it shown in **Figure 4** that extracted OSA is linearly correlated with the quantity of NaOH (s) from 20 to 100 g NaOH tested ($R^2 = 0.99^{**}$).

The results of this experiment indicate that the HCl pre-washing is important to eliminate the other element contaminants of the Si-bearing materials. The use of HCl-NaOH extraction technique on quartz sand yielded as high as 183 $g \cdot kg^{-1}$ OSA up to 100 g NaOH (s). Many researches have been reported relating to extraction of silica. Reference [37] synthesized of silica particles from rice straw waste by using KCl and KOH, while [38] produce pure silica from rice hull ash by using 1 N HCl and 60 mL NaOH. It is assumed that the efficiency of extraction using this double acid-base extraction to some extent depending on the Si-bearing material. Our data show that by using other Si-bearing mineral, *i.e.* Bayah natural zeolite, the optimum level of NaOH (s) quantity was 80 g to obtain its highest water-soluble Si, which is only 104.2 $g \cdot kg^{-1}$ (**Figure 5**). Improvement of extractable Si due to pre-treatment on the quartz sample is shown in **Table 1**. An acid-prewashed sand and its origin were insoluble in water, whereas the treatment with heated NaOH (s) on the former improved significantly the water solubility up to almost 800 $g \cdot kg^{-1}$. Extractable Si of acid pre-washed sample was considerably low (0.067 +/– 0.007 $g \cdot kg^{-1}$), while the addition with heated NaOH (s) yielded significantly high values (119.835 +/– 2.165 $g \cdot kg^{-1}$).

3.3. Mineralogical Evidences

In natural condition, Si mineral has a strong crystalline structure. The use of strong acid wash and strong base extraction will alter the crystallinity of the mineral and as a consequence releasing more Si into solution. **Figure 6** shows the XRDs indicating that the addition of HCl and NaOH (s), respectively, yielded in drastically decreasing of quartz peak intensity at 2θ of 26.5˚ and other minerals like crystobalite and feldspar. The composition of minerals was suggested to be dominated by amorphous Si material. The evidences of peak bumps appeared at between 10˚ and 20˚ 2θ is presumably due to the effect of extracting

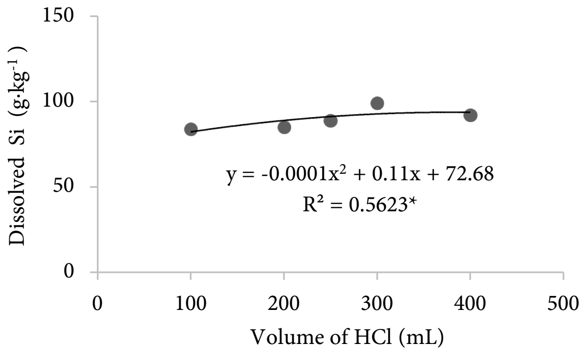


Figure 3. Correlation between volume of 5 N HCl with OSA extracted from acid-pre-washed quartz sand.

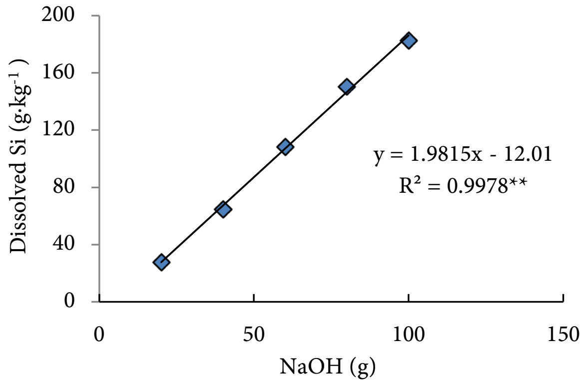


Figure 4. Correlation between quantity of NaOH (s) with OSA extracted from acid-pre-washed quartz sand.

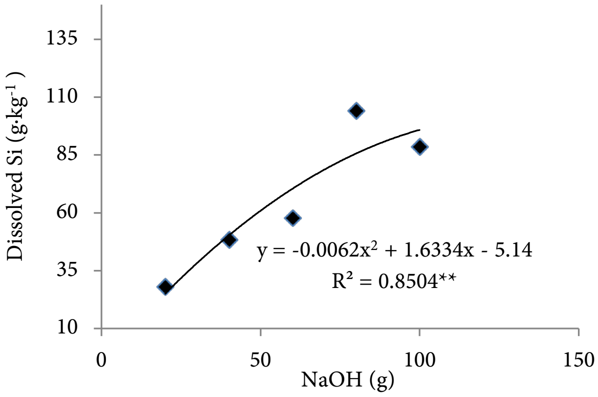


Figure 5. Correlation between quantity of NaOH (s) with OSA extracted from acid-pre-washed zeolite.

Table 1. Effects of acid-base pretreatments of quartz sand on its water solubilization and extractable Si.

Quartz Sand Treatment	Solubility in H_2O (%)	Extractable Si (%)
Untreated (origin)	0	
HCl-washed and boiled (A)	0	0.0060 - 0.0073 (0.0067 ± 0.0007)
A + NaOH (s)-mixed and heated	79.5	11.7670 - 12.2000 (11.9835 ± 0.2165)

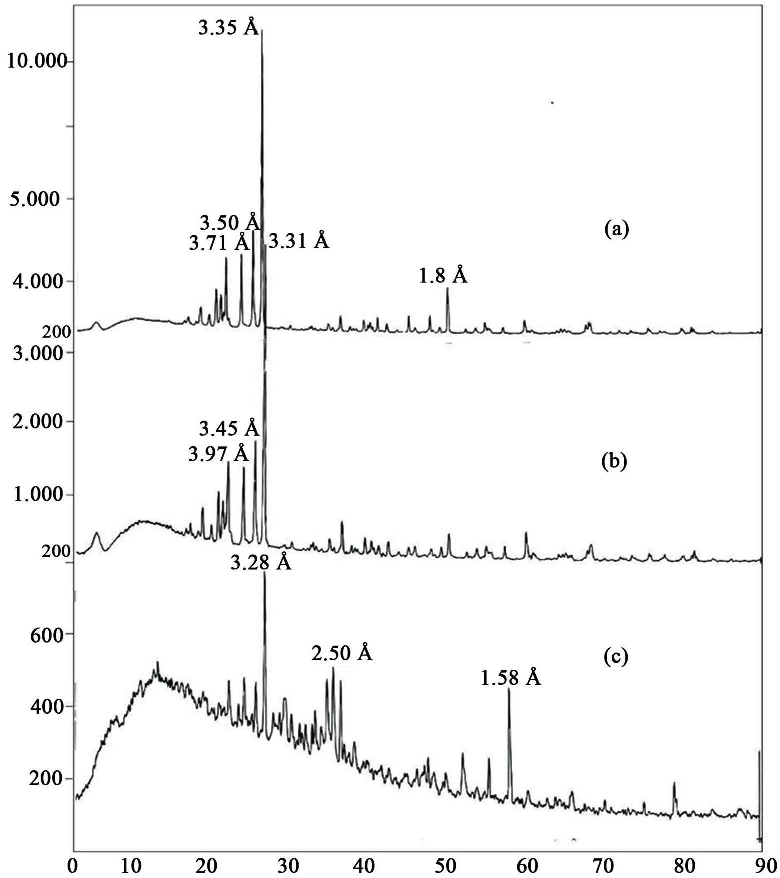
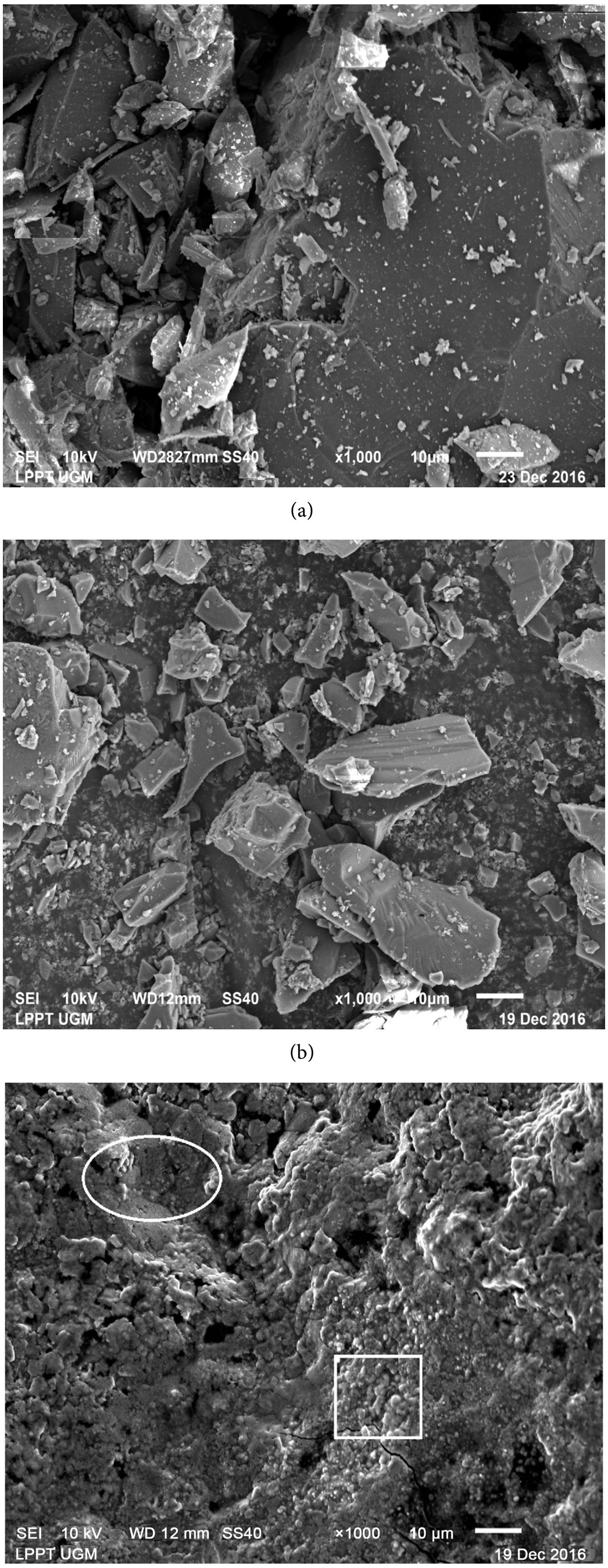


Figure 6. X-ray diffractograms of original Belitung quartz sand (a); HCl pre-washed (b); NaOH (s) reacted (c).

chemicals (*i.e.* HCl) which removed Fe-and Al oxides/hydroxides and revealed other contaminant minerals' peaks (**Figure 6(b)**). The intensity of these bumping peaks was strengthened after the sample treated with NaOH (**Figure 6(c)**). It leads to an assumption that a new mineral group (*i.e.* zeolites) was synthesized when Si reacted with hot NaOH as reported by some researchers previously [39] [40] [41]. Based on energy dispersive X-ray spectroscopy data the HCl-NaOH treated sample has mass composition of C (6.1%), O (55.4%), Na (29.3%), and Si (9.3%) with atomic distribution 9.1, 62.1, 22.9, and 6.0% of C, O, Na, and Si, respectively. The liquid obtained by dissolving of 150 g of HCl-NaOH(s) treated sand with 400 mL of distilled H_2O was dominated by amorphous Si material presumably OSA. This assumption was supported by the evidences obtained from SEM analysis (**Figure 7**).

The SEM image of the original sample of quartz (a) shows the dominance of large crystals, whereas by acid treatment (b), some quartz dissolved into smaller crystals, and by adding NaOH (c) alot of quartz dissolved to be an amorphous material and small crystalline minerals which can be shown by the image in an oval and a square sign respectively. On the other hand, it shows in **Figure 8** that the appearance of water-soluble NaOH pre-treated quartz sand resembles those of Na_2SiO_3 crystallite structure.



(a)

(b)

(c)

Figure 7. Scanning-electron microscopy (SEM) micrographs of original quartz sand (a); HCl pre-washed (b); NaOH (s) reacted (c).

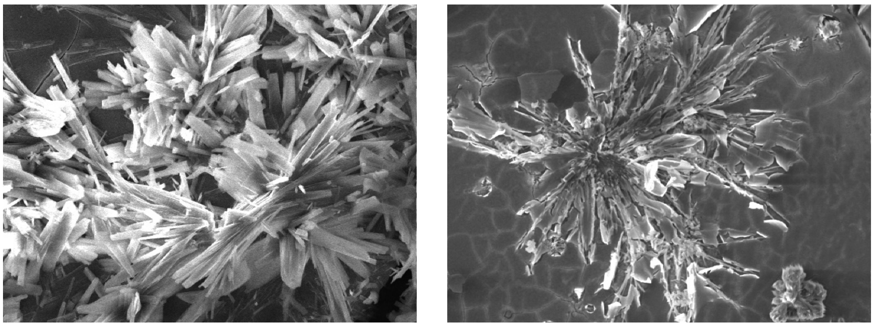


Figure 8. Scanning-electron microscopy (SEM) micrographs of water-soluble NaOH pre-treated quartz sand (a); and Na_2SiO_3, (sodium metasilicate) (b).

4. Conclusion

The results from this study show that by using HCl-NaOH (s) extraction technique on a quartz sand (970 - 990 g·kg^{-1} total SiO_2) could yield considerably high concentration of OSA suitable for silica liquid fertilizer formula. Optimum OSA yield (183 g·kg^{-1}) was obtained from this material by employing 300 mL HCl 3.4 N, reacted with 80 g NaOH (s), and diluted into 400 mL distilled water. Considering the abundantly available of the raw material, an efficient production cost for silica liquid fertilizer could be achieved.

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