Identification and Purification of Nyalo River Silica Sand as Raw Material for the Synthesis of Sodium Silicate

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Abstract. This research is on identification and purification of silica sand from Nyalo River. It will be used as a raw material for synthesis of sodium silicate. Silica sand was separated from clay by washing it with water, and then the existing alumina and iron oxide were removed by soaking the silica sand with 1 M HNO₃ solution. Qualitative and quantitative analysis of the silica sand with X-ray diffraction and X-ray fluorescence revealed that, silica sand existed in quartz form and contained a small amount of impurity oxide such as Al₂O₃, K₂O, MgO, CaO, Fe₂O₃ with percentage below the minimum threshold. The percentages of silica were 80.59% before purification. After three purification steps the silica percentage become 98.38%. It exceedsthe minimum threshold of silica percentage for industry. So, the silica sand from Nyalo River has high potency as a raw material for sodium silicate synthesizing.

1. Introduction
The Silica sand is one of Indonesia’s natural wealth, the most abundant is in West Sumatra. The silica sand of West Sumatra is 82% of Indonesia's silica sand reserves, but has not been optimally utilized to meet the needs of the Sodium silicate industry [1]. Sodium silicate is a necessary reagent as a raw material and filler of various industries such as; industrial of silica gel, paper, soap, detergent, zeolite, mesoporous silica (for HPLC, metal nanoparticles host, etc)[2]. Sodium silicate for the Indonesia industry is still imported, while the industry demand on sodium silicate is increase continually.BPS Semarang reported that the increasing of sodium silicate import from 2012-2014 is 39,093,30 to 45,664,41 ton[3].

It is predicted for the coming year, the need for sodium silicate will continue to increase. The industrial demand on sodium silicate must be accompanied by the production of sodium silicate from local materials. While, the types and chemical composition of silica sand in Indonesia is quite varied, depending on the process and the influence of impurities mineral formed during sedimentation method. Therefore, the first step of the research is on the identification and purification of silica sand as raw material which can be used as raw material for synthesis of sodium silicate.

2. Experimental Section
In this study, the identification and purification of silica sand is carried out in four step of activity as follows:
2.1. Sample collection and physical treatment
The silica sand of 1 mm to 6 mm in size with high silica content collected from the Nyalo River in the southern coastal district, 80.5 km from Padang. The clear silica sand-like glass samples are physically separated from black sand, and washed with water to release clay, dried in an oven at 110 °C for 3 hours, mashed and sieved with a 100 mesh sieve (150 µm). The third step, the dry silica sand separated using magnet, placed in plastic bottles, put in desiccators, characterize using X-Ray fluorescence, X-ray diffraction, and treated for chemical purification.

2.2 Chemical treatment
Fifty grams of fine and clay-free silica sand and minimum quantity magnetic material were soaked in 100 ml of 1M HNO₃ solution for 20 hours, then washed with water until neutral, dried in an oven at 110 °C for 3 hours, put in desiccators, and characterized using X-Ray fluorescence.

2.3 Characterization
X-Ray Fluorescence (XRF) type DWXRF minipal 4 PW403045B was used for characterization of chemical composition of silica sand. Meanwhile, the crystallinity of silica sand was characterized using X-ray diffractometer.

3. Results and discussion
The ease of retrieval silica sand, safe and cheap purification processes, high silica content, and low impurities are favorable offerings for the exploitation of silica sand for an industrial raw material [4]. The silica sand deposits of the Nyalo River is located along the river to the mouth of the river, in accordance with the silica sand formation process (Fig. 1a). Based on location, silica sand can be taken through two attractive transport routes; first, road transport, through the coastal Bungus, the area of Pisang and Pinang River, until the Nyalo River. While, the second, sea transport through the Mandeh island tourism area. The silica sand deposits of the Nyalo River are the result of weathering of rocks, carried by water and undergoing sedimentation along the river, at the mouth of the river, and along the beach. Silica sand deposits of Nyalo River are quite large. The hills around the river, estuaries and beaches looked white (Fig.1). Silica sand in this area reflects light at night. So, this area is named Nyalo river (Nyalo = glow)(local opinion).
Currently, the silica sand of West Sumatera has not been used for sodium silicate industrial. Therefore, the researchers have many opportunities to identify and purify it. Identification and purification product of silica sand is a reference point for all processing tests in the later steps of silica sand assessment procedure, for example, the silica sands a raw material for the synthesis of sodium silicate [5]. Sodium silicate can be used as raw material for paper, detergent, zeolite, and silica mesoporous industry.

There are 4 common steps for purification of silica sand; (a) mechanical process, (b) physical process, (c) chemical treatment, and (d) thermal treatment [4]. The first step has been done by separating the transparent sand from the black sand. The second step silica sand was washed to remove clay. The third step, clay-free silica sand was smoothed and sieved, to obtain larger surface of silica sand, and then it was easy to clean at the chemical treatment step, and facilitated magnetic separation. The impurity of silica sand as alumina and iron oxide had been removed by soaking the silica sand sample using 1M HNO₃ solution.

Fig. 2 shows the Nyalo River silica sand after washing with water has the same morphology and color as commercial silica sand of Sam & Bros. Dubai production. The brown appearance of silica sand showed the presence of Fe₂O₃ as impurity compounds. Therefore, the silica sand needs to be mashed first to extend the touch area with the solvent, and so on dissolved the impurity compound with 1 M HNO₃ solution. The silica sand before and after purified with HNO₃ can be seen in Fig. 3. The silica sand after magnetic separation and after wash with HNO₃ has a brighter color than silica sand before purification.
Figure 3. Silica sand before and after magnetic separated and purified with HNO₃ 1M

Percentage of impurity oxide and SiO₂ of silica sand before and after purification can be seen in Table 1. Table 1 shows the three steps of purification of silica sand are capable to reduce impurity oxides such as Al₂O₃, Fe₂O₃, MgO, K₂O and Na₂O (<2%) in silica sand, with percentage below the minimum threshold, and obtained silica sand with SiO₂ percentage was 98.38%. This percentage exceeded the minimum threshold of silica percentage for sodium silicate industry[6,7].

Table 1. Chemical Composition of silica sand before and after purification

| Oxide  | Silica sand before purification | Silica sand after being purified with water and separated by magnet | Silica sand after purified with 0.1 M HNO₃ |
|--------|---------------------------------|---------------------------------------------------------------|------------------------------------------|
| SiO₂   | 80.59                           | 96.78                                                         | 98.38                                    |
| Al₂O₃  | 13.08                           | 2.54                                                          | 0.27                                     |
| Fe₂O₃  | 5.92                            | 0.06                                                          | 0.06                                     |
| CaO    | 0.12                            | 0.09                                                          | 0.01                                     |
| MgO    | 0.20                            | 0.10                                                          | 0.09                                     |
| K₂O    | 0.05                            | 0.09                                                          | 0.14                                     |

The type and crystallinity of silica sand was characterized using X-ray diffractometer. The x-ray diffraction pattern of silica sand (Fig.4) shows that, the silica sand of the Nyalo river is quartz in the presence of the main peak of quartz at 2θ; 20.857, 26.64, 36.542, 39.75, 50.141, 59.953, 68.147. These peaks are the same as diffraction pattern of standard quartz (Reference code: 01-085-0797). Meanwhile, 2θ peaks of alumina and iron oxide are invisible. The results of characterization using X-ray diffractometer are in line with x-ray fluorescence, supported by the colour of silica sand after washing. Hence, iron oxide and alumina as impurities of silica sand are small and unobserved.

Figure 4. X-ray diffraction patterns for Fe₂O₃, Fe₃O₄, SiO₂ and silica sand of Nyalo River
Most sources of sand used by the sodium silicate industry are quartz. The existence of alumina at silica sand, and sodium silicate for zeolite and mesoporous silica industry can be allowed up to 1.5% , because, $\text{Al}_2\text{O}_3$ can substitute some $\text{SiO}_2$ in three-dimensional networks[6,7].

4. Conclusion
The silica sand of the Nyalo River could be purified by only three steps. Silica sand was separated from clay by washing it with water, and then the existing alumina and iron oxide were removed by magnetic separations, soaked with 1 M HNO$_3$ solution. The silica sand of the Nyalo River are quartz. Silica sand collection can be done via road or sea transport. Physical purification results show that, the silica sand of Nyalo River has the same morphology as commercial silica. Characterization and purification study has proved that silica sand has the highest SiO$_2$ percentage of 98.87% above the minimum threshold of industrial silica sand. The silica sand after purification has $\text{Al}_2\text{O}_3$, $\text{K}_2\text{O}$, CaO, MgO, Fe$_2$O$_3$ impurity oxides below the minimum threshold. The Nyalo river silica sand is very promising and can be utilized for a variety of industrial purposes after simple purification processes, especially as raw material for the synthesis of sodium silicate.

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