INDONESIAN MINING JOURNAL Vol. 16, No. 3, October 2013 : 149 - 153

PREPARATION OF NANO SILICA FROM SILICA SAND THROUGH ALKALI FUSION PROCESS

PENYIAPAN NANO SILIKA DARI PASIR SILIKA MELALUI FUSI ALKALI

AGUS WAHYUDI, DESSY AMALIA and SARIMAN
R&D Centre for Mineral and Coal Technology
Jalan Jenderal Sudirman 623 Bandung, 40211, INDONESIA
Phone. +62.22.6030483, Fax. +62.22.6003373
Email: wahyudi@tekmira.esdm.go.id

ABSTRACT

Silica (SiO_2) materials play an important role for industries, especially those in micron or even nano-scale size. The later has better properties and improves its quality. Nano silica is applied widely in building material, notably as a mixture of concrete. The material is also promising to be developed into amorphous nano silicon for solar cell materials. Indonesia has a lot of silica sand resources and faces a challenge to increase its quality into high product such as nano silica. Synthesizing silica nano through alkali fusion is a process that includes using the particles along with sodium hydroxide at temperature of 400-1100 °C then recrystalizing the molecules to get materials in nano size. The recrystalizing process was conducted by water leaching and filtration. The derived nano particles (gel) ranged between 40-60 nm. TEM characterization showed that the products are homogeneous, well dispersed and has specific surface area around 157 m^2/g.

Keywords: nano silica, silica sand, synthesizing, alkali fusion, particle size

SARI

Silika (SiO_2) memiliki peranan penting dalam industri terutama silika berukuran mikron bahkan skala nano. Silika nano memiliki sifat fisis dan kualitas yang lebih baik. Material ini telah digunakan dalam campuran material bangunan dan juga menjanjikan untuk dikembangkan menjadi silikon nano untuk material pembuatan sel surya. Indonesia memiliki sumberdaya pasir silika yang berlimpah dan menghadapi tantangan untuk meningkatkan nilai tambah menjadi produk kualitas tinggi seperti nano silika. Penelitian ini bertujuan untuk mensintesis nano silika dari pasir silika melalui proses fusi alkali. Proses ini dilakukan dengan cara melebur percontoh bersama NaOH pada temperatur 400-1100 °C dan kemudian direkristalisasi hingga membentuk molekul-molekul berukuran nanometer. Proses rekristalisasi yang dilakukan meliputi pelidian dengan air dan filtrasi; filtrat yang diperoleh kemudian direaksikan dengan asam klorida sehingga terbentuk endapan halus. Hasil yang diperoleh adalah gel nano silika berukuran 40-60 nm. Berdasarkan karakterisasi TEM, produk yang dihasilkan tampak homogen, terdispersi dengan baik dan memiliki luas permukaan spesifik sekitar 157 m^2/g.

Kata kunci: nano silika, pasir silika, sintesis, alkali fusion, ukuran partikel

INTRODUCTION

Silica (SiO_2) plays an important role for industries, as either raw materials or enhanced ones, such as cement industry, glass, bottles and crockery, enamel, paints, ceramics, electronics, industrial tire and even cosmetics (Sudradjat, 1997). Technology development results of using silica in industries is rapidly increase, especially small size silica in microns or even nano-scale (1 nm = 10-9 m), which has better properties and improving its quality.

Received : 18 June 2013, first revision : 29 August 2013, second revision : 23 September 2013, accepted : October 2013

Nano silica is applied widely in building material, notably as a mixture of concrete (Buitelaar, 2004 and Quercia, 2010). Any empty spaces between cement particles will be filled by nano silica that serves as concrete reinforcing materials (mechanical properties) and increases its durability. Recently the need of micro/nano-silica filled with imported products. Another application of nano silica is for additive material in the rubber and tire industries. The advantage of nano silica addition for tires is to make the tire adhesion much better, especially when the road is full of snow, reduce the generated noise and provide a better display than the old tire products (Chen, 2008 and Zhou, 2007). Nano silica is also promising to be developed into amorphous nano silicon for solar cell materials.

Indonesia is a country that has a lot of silica sand resources (Ministry of Energy and Mineral Resources RI, 2009). The country faces a challenge to increase its quality into high product. Currently, Indonesian silica sand is only used as raw material for glassware and cement industries. Through this research we try to synthesis nano silica from silica sand so that the function will be able to full fill the need for advance materials as mentioned above.

Special treatment is required when sizing the silica sand into nano size. The microsilica size can usually be obtained from top-down process, namely crushing and grinding with special milling method. Referring to such a milling method, it has been specially modified hence the ability to destroy material is much more effective. Using this method is also possible to get nanosize material (Suryanarayana, 2001). Based on the research in nanomaterial synthesis, top down method is quite easy to be conducted; however, the product was generally not homogeneous and still contains agglomerated particles as a result of milling effect (Wahyudi, 2012). This phenomenon can be avoided when using bottom up methods.

Alkali fusion is one of the bottom up methods that conducted by fusing the particles with alkali at a certain temperature and then recrystalizing the molecules into nanosize. This process is already used to separate zircon sand ($ZrSiO_4$) into high purity zirkonia and nano silica powder as a by product (Yamagata, 2010). The nano silica looks homogen in particle size, high specific area and well dispersed.

METHODOLOGY

Silica sand was obtained from Bangka, Indonesia. It was dried at 105 °C and then crushed into -200 mesh using jaw crusher and ball mill. Alkali fusion process was conducted by reac ting the sand along with NaOH in stoichiometri ratio of 1:1. The temperature process was varied between 400-1100 °C and fusing duration lasted for two hours in the furnace. The samples were leached using water to separate Na-silica from other minerals. Na-silica filtrate was then titrated with HCl up to pH 7 until get a silica gel in nano size. Material characterization included analyzing particle size by (PSA), measuring specific surface area by Brunauer Emmett Teller method (BET) and characterizing product morphology by transmission electron microscope (TEM).

RESULTS AND DISCUSSION

The Bangka sand contains 98.75% SiO_2, 0.079% TiO_2, 0.054% Al_2O_3, 0.389% Fe_2O_3, 0.003% CaO, 0.217% NaO and 0.25% LOI. Based on mineralogy characteristics from XRD analysis, the mineral in the samples was 100% quartz (Figure 1).

The Alkali fusion method for making nano silica particles was conducted by melting the sample at certain temperature and then precipitated the molecules into nanometer scale. The steps of alkali fusion process are shown in Figure 2. This process was influenced by temperature, time, acid concentration and pH. The product was then characterized by PSA to measure the particle size, as shown in Figure 3.

Alkali fusion causes binding condition of silica due to NaOH to form sodium metasilicate. Thus it can be separated from other gangue minerals. Sodium metasilicate treats with acid mineral (HCl) yields hydrous silica and silicic acid. Washing the product will have nano silica gel. The following equations describe the reaction (Greenwood, 1984):

$SiO_2 + 2\ NaOH \rightarrow Na_2SiO_3 + H2O$................ (1)
$Na_2SiO_3 + 2\ HCl \rightarrow SiO_2 + 2\ NaCl + H_2O$ ..... (2)

The above data shows that the alkali fusion runs effectively at all fusion temperature, even in the lowest temperature (400 °C). This suggests that

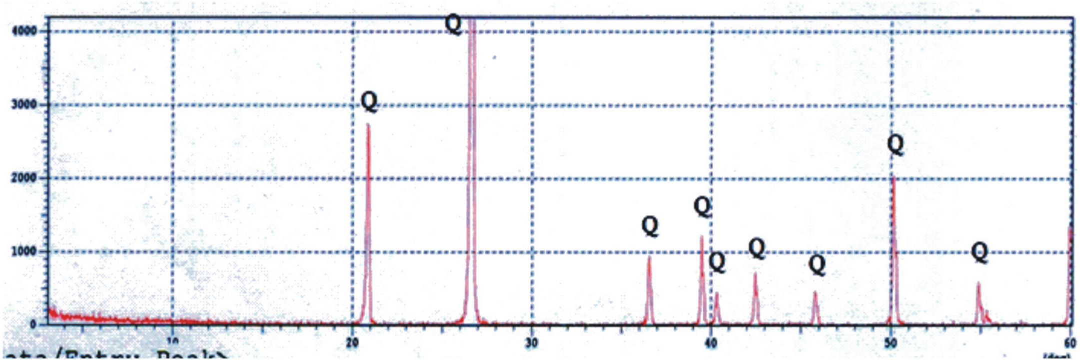


Figure 1. Result of XRD analysis on Bangka Sand

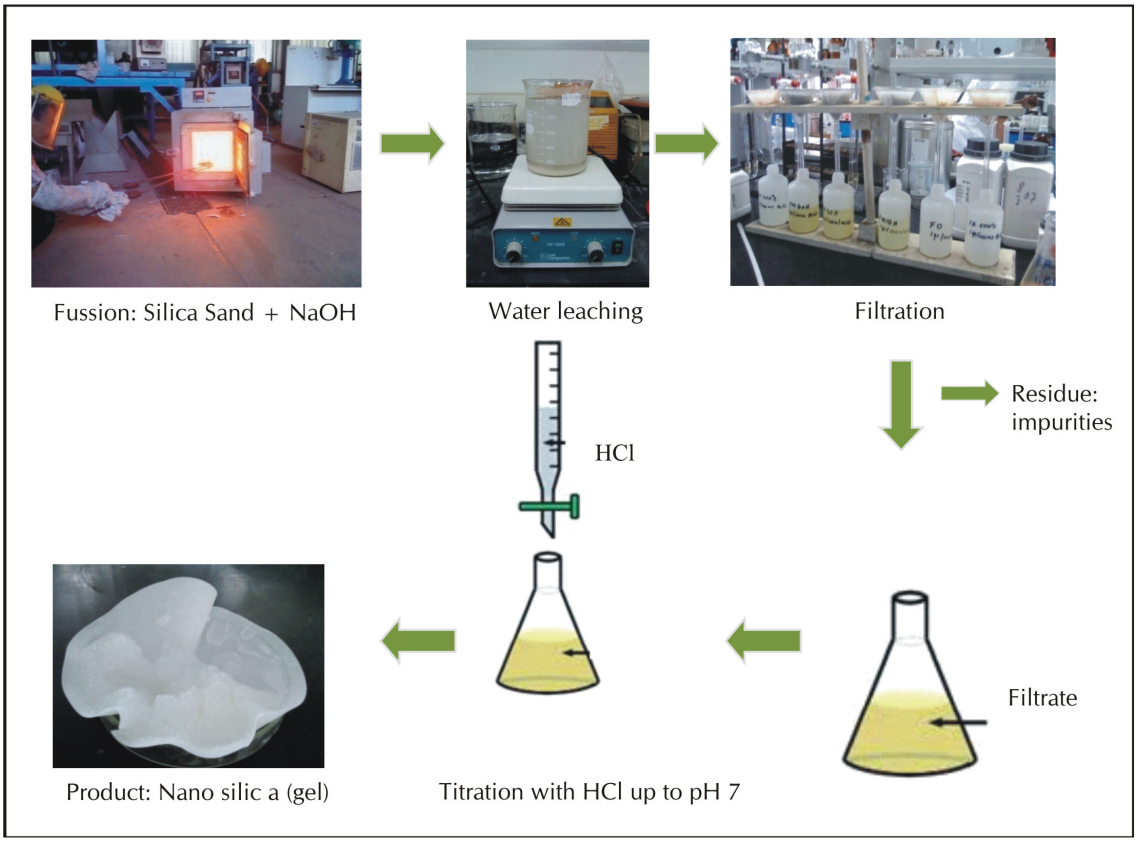


Figure 2. Synthezing nano silica by alkali fusion method

the process is effective and efficient to produce nanoparticles and does not require high energy.

The main mineral in silica sand is quartz that has tetrahedron structure as shown in Figure 4. At very high temperatures, each tetrahedron will separate from each other since the bond between the anion and the cation is not too strong (Schweigert, 2001) as shown in Figure 5.

Nano silica resulted from alkali fusion has a good characters, namely more homogenous in size and well dispersed (not agglomerated) compared to nano silica from milling processs as shown by TEM characterization (Figure 6); the synthesis nano silica by milling process tends to aglom-erating (clumping) due to van der Waals force between particles at the surface (Suyanarayana, 2001).

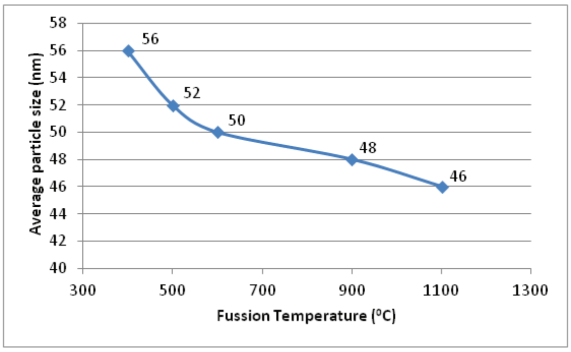


Figure 3. Size of nano silica derived from several fusion temperatures

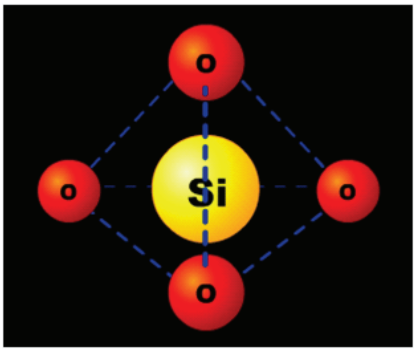
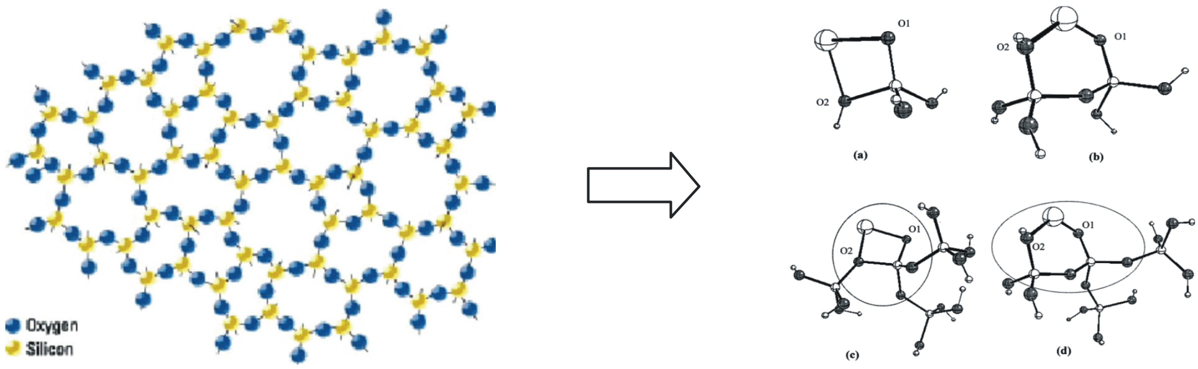
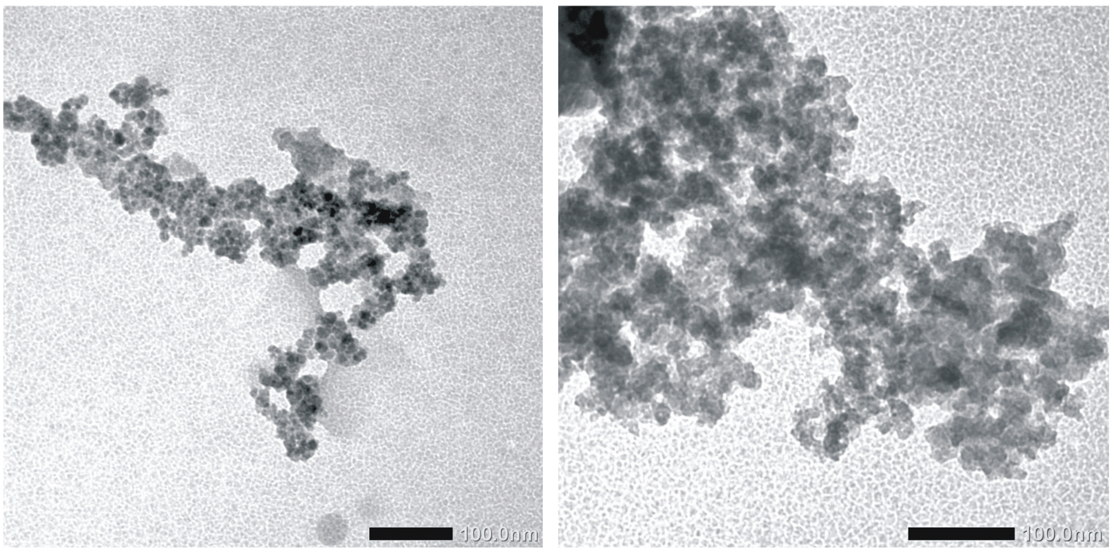


Figure 4. Tetrahedron structure of silica mineral (Schweigert, 2001)



(a) (b)

Figure 5. a) Bonding structure of tetrahedron silica, b) termination of tetrahedron silica structure (Zuraw, 2003)



(a) (b)

Figure 6. TEM photo of nano silica resulted from: (a) alkali fusion process at 900 °C; and (b) milling process (Wahyudi, 2012)

Measurement specific surface area by BET method shows that the nano silica has specific surface area up to 157 m^2/g at 900 °C fusion temperature. It seems that the higher fusion temperature the higher spesific surface area (Figure 7).

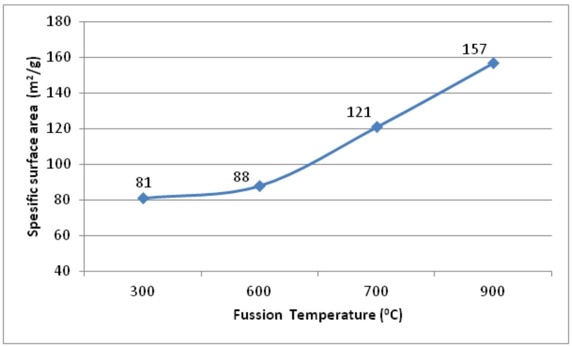


Figure 7. Spesific surface area of nano silica at several fusion temperatures

This phenomenon can be explained as follows: separation of silica sand is conducted by making dissolved and undissolved phases in water. The former is obtained by adding the sodium from NaOH to be sodium silica. Regardless of forming the dissolved phase, sodium can also damage the tetrahedral structure of SiO_2 hence the separation process can be carried out at not too high temperature. If the fusion temperature increases the bonding association between anion and cation in tetrahedron system will be weak and broken. In this case the higher the temperature, the more separated the tetrahedron particle size becomes smaller. As a consequence the surface area will increase (Schweigert, 2002).

CONCLUSIONS

The average size of synthesis silica nanoparticles from Bangka silica sand is 40-60 nm. The particles come from the alkali fusion process. Characterizing the particles by TEM shows well dispersed and homogenous size while the measured surface area of the particles reaches up to 157 m^2/g.

ACKNOWLEDGMENT

The authors would like to acknowledge Mr. Tatang Wahyudi, Ir., M.Sc for his valuable insight and criticism which improved the quality of this paper.

REFFERENCES

Buitelaar, P., 2004. Ultra High Performance Concrete: Developments and Applications during 25 years. *Proceeding of Plenarry Session International Symposium on UHPC*, Germany.

Chen, Y., 2008. Natural Rubber Nanocomposite Reinforced with Nano-Silica. *Jounal Polymer Engineering and Science*, p. 1674-1677.

Greenwood, N.N. and Earnshaw, A. (1984). Chemistry of the Elements. *Oxford: Pergamon Press*. p. 393–99.

Ministry of Energy and Mineral Resources RI, 2009. *Indonesian Geology Resources*, 2nd Book: Sumatera Island.

Quercia, G. and H.J.H. Brouwers, 2010. Application of Nano-Silica in Concrete Mixtures. *Proceeding of 8th fib PhD Symposium in Kgs*. Lyngby, Denmark.

Schweigert, I.V., K.E.J. Lehtinen, M.J. Carrier, and M.R. Zachariah, 2002. Structure and Properties of Silica Nanoclusters at High Temperatures. *Phisical Review B*, Vol 65.

Sudrajat, Adjat, Supriatna Suhala dan M. Arifin, 1997. *Pasir Kuarsa (Bahan Galian Industri)*, Pusat Penelitian dan Pengembangan Mineral, p. 260-279.

Suryanarayana, C., 2001. Mechanical Alloying and Milling. *Progress in Material Science*, Pergamon, p.122-124.

Wahyudi, A., Teguh N., dan Rochani, S., 2012. Sintesis nanopartikel silika dari mineral alam Indonesia secara Mekanis dan prospek pemanfaatannya, *Proceeding of Seminar JASA KIAI*, Yogyakarta.

Yamagata, Chieko, Joao B.A., Valter U., Nelson B.L., Jose O.A.P., 2010. High purity zirkonia and silica powders via wet process: alkali fusion of zirkon sand, *6th International Latin-American conference of powder technology*, Brazil.

Zhou, Hong Jun, et.al., 2007. Role of Reactive Compatibilization in Preparation of Nano-Silica/Polypropylene Composites, *Journal Polymer Engineering and Scince*, Vol. 47 (4), p. 499-509.

Zuraw, Michael K., 2003. *Principles of Chemical Vapor Deposition*, Kluwer Academic Publisher, Netherland, p. 199.