# Nanosilica reinforced-composites Al/$SiO_2$ as anti-corrosion materials at high temperatures



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## Backgraound

In geothermal exploitation, besides water steam, the contaminants such as sulfur, vanadium, sodium, etc. are also emitted as by-products, which can react at high temperatures with air and water forming *molten salts* which may probably accelerate corrosion. Therefore, for high temperature applications, materials having mechanical strength, high resistance to corrosion and oxidation are absolutely necessary (Sudiro, 2010). The focus of this research is to fabricate aluminum matrix composites with nanosilica reinforcers to provide anti-corrosion materials especially for high temperature applications such as geothermal installation. Nano-silica particles will be synthesized from natural materials (quartz sands).

## Methods and Materials

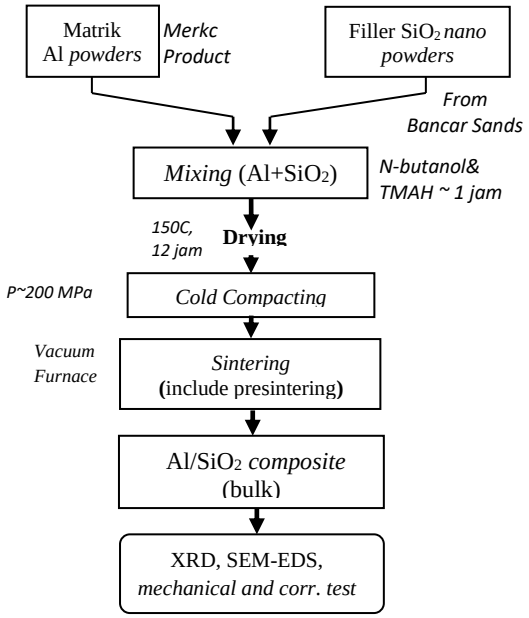


Figure 1 Fabrication process of Al/$SiO_2$ composite with powder metallurgy methode.

## Result and Discusion

Fabrication of Al/$SiO_2$ composites with addition of N-butanol and tetra-metil ammonium hydroxide (TMAH) as active solutions, followed by sintering at 500 °C for 2 hours in vacuum furnace, has been successfully conducted using simple powder metallurgy approach. The major starting materials were commercial Al powders and extracted amorphous $SiO_2$ powders from Indonesian silica sands. The composites were comprehensively characterized by means of XRF, XRD, and SEM-EDS. Further characterizations in terms of physical, mechanical, and corrosion rate tests were also done. The addition of N-butanol into Al/$SiO_2$ (later abbreviated as Al/$SiO_2$(B)) leaded to broader. Beside Al, γ-$Al_2O_3$ crystal was also detected in the composites which was well confirmed by microstructural analysis. The unhomogenous distribution of $SiO_2$ with agglomeration characteristic within Al sheets was observed.

Introducing the amorphous $SiO_2$ into Al matrix decreased the density or increased the porosity of the composites. In addition, compression strength to reduce as the addition of $SiO_2$. The highest values of yield strength, compression strength and modulus of elasticity were observed in the composites with 5% $SiO_2$. In general, the corrosion rates in 1 M NaCl medium for Al/$SiO_2$(T) were lower than Al/$SiO_2$(B) composites. For comparison, corrosion rates of 1.6144 mm/y and 0.16936 mm/y were obtained by introducing 20% of $SiO_2$ into Al/$SiO_2$(B) and Al/$SiO_2$(T), respectively

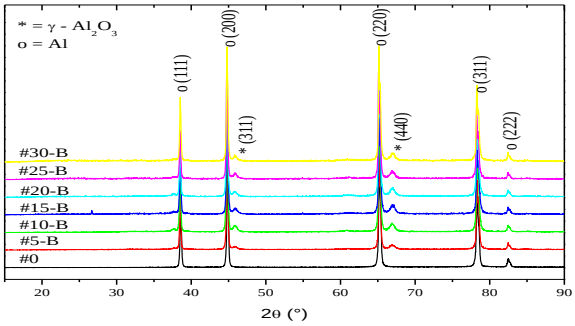


Figure 2 XRD pattern of Al/$SiO_2$ composite (Bulk) after sintering

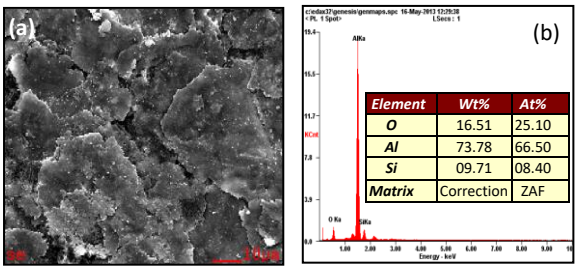


Figure 3.(a) SEM images, (b) EDX and element analysis of Al/$SiO_2$ composite (bulk) .

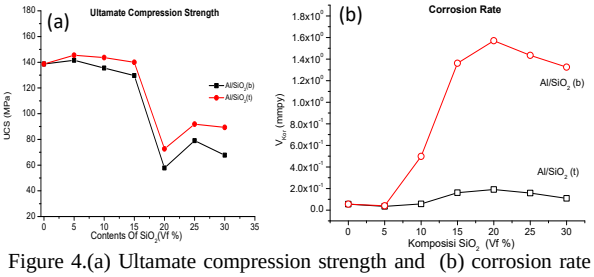


Figure 4.(a) Ultimate compression strength and (b) corrosion rate of Al/$SiO_2$ composite (bulk) .

## Acknowledgments

One of the authors (*Munasir*) would like to acknowledge the finacial support from Ministry of Education and Culture through postgraduate study scholarship at ITS Surabaya, Indonesia.

## Reference

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# SUPERKAPASITOR DENGAN ELEKTRODA BERBASIS BAHAN ALAM

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## PENDAHULUAN

Baterai telepon selular maupun laptop yang tersedia di pasaran banyak yang tidak sesuai dengan kebutuhan pengguna, karena memiliki rapat daya yang cukup kecil dan saat pengisian muatan (charge) membutuhkan waktu cukup lama. Oleh sebab itu, dibutuhkan teknologi baterai yang memiliki rapat daya yang besar, proses pengisian muatan yang cepat dan usia pemakaian (life cycle) yang lama, salah satunya adalah superkapasitor. Proses penyimpanan muatan pada superkapasitor ini tergantung dari bahan penyusun elektroda, umumnya digunakan karbon aktif. Namun nilai kapasitansinya rendah, sehingga diperlukan paduan untuk dijadikan sebagai elektroda superkapasitor. Paduan tersebut antara lain Fe3O4 dari pasir Lumajang, rGO dari tempurung kelapa, polimer seperti PANI dan PVDF. Adanya paduan dari elektroda tersebut, diharapkan nantinya dapat dijadikan sebagai devais superkapasitor dengan elektroda dari bahan alam yang memiliki nilai kapasitansi terbaik sehingga dapat dijadikan sebagai alternative baterai yang ramah lingkungan.

## METODE YANG DIGUNAKAN

### TAHUN PERTAMA

**PEMBUATAN ELEKTRODA AKTIF BAHAN ALAM**

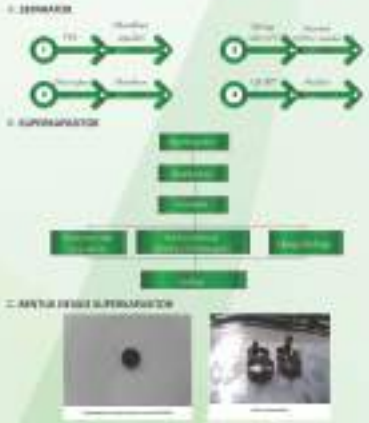



**PEMBUATAN ELEKTRODA KARBON AKTIF DAN KOMPOSIT MENGGUNAKAN DRY MIXING**




### TAHUN KEDUA

PEMBUATAN DEVAIS SUPERKAPASITOR DENGAN ELEKTRODA BAHAN ALAM DAN POLIMER HYDROSOL



## HASIL YANG DICAPAI

### TAHUN PERTAMA

| No. | Jenis Kaliber Mesh | Luas Area (m²/g) | Nilai Kapasitansi (Cs/Fg) |
|---|---|---|---|
| 1 | Tempurung Kelapa | [illegible] | [illegible] |
| 2 | Tempurung Kelapa | 1,5 | [illegible] |
| 3 | Tempurung Kelapa | [illegible] | [illegible] |
| 4 | Bio Kapasit | [illegible] | [illegible] |
| 5 | Kulit Kelapa | [illegible] | [illegible] |



BENTUK KURVA VOLTAMOGRAM



Berdasarkan hasil sebelumnya aktif untuk elektroda karbon aktif dengan material lain seperti Fe3O4, PANI, rGO maupun PVDF, material yang kapasitansi untuk perlu dikembangkan dalam bentuk devais superkapasitor adalah pada elektroda karbon aktif/sekarta PVDF, dengan nilai kapasitansi terbaik pada perlakuan tahun pertama ini, sebesar 12,134 F/g

### TAHUN KEDUA



[illegible] | [illegible] | [illegible]

## KESIMPULAN & SARAN

1 — 2 — 3 — 4

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# Fabrikasi Nanokomposit Pani-$SiO_2$/Acrylic Paint Sebagai Prototipe Material Pelapis Anti-Korosi Pada Pipa Power Plant Energi Geotermal



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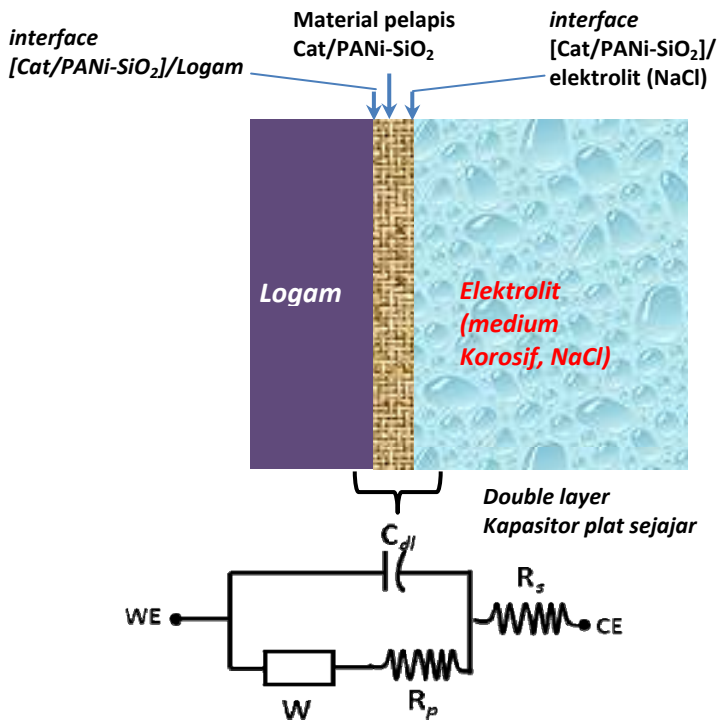
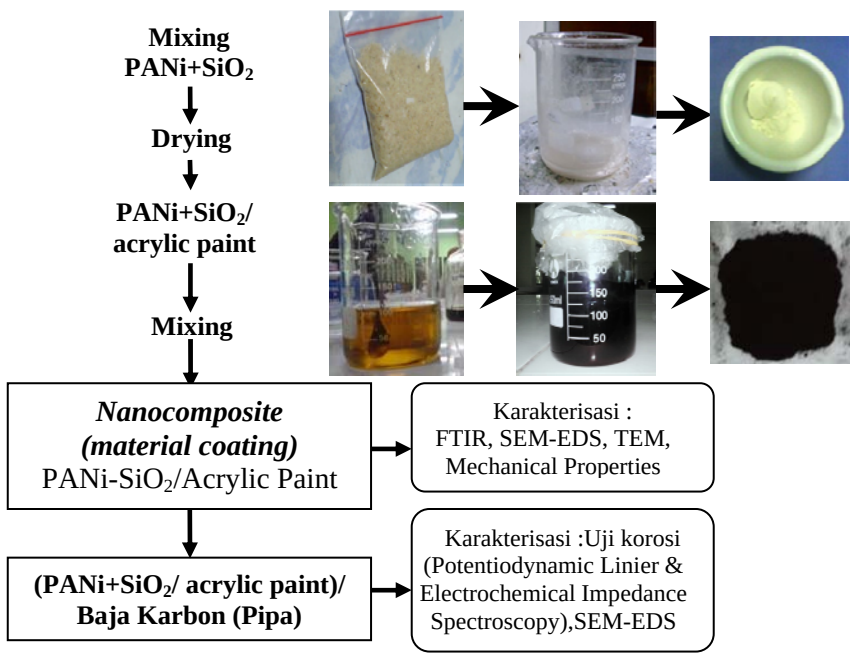
## Latar Belakang

Polianilin (PANi) adalah salah satu bahan polimer konduktif yang banyak dikaji pada lebih dari dua dekade terakhir karena sifat fisika dan kimianya yang khas sehingga memiliki potensi aplikasi yang luasPolimer konduktif sebagai bagian sistem perlindungan korosi terbukti mempunyai keunggulan tertentu, apalagi jika digabung dengan nanosilika yang merupakan partikel polar dan memiliki sifat mekanik dan isolator yang baik. Nanokomposit PANi-$SiO_2$ adalah sebuah alternatif yang sangat menjanjikan sebagai material pelindung anti-korosi pada permukaan logam. Bahan $SiO_2$ nanopowders disintesis langsung dari bahan alam (pasir kuarsa), yang tersedia secara melimpah di Indonesia (*local resource*).




**Gambar 1.** Model interface pada pelapisan anti-korosi pada permukaan logam PANi-$SiO_2$/Acrylic Paint & madium korosif (NaCl)

## Metode dan Material



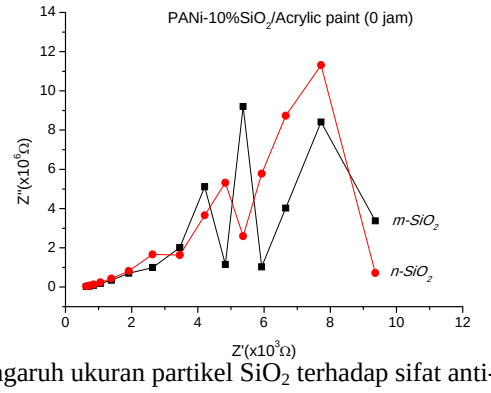

**Gambar 2**. Tahapan Sintesis PANi/$SiO_2$/Acrylic Paint



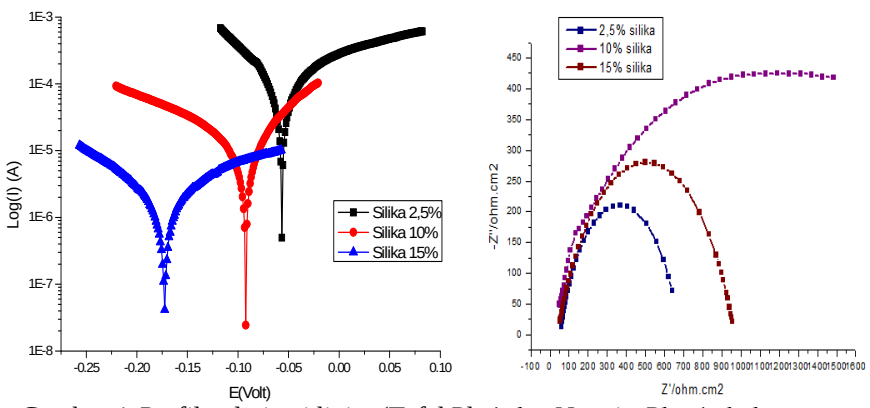
**Gambar 2**. Tahapan Pelapisan PANi/$SiO_2$/Acrylic Paint pada plat

## Hasil dan Diskusi

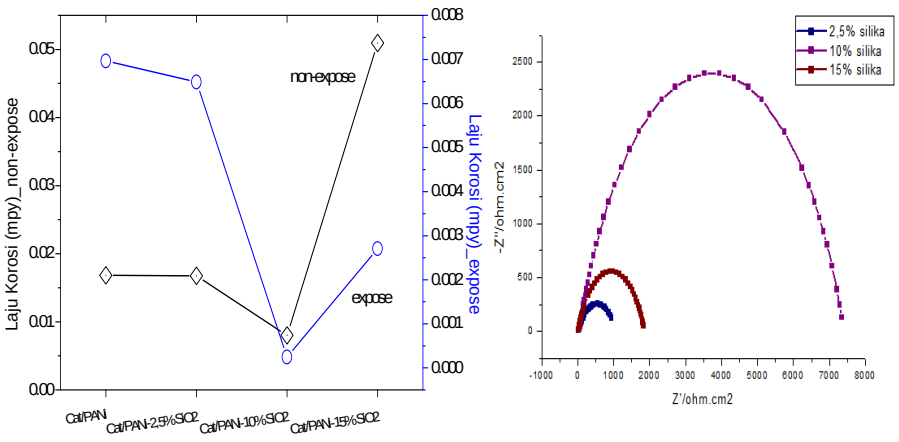
Ukuran partikel (nano atau mikro) silika $SiO_2$ menunjukan ada perbaikan sifatanti-korosif untuk ukuran yang lebih kecil (nano). Prototipe material pelapis anti-korosi PANi-$SiO_2$/*Acrylic Paint* telah diuji coba secara eksperimen, dilapiskan pada permukaan baja (pipa power plant) di diexpose didalam medium (sintetis) geotermal kumudian dianalisis sifat performa anti-korosinya (laju korosi dan hambatan polarisasinya, Z) dengan menggunakan metode potensiodinamik (Tafel plot) dan electrochemical impedance spectroscopy (Nyquist plot). Prototipe komposisi material pelapis PANi-$SiO_2$/*Acrylic Paint*, dengan pengujian potensiodinamik (Tafel-kurva) dan EIS (Nyquist plot dan Bode plot) menunjukan mempunyai performa ketahanan korosi yang baik, baik untuk kondisi sebelum ekspose maupun setelah ekspose; masing-masing dengan laju korosi 0,008 *mpy* (sebelum expose) dan 0,000244 *mpy* (setelah expose); dan dengan hamabatan polarisasi (Rp) berturut-turut adalah 3.760 Ohm (sebelum expose) dan 7.370 Ohm (setelah expose). Tampak disini bahwa material pelapis mempunyai karakteristik performa anti-korosinya semakin membaik ketika berada didalam medium expose (geotermal).




Gambar 3. Pengaruh ukuran partikel $SiO_2$ terhadap sifat anti-korosi ( Nyquist Plot)




Gambar 4 Profil polarisasi linier (Tafel Plot) dan Nyquist Plot (sebelum expose)




Gambar 5. Laju korosi dan Nyquist Plot (setelahexpose)

Analisis EIS digunakan untuk menunjang pemahaman mengenai mekanisme korosi yang terjadi pada masing-masing sampel. Diagram Nyquist dari sampel 2,5% silika, 10% silika, 15% silika menggambarkan plot impedansi imajiner terhadap impedansi real. Dari ketiga sampel juga menunjukkan plot semi-circle dengan diameter berbeda dari masing-masing komposisi silika pada sampel. Pada Gambar 17, tampak diagram Nyquis untuk sampel Cat/PANi-$SiO_2$ sebelum dan sesudah ekpose. Impedansi Z, terdiri komponen imaginer ($Z_{im}$) dan riil ($Z_{real}$); bagian riil menunjukan bagian hambatan konduktif (R) yang mencakup $R_s$ adalah tahanan larutan dan Rct/Rp adalah hambatan transfer muatan, dan bagian imaginer (sumbu vertikal) menunjukan bagian kapasitansi lapis rangkap listrik CdI/CPE.

## Ucapan Terimakasih

Ucapan terimakasih disampaikan kepada DP2M Dikti melalui skim Hibah Bersaing. Dan terimakasih disampaikan juga kepada LPPM-Universitas Negeri Surabaya. Peneliti mendapat dukungan finansial melalui dana BOMPTN-UNESA dengan SPK: 170/UN.38.9/HK/LT/2015.

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RISTEKDIKTI

# Nanoparticle of $Fe_3O_4/SiO_2$-Amorphous: Water Treatment Material Application

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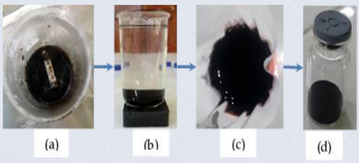
## RESEARCH IN BRIEF

This work presents, The aim study, $Fe_3O_4/SiO_2$ composites have been synthesized through two stages; first, the synthesis of $Fe_3O_4$ particles from iron sands; second, the synthesis of $Fe_3O_4/SiO_2$ using the in-situ method by mixing Fe3O4 nanoparticles with ammonia, ethanol, and TEOS in one pot as a chemical reaction site.. Characterization results showed that the phase structure of the $Fe_3O_4/SiO_2$ composite was successfully formed. The VSM test results showed that the magnetization saturation of $Fe_3O_4/SiO_2$ composites was 18 emus/g and based on the TEM test results it was seen that $Fe_3O_4$ particles were coated by $SiO_2$ particles with $Fe_3O_4$/SiO2 particle size of ~ 100 nm.

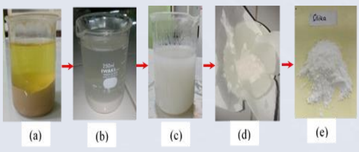
## EXPERIMENT




Synthesis process of MNPs Fe3O4

Testing



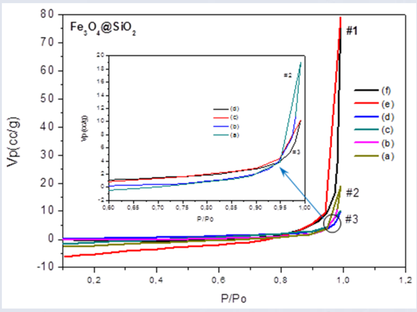




Synthesis process of NPs SiO2

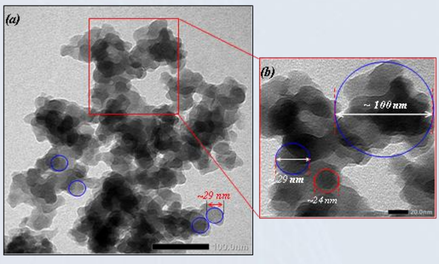
The final M-B is absorbed




## RESULT AND DISCUSSION

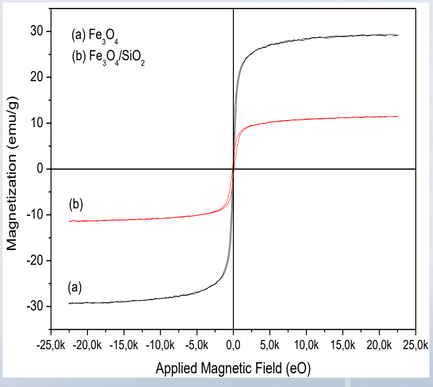



Adsorption-Desorption isothermic of nitrogen gas for Fe3O4/SiO2




TEM of NPs-SiO2

The Fe3O4 nanoparticles powder has been successfully synthesized from iron sand mineral extracted by the co-precipitation method and the SiO2 (amorphous) nanoparticles powder from quartz sand




VSM testing of Fe3O4 NPs and Fe3O4/SiO2

## CONCLUSION

The $Fe_3O_4/SiO_2$ composites have been successfully synthesized by the In-situ method, and magnetic properties can be controlled by increasing the number of $SiO_2$ particles added. The VSM test results indicate that as the volume of TEOS increases of $SiO_2$ magnetization decreases, the characteristics of the magnetization curve include soft superparamagnetic materials. Furthermore, it has good absorption properties for dye solutions, such as methylene blue.

## ACKNOWLEDGMENT

The author thanks the State University of Surabaya, which has provided support to the author, so that the data collection and writing can be completed. the author also wishes to thank the ministry of research and technology (DRPM-Kemenristek), who have supported financially.

# Synthesis and Characterization of amorphous Silica from Mud Vulcano (LuSi)



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## Backgraound

The Sidoarjo mud is a type of volcanic mud that covers an area of over 6.5 square kilometers, it has been dislocating more than 30.000 people since the first flow. On 30th of October 2008, the flow reached 100.000 $m^3$ per day, and it is predicted to be continue for the next 30 years [Jalil,et.al., 2010]. The Sidoarjo mud, however, can be well proposed to be an abundant source of silica and has a great chance to be used for the development of science and technology. It is based on many studies that has tried to take the advantages of the mud, such as a mixture of bricks, zeolite, etc [Taufiqur Rahman, 2006] concluding that the silica content in the Sidoarjo mud was significant enough to be separated.

The limitation of this study is to find answers to questions: (1) how to purify and synthesize the silica of Sidoarjo Mud from the other chemical elements or mixed oxides, (2) how to produce nano-sized powder of silica powder from Sidoarjo mud by means of coprecipitation method.

## Methods and Materials

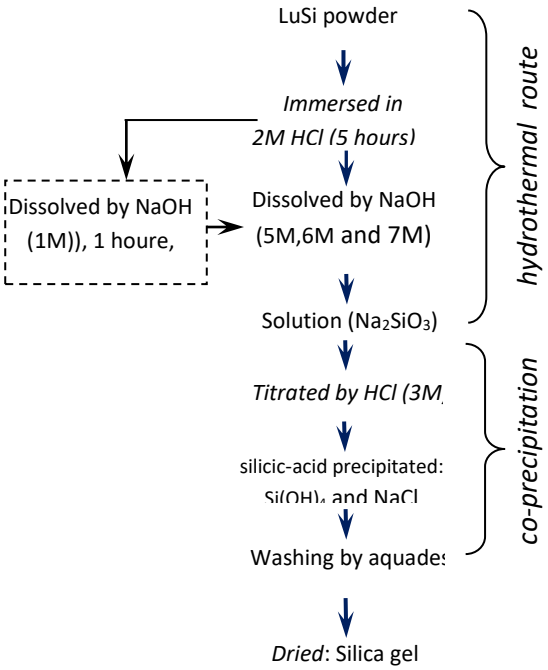


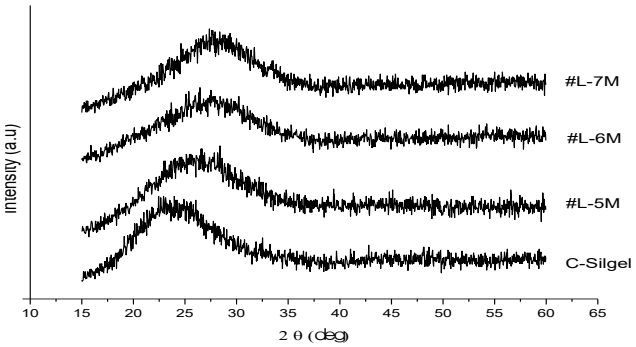

Figure 1 Synthesis process of nano silica powders with alkalifusion methode.

## Result and Discusion

Lusi powder ($xSiO_2$) +2NaOH → $Na_2O.xSiO_{2\,(l)}$+ $H_2O_{(l)}$ (1)

$Na_2O.xSiO_{2\,(l)}$ + $2HCl_{(l)}$ + $H_2O_{(l)}$ → $2NaCl_{(l)}$ + $Si(OH)_{4(aq)}$ (2)

$Si(OH)_{4(aq)}$ → $SiO_{2(s)}$ + $2H_2O$ (3)




| *Sample* | *Si Atomic (%)* | *$SiO_2$ (%)* |
|---|---|---|
| Sand Quartz | 58,20 | 65,90 |
| a-$SiO_2$ (product) | 96,40 | 98,5 |

Figure 2 XRD pattern and XRF Characterization of Quarzt Sands and Silica Amorphous (Synthesis)

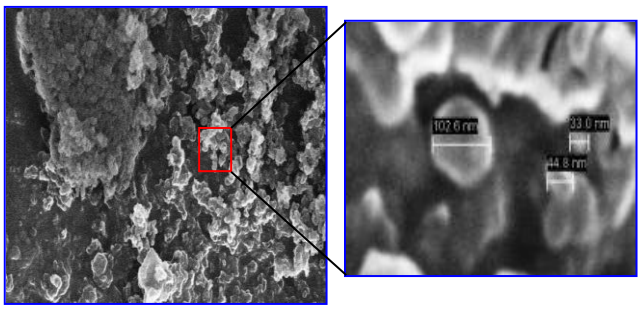



Figure 3 TEM images of silica produced (extraction with 7M NaOH).

According to the experimental results, it can be concluded that a coprecipitation method can be performed to synthesize amorphous silica from Sidoarjo mud. A separation of $SiO_2$ from its impurities like $Al_2O_3$, $Fe_2O_3$, CaO, $K_2O$ and $Na_2O$ can be done by using NaOH and HCl solvents. Variation of NaOH molarities (i.e. 5M, 6M, and 7 M) obtained various final mass product of silica gel, i.e. 0.12 g, 0.19 g, and 0.553 g, respectively. Finally, the coprecipitation method can be used to precede silica purification from Sidoarjo mud with a high purity of 95.7% of agglomerated spherical silica and particle size of about 33 nm. It, however, needs to chase a further study to produce a higher purity of silica that can be extracted from the Sidoarjo mud and to obtain the maximal mass product of silica. A temperature treatment in term of applying heat is also desired to break the chemical bonding in $SiO_2$ to be able to form sodium silicate ($Na_2SiO_3$) and hence obtain more $SiO_2$.

## Acknowledgments

One of the authors (*Munasir*) would like to acknowledge the finacial support from Ministry of Education and Culture through postgraduate study scholarship at ITS Surabaya, Indonesia.

## Reference

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2. Jalil, Aishah.A., et.al. (2010). ***Journal of Hazardous Materials***, 181:755-762.

# Synthesis of Silica Nano Powder Extracted from Slopeng Natural Sand via Alkalifussion Route



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## Backgraound

Crystal structure of silica ($SiO_2$ ) are Quartz, Tridymite and Crystobalite, and also orde nano of $SiO_2$ have an Amorf phase. SiO2 is formed by strong, directional covalent bonds and very strong, has a high melting point~1700ºC, hard, and insolube in water an organic solvent. The bond angles around O-Si-O are essentially the tetrahedral angle, 109 degrees; the Si-O distance is 1.61 A (0.16 nm) with very little variation,

Trabelsi has been successfully synthesized an amorphous silica from inorganic material, such as sand of Deuriet by reacting sodium carbonate ($Na_2CO_3$) with heating temperature of 1030ºC [1]. Hidetsugu Mori has been proposed another synthesis approach to obtain 99.9% of silica in a way of breaking the chemical bonding using alkali solution, like KOH and NaOH and then followed by bonding the silica ($SiO_2$) from waste colore glasses [2-3], this method is known by alkalifussion method.

## Methods and Materials

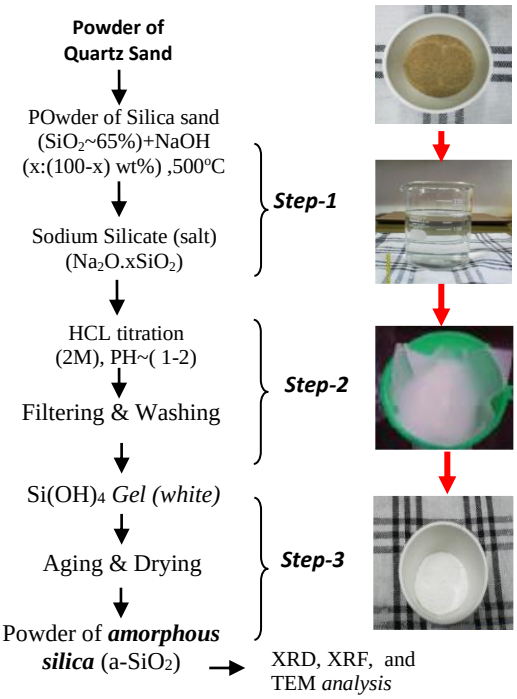
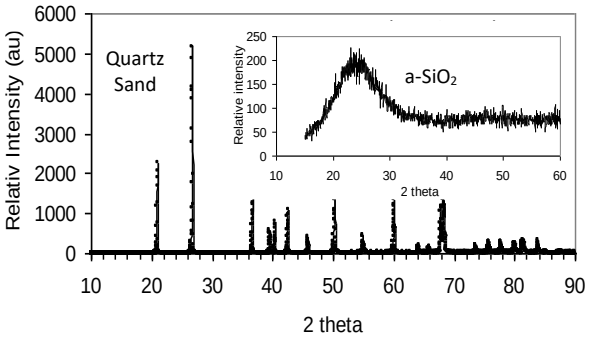



Figure 1 Synthesis process of nano silica powders with alkalifusion methode.

## Result and Discusion

The silica nano-order has to be purified and synthesized from quartz sand taken from Madura Slopeng with alkalifusion method. Quartz powder mixed with NaOH in the ratio 12,5:87,5 wt% and burned in a furnace at 500ºC temperature with holding time 2 hours. Sodium silicate obtained and processed as above flowchart, obtained SiO2 powder with nano-order size and an amorphous phase.




| *Sample* | *Si Atomic (%)* | *$SiO_2$ (%)* |
|---|---|---|
| Sand Quartz | 58,20 | 65,90 |
| a-$SiO_2$ (product) | 89,50 | 98,9 |

Figure 2 XRD pattern and XRF Characterization of Quarzt Sands and Silica Amorphous (Synthesis)

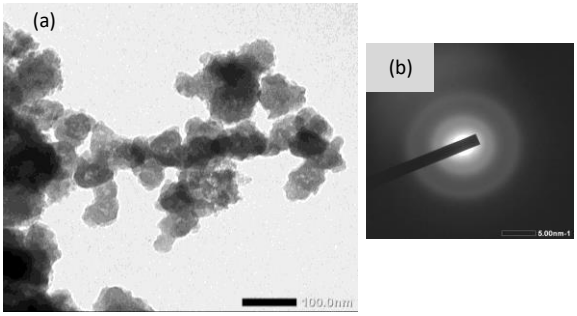



Figure 3 TEM images of silica produced (extraction with 87,5% NaOH), (a) particle size is < 100 nm, particles agglomeration and (b) amorphous phases.

The result of this works: (1) An amorphous silica can be well synthesized from quartz sand by means of alkalifussion method using NaOH at heating temperature of 500ºC.; (2) The amorphous silica produced by alkalifussion method is 98,9 wt%; (3)Comprehensive characterizations by XRD and TEM revealed to the same results that the silica exhibits a nanomaterial behavior having the particle size of ~70 nm and amorphous phase.

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