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Synthesis of Mesoporous SiO_2 Xerogel from Geothermal Sludge using Sulfuric Acid as Gelation Agent

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ABSTRACT

A large amount of sludge is produced by the geothermal brine at the Dieng Geothermal power plant, exceeding 165 tons per month. This sludge is generally not utilized, except for use in landfills. The precipitate (sludge) is primarily composed of silica. The aim of this research is to synthesis mesoporous silica (SiO_2) xerogel from geothermal sludge (GS) and to investigate the effects of pH as an effort to elevate the economic value of sludge through alkaline extraction followed by acidification. SiO_2 xerogel was prepared by extracting the GS to become sodium silicate (Na_2SiO_3) assisted by a base NaOH and precipitated using H_2SO_4 as a gelation agent. The FTIR analysis of the SiO_2 xerogel showed a group of silanol (Si-OH) and siloxane (Si-O-Si). The XRD analysis indicated that SiO_2 xerogel was amorphous. Furthermore, it was observed from nitrogen absorption-desorption using BET (Breneur-Emmet-Teller) method test that decreased pH tends to the specific surface area increase, and the pore size becomes decrease. The largest specific surface area observed at SiO_2 xerogel prepared at pH of 5.5 reached 400.10 m^2/g with a pore size of 4.5 nm. The pore sized for all cases was in the range of 4 ~12 nm, indicating that the SiO_2 xerogels were mesoporous. Pore size of the as-prepared silica affected the thermal stability property of the sample.

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1. INTRODUCTION

Finding new generations of energy sources highly recommended due to an increase in energy demand worldwide. The one alternative solution to overcome potential problems of worldwide energy deficiency is using nepw and renewable energy (NRE). One of the promising NRE technology for developing countries such as Indonesia is geothermal energy. Geothermal energy harnesses the energy generated below the surface of the earth via steam generation. Such energy source is available abundantly and does not depend on the availability of fossils-based fuel [1]. One of the problems faced while producing electricity from geothermal energy is solid and liquid wastes (sludge). [2]. The liquid waste was handled by re-injection into the earth's surface layer. [3]. The solid waste also needs to be handled due to the clogging effect of SiO_2 deposit inside the well tube, causing a reduction of power generation. The production of geothermal sludge (GS) in one of the geothermal power plants in Indonesia could exceed 165 tons per month while its utilization is minimal

SiO_2 has good chemical stability, is not soluble in water, and resistant to high temperatures. In general, SiO_2 can be obtained from inorganic materials and organic materials. The most widely known silica is silica TEOS (Tetraethyl orthosilicate) and TMOS (Tetramethyl orthosilicate), which have the advantage of being able to bind aggregate rock into monolithic material. However, this silica has a weakness, both of which have prices that are relatively expensive, difficult to obtain, and not environmentally friendly [4]. Based on the disadvantages

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of silica source, TEOS and TMOS, the previous researchers conducted a lot of research to obtain alternative silica by utilizing natural ingredients that were not too useful before.

SiO_2 can be produced in several forms including fused quartz, crystals, irritated silica (or pyrogenic silica, colloidal SiO_2, SiO_2 gel and aerogels [5-7]. SiO_2 aerogel is a substance made from silicon and is the low density solid. It is formed from 99.8% air and rigid foam with a density of 3 mg cm^{-3}, low density, high surface area and low thermal conductivity [8]. With the advancing technology, SiO_2 from the waste could also be utilized as precursors of nanosilica [9,10] or mesoporous SiO_2 xerogel which could be applied as catalysts [11,12], adsorbents [13–15], ultrafiltration [16,17], drug delivery [18,19] and other applications [20-22].

Previous researchers have done a lot of research to synthesis SiO_2 using various plants such as rice husk waste [7,8]. However, there are still few researchers conducted research by utilizing geothermal sludge (GS) as an alternative raw material for SiO_2. SiO_2 from wastes or natural resources could be obtained through facile alkaline extraction and acidic precipitation method [23-25]. This process and its product characteristics are strongly affected by pH level during the synthesis. Muljani et al. [26] have succesfully produced mesoporous SiO_2 gels from geothermal sludge HCl and tartaric acid as gelling agents. In the sol-gel preparation, others hydrolysis catalysts were used such as HNO_3 and H_2SO_4 as gelation agent. It has also been reported that sulfated SiO_2 as solid acid catalyst is one of the modified silica gel products by reacting silica gel with sulfuric acid. The utilization of SO_4^{2-} anion display an increase in acidic properties of silica as well as physical properties [27, 28].

Different from the previous research, herein, we improved a modification of synthetic route to prepare mesoporous SiO_2 xerogel with geothermal sludge (GS) from Dieng Mountain as raw material. The modification is done to maximize the work of SiO_2 gel, especially for catalysts in the acidification process using H_2SO_4. We further investigate the effects of pH during the formation of mesoporous SiO_2 xerogel. This work is done as our contribution and effort to elevate the economic value of geothermal sludge (GS).

2. MATERIALS AND METHODS

2. 1. Material and Synthesis The materials used in this study were Mount Dieng Geothermal Sludge (GS), Indonesia. Solvents and reagent used were analytical grade of ethanol (Merck, 96%, distilled water, sodium hydroxide (NaOH, Merck), and sulfuric acid (H_2SO_4, Merck 96%).

The GS was dried in a oven at 80°C for 60 minutes, ground and screened using 80 and 200 meshes filters. 10 g of the prepared solid waste was extracted in reflux using 1.25 M NaOH (Merck) solution for an hour at 80°C and stirred continuously at 300 rpm. Extraction solution was cooled to room temperature and filtered using Whattman number 42 filter paper. The filtrate was sodium silicate. The sodium silicate solution was dissolved 5 times in demineralized water. 1 N solution of H_2SO_4 were added into the dissolved solution until the pH reached 10. The as-prepared solution was aged for 2 hours and reintroduced with 1N H_2SO_4 until the solution reaches various pH level (9, 8, 7, 6 and 5.5). After the desired pH level achieved, each solution was aged for 18 hours. The formed gel was dissolved using 300 mL of demineralized water and filtered by vacuum. The residue was dried in a oven at 80°C for 12 hours and washed three times using demineralized water. The washed residue was dried furtherly at 100°C for 24 hours. The SiO_2 xerogel was obtained. Figure 1 shows the schematic diagram of the material preparation.

2. 2. Material Characterization The functional groups of starting material and samples were analyzed using Shimadzu IR Prestige 21 FTIR (Fourier Transform Infra-Red). X-ray diffraction pattern of starting material and SiO_2 xerogel sampel were studied using Shimadzu XRD-7000 Maxima X-Ray Diffractometer (XRD) with CuKα (λ= 0.154 nm), scanning speed of 0.02°, 2θ angle range of 10° – 90°, and applied power of 3 kW. The surface area were analyzed using BET (Breneur Emmet Teller) method using ASAP 2020 V4.20E Surface Area Analyzer (SSA). The morphology was characterized by Scanning Electron Microscopy (JEOL, JCM-7000 NeoScope™ Benchtop SEM) with voltage of 10 kV. TG/DSC (Hitachi STA200RV) was used to characterize the thermal stability, adjusted to a temperature range from 25 to 1000 °C in an air atmosphere at a heating rate of 10 °C/min.

3. RESULTS

The formation of SiO_2 xerogel from PLTP (Geothermal Power Plant) Dieng by alkaline extraction are expressed

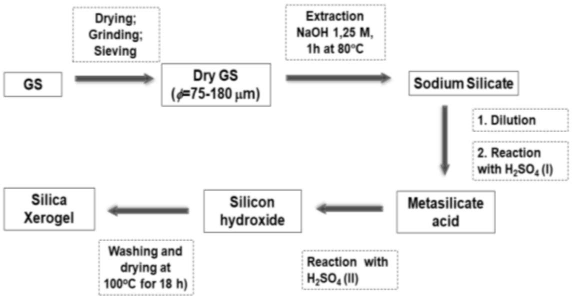

Figure 1. Experimental procedure for the preparation of mesoporous SiO_2 xerogel from geothermal sludge

in Equations (1), (2) and (3) [4, 29].

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \qquad (1)$$

During the extraction of silica, the silica reacts and dissolved into this alkaline NaOH solution to form sodium silicate (Na_2SiO_3) solution. To reduce the particle sizes of SiO_2 xerogel, demineralized water is added to the sodium silicate solution to increase its concentration. Thus, when precipitation occurs, small precipitate is formed due to slow nucleation process.

$$Na_2SiO_{3(aq)} + H_2SO_{4(aq)} \rightarrow H_2SiO_{3(aq)} + Na_2SO_{4(aq)} \qquad (2)$$

The next process is a metasilicate acid formation by adding aqueous sulfuric acid H_2SO_4 into the sodium silicate solution. With the increasing amount of H^+, a polymerization reaction of silicate acid occurs.

$$H_2SiO_{3(aq)} + H_2O_{(aq)} \rightarrow Si(OH)_{4(aq)} \qquad (3)$$

Gel formation occurs due to hydrolysis reaction of polymeric silicate acid with demineralized water while the xerogel were formed due to the drying of gel at 100°C for 24 hours. The porous xerogel is formed. Infra-red adsorption from difference group functions in geothermal sludge and prepared samples are shown in Figure 2. FTIR characterization of each samples are based on the study performed by Brinker and Scherer. The adsorption on wave number 3400 cm^{-1}, 1630 cm^{-1}, 1430 cm^{-1}, 1099 cm^{-1} and 950 cm^{-1} show stretch vibration of –OH from Silicanol (Si-OH) or H_2O, -OH from Si-OH, C-H, Si-O from Siloxane (Si-O-Si), Si-O from Si-OH respectively. Wavenumber of 790 cm^{-1} shows symmetrical Si-O from Si-O-Si stretch vibration, wavenumber of 450 cm^{-1} and 2300 cm^{-1} show bending vibration of Si-O=Si [30–32].

The increasing of adsorption Intensity at wavenumber 1630 cm^{-1} from sample S0 (waste), S1 (pH 9), S2 (pH 7) and S3 (pH 5.5) happened due to the proton content in the solution during the synthesis forming Si-OH. High level of pH causes the reaction tendency to form Si-O-Si (siloxane) proved by the increasing intensity at 1060 cm^{-1}. The hydrocarbon impurities (1430 cm^{-1}) are detected in geothermal sludge sample and the peak is shifted after the end of the process or when the xerogel is formed.

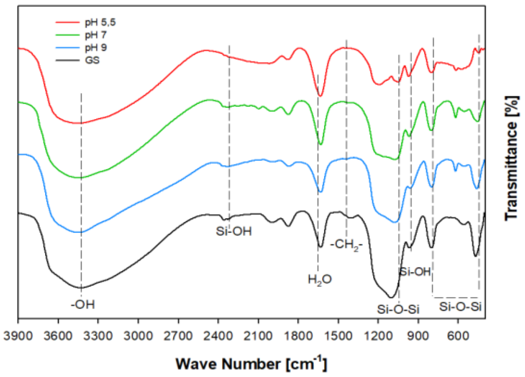

Figure 2. FTIR spectra of geothermal sludge and SiO_2 xerogel synthesized at various pH level

X-ray diffraction patterns of the samples are presented in Figure 3. The XRD pattern showed the amorphous nature of the SiO_2 xerogels. The broad XRD pattern was typical for an amorphous structure [33]. This is indicated by the appearance of a widening peak centered at an area of 2θ around 25˚ where silica with an amorphous structure gives a diffraction that widens at 2θ around 21-25˚. Impurities peaks (19° and 31°) of sodium sulfate (Na_2SO_4) is detected in sample 2 (pH 9) and sample 3 (pH 7). The presence of impurities could be caused by an unfinished cleaning process or salt entrapment in SiO_2 matrices [29]. Aside from the impurity peak, all peaks from all samples are well indexed to JCPDS 7757-82-628.

Surface analysis of each sample is performed by BET method and the result is presented in Table 1. Based on the result, the highest surface area, 400.10 m^2/g, is exhibited by sample synthesized at pH level of 5.5. With the increasing pH, the surface area of the sample is

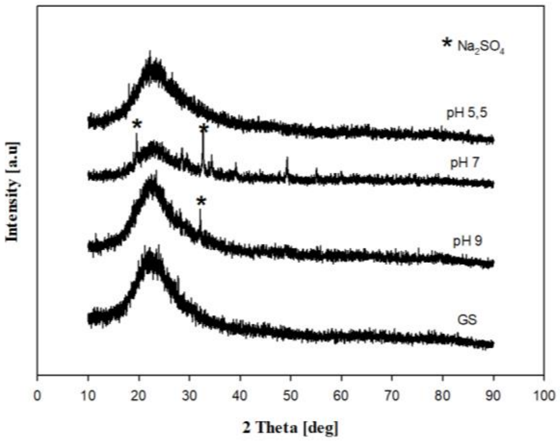

Figure 3. XRD Pattern of geothermal sludge and silica xerogel synthesized at various pH level

TABLE 1. BET analysis result of solid waste and samples synthesized at various pH level

Samples	Specific Surface Area [m^2/g]	Specific Pore Volume [cm^3/g]	Pore Diameter [nm]
GS	100.50	0.11	4.3
pH 9	159.55	0.47	11.8
pH 8	151.74	0.34	8.9
pH 7	223.44	0.48	8.7
pH 6	321.25	0.40	4.9
pH 5.5	400.10	0.45	4.5

decreased. This phenomenon is mainly caused by the slow formation of gel and slow nucleation of SiO_2 occurred at low pH level [29].

The amount of Nitrogen (N_2) adsorbed into the SiO_2 xerogel at various pH level synthesize shown in Figure 4. The isothermal adsorption of N_2 shows the pore volume capacity of SiO_2 xerogel. The highest pore volume is exhibited by the sample synthesized at pH 7 with the value of 0.48 cm^3/g. Based on the pore volume data stated in Table 1, there is no significant effect of pH toward the pore volume of SiO_2 xerogel. In the other hand, the pore diameter is significantly impacted by the pH level of synthesis. Figure 5 shows that the pore diameter is decreased with the decreasing pH level while the specific surface area is increasing. The specific surface area in samples aged at pH level 5.5 increased by 2.5 times compared to samples aged at pH 9 and 4 times compared to raw material (GS). The pore diameter of samples is within the range of 4-12 nm which could be concluded that the porous material has mesoporous size (2-50 nm).

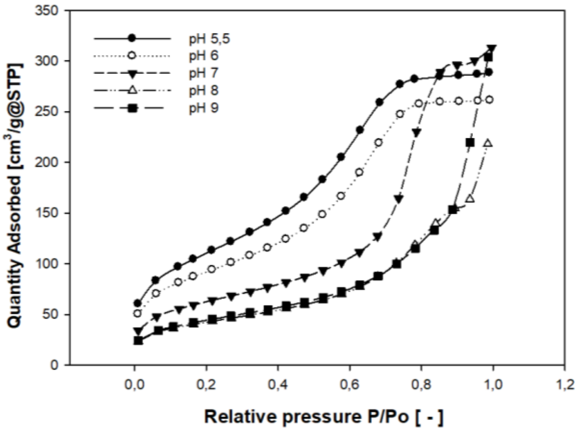

Figure 4. Isothermal adsorption of N_2 in SiO_2 xerogel synthesized at various pH level

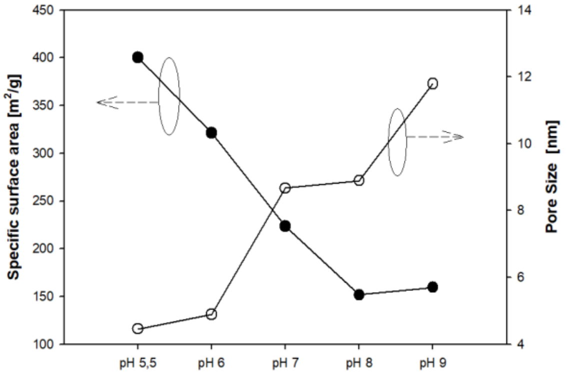

Figure 5. The effect of pH level during synthesis of silica xerogel on the surface area and pore diameter

A side from the pore diameter, the mesoporous SiO_2 xerogel is classified based on its adsorption-desorption profile. Figure 6 shows type-IV-like adsorption-desorption curve which could be applied for porous material hence the samples exhibits mesoporous size (2-50 nm). Type-IV curve initiated with slow adsorption due to the stronger intermolecular interaction between adsorbate than with the adsorbents. Then the curve is slightly bent due to the pore filling by the adsorbate [34–36].

Structure and properties of xerogels are influenced by gelation pH. Because condensation reactions are favored and hydrolysis reactions are restricted under alkali conditions formed particles are fewer in number but larger and denser than particles formed under acidic conditions. Since pH affects the coagulation process, experiments were run at different initial pH 1N H_2SO_4 until the solution reaches various pH level (9, 8, 7, 6 and 5.5). SEM results showed that SiO_2 particles are agglomerated at different pH values. The morphology of materials has distributed with diverse morphological structure. Figure 7f shows the chemical composition of the produced silica xerogel. The sample consist of Si (silicon) and O (oxygen).

TG/DSC was performed to characterize the thermal stability of SiO_2. Figure 8 shows the TG/DSC of SiO_2 prepared at different pH level at temperature from 25 to 1000°C in air atmosphere at heating rate of 10°C/min.

The weight loss and endothermic peak below 150°C are primarily caused by the desorption of physically adsorbed water [37]. The weight loss and broad

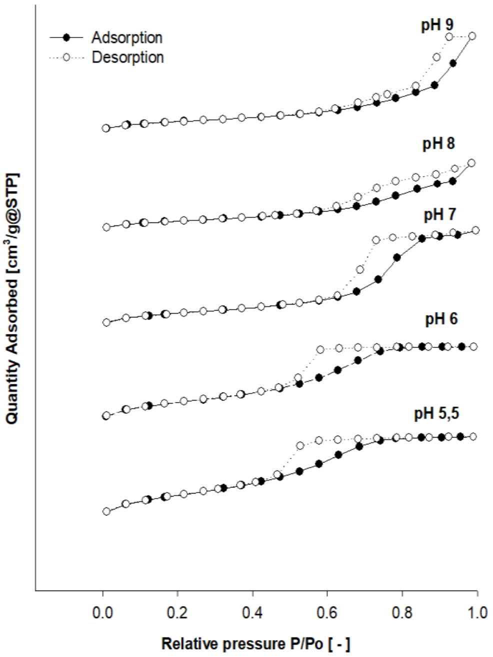

Figure 6. Adsorption-desorption curve of silica xerogel

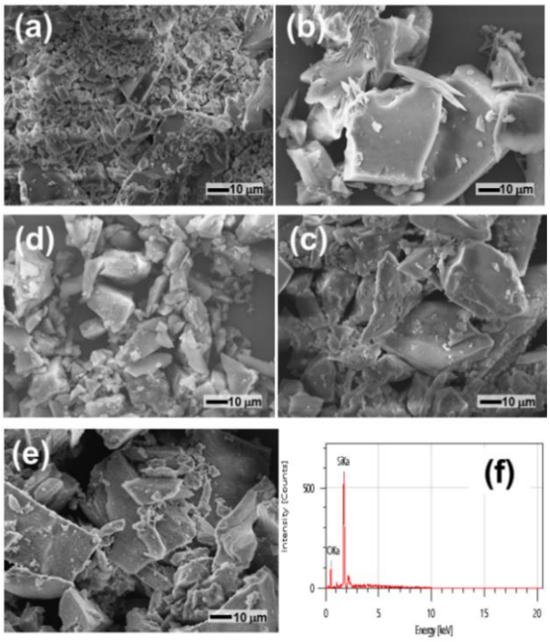

Figure 7. SEM images of SiO_2 particle at different pH values. (a) pH=5.5 ; (b) pH=6; (c) pH=7; (d) pH=8 (e) pH=9, and (f) EDX-spectra of sample

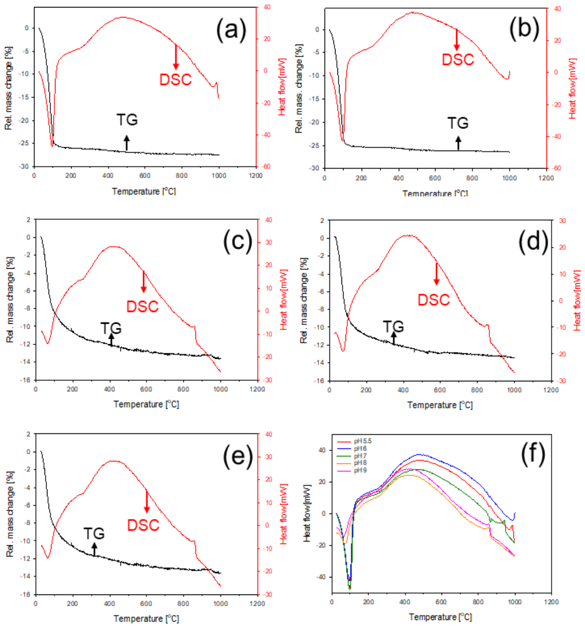

Figure 8. TGA/DSC thermograms of synthesized SiO_2 under different pH values. (a) pH= 5.5; (b) pH = 6; (c) pH=7; (d) pH=8; (e) pH=9, and (f) DSC curve of SiO_2 at different pH level

exothermic peak from 200 to 1000 °C might be attributed to the decomposition of the residual organics and the structural transition from amorf silica to crystalline silica. Obviously, at temperatures below 150, the weight loss in samples aged at pH 5.5 and 6 (acidic condition) was 25% and was greater than that in samples aged at pH 7 and above (neutral and alkaline condition), which was 8%. Figure 8f shows the DSC curve of the samples at various pH levels. Obviously, at temperatures below 150, the heat flow of the endothermic process in the sample aged at acidic conditions (5.5 and 6) was greater than that in the sample aged at pH level 7 and above. Meanwhile, at temperatures between 800-1000, exothermic peaks were seen, where the samples which were aged at acidic pH levels (5.5 and 6) showed exothermic peaks at higher temperatures compared to samples that were aged at neutral and alkaline conditions. This is possible due to the specific surface area and porosity of the sample.

4. CONCLUSIONS

SiO_2 xerogel was successfully synthesized from geothermal sludge by alkaline extraction and acidic precipitation using sulfuric acid. The characteristics of prepared material are strongly affected by the pH level of synthesis. From the FTIR analysis, silanol group and siloxane group is detected in every sample. XRD pattern shows amorphous structure of xerogel. Based on the BET analysis, at a lower pH level of synthesis (pH=5.5), larger surface area was produced (400.10 m^2/g). The pore diameter of each SiO_2 xerogel was categorized as mesoporous (2-50 nm). Pore diameter and specific surface area of the as-prepared silica affected the thermal stability property of the sample. SEM results showed that SiO_2 particles are agglomerated at different pH values.

5. ACKNOWLEDGMENT

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6. REFERENCES

1. Abdolalipouradl. M, Khalilarya, S, and Jafarmadar. S, “Energy and Exergy Analysis of a New Power, Heating, Oxygen and Hydrogen Cogeneration Cycle Based on the Sabalan Geothermal Wells”, ***International Journal of Engineering, Transactions C: Aspects***, Vol. 32, No. 3, (2019), 445-450, doi: 10.5829/ije.2019.32.03c.13.
2. Manzella. A, Bonciani. R, and Allansdottir. A, “Environmental and social aspects of geothermal energy in Italy”, ***Geothermics***, Vol. 72, (2017), 232-248, doi: 10.1016/j.geothermics.2017.11.015.
3. Finster. M, Clark. C, Schroeder. J, and Martino. L, “Geothermal produced fluids: Characteristics, treatment technologies, and management options”, ***Renewable and Sustainable Energy Reviews***, Vol. 50, (2015), 952-966, doi:

10.1016/j.rser.2015.05.059.

4. Affandi. S, Setyawan. H, Winardi. S, Purwanto. A, and Balqis. R, "A facile method for production of high-purity silica xerogels from bagasse ash", ***Advanced Powder Technology***, Vol. 20, (2015) 468-472, doi: 10.1016/j.apt.2009.03.008.

5. Brinker. C. J, and Scherer. W, "Sol-gel sciences,' in The Processing and the Chemistry of Sol-Gel Processing", ***Academic Press***, Vol. 26, No. 2, (1990), 211-212, doi: 10.1016/0254-0584(90)90039-d.

6. Mathieu. B, Blacher. S, Pirard. R, Pirard. J. P, Sahouli. B, and Brouers. F, "Freeze-dried resorcinol-formaldehyde gels", ***Journal of Non-Crystalline Sollids***, Vol. 212, No. 2-3, (1997), 250-261, doi: 10.1016/S0022-3093(97)00025-2.

7. Klvana. D, Chaouki. J, Repellin-Lacroix. M, and Pajonk. G. M, "A new method of preparation of aerogel-like materials using a freeze-drying process", ***Le Journal de Physique Colloques***, Vol. 50, (1989), C4-29-C4-32, doi: 10.1051/jphyscol.

8. Pajonk. G. M, "Drying Methods Preserving the Textural Properties of Gels", ***Le Journal de Physique Colloques***, Vol. 24, No. C4, (1989), C4-13-C4-22, doi: 10.1051/jphyscol:1989403.

9. Gurav. J. L, Rao. A. V, Rao. A. P, Nadargi. D. Y, and Bhagat. S. D, "Physical properties of sodium silicate based silica aerogels prepared by single step sol-gel process dried at ambient pressure" , ***Journal Alloys and Compounds***, Vol. 476, No. 1-2, (2009), 397-402, doi: 10.1016/j.jallcom.2008.09.029.

10. Shebl. M, Saif. M, Nabeel. A. I, and Shokry. R, "New non-toxic transition metal nanocomplexes and Zn complex-silica xerogel nanohybrid: Synthesis, spectral studies, antibacterial, and antitumor activities", ***Journal of Molecular Structure***, Vol. 1118, (2016), 335-343, doi: 10.1016/j.molstruc.2016.04.037.

11. Capeletti. L. B, do Carmo. M. A. M, Cardoso. M. B, and dos Santos. J. H. Z, "Hybrid silica based catalysts prepared by the encapsulation of zirconocene compound via non-hydrolytic sol-gel method for ethylene polymerization", ***Applied Catalysis A: General***, Vol. 560, (2018), 225-235, doi: 10.1016/j.apcata.2018.03.013.

12. Campos-Molina. M. J, Corral-Pérez. J. J, Mariscal. R, Granados. M. L, "Silica-poly(styrenesulphonic acid) nanocomposites as promising acid catalysts", ***Catalysis Today***, Vol. 279, (2017), 155-163, doi: 10.1016/j.cattod.2016.06.042.

13. Jaiboon. V, Yoosuk. B, and Prasassarakich. P, "Amine modified silica xerogel for H2S removal at low temperature", ***Fuel Processing Technology***, Vol. 128, (2014), 276-282, doi: 10.1016/j.fuproc.2014.07.032.

14. Salehi. S, and Anbia. M, "Investigation of Carbon Dioxide Adsorption on Amino-Functionalized Mesoporous Silica", ***International Journal of Engineering, Transactions C: Aspects***, Vol. 28, No. 6, (2015), 848-854 doi: 10.5829/idosi.ije.2015.28.06c.04.

15. Castillo. X, Pizarro. J, and Ortiz. C, "A cheap mesoporous silica from fly ash as an outstanding adsorbent for sulfate in water", ***Microporous and Mesoporous Materials***, Vol. 272, (2018), 184-192, doi: 10.1016/j.micromeso.2018.06.014.

16. Scherdel. C, and Reichenauer. G, "Highly porous silica xerogels without surface modification", ***The Journal of Supercritical Fluids***, Vol. 106, (2015), 160-166, doi: 10.1016/j.supflu.2015.08.016.

17. Echeverría. J. C, Estella. J, Barbería. V, Musgo. J, and Garrido. J. J, "Synthesis and characterization of ultramicroporous silica xerogels", ***Journal of Non-Crystalline Solids***, Vol. 356, No. 6-8, (2010), 378-382, doi: 10.1016/j.jnoncrysol.2009.11.044.

18. Adhikari. C, Mishra. A, Nayak. D, Chakraborty. A, "Drug delivery system composed of mesoporous silica and hollow mesoporous silica nanospheres for chemotherapeutic drug delivery", ***Journal of Drug Delivery Science and Technology***, Vol. 45, (2018), 303-314, doi: 10.1016/j.jddst.2018.03.020.

19. de Lima. H. H. C, Kupfer. V. L, Moisés. M. P, Rinaldi. J. C, Felisbino. S. L, Rubira. A. F, and Rinaldi. A. W, "Bionanocomposites based on mesoporous silica and alginate for enhanced drug delivery", ***Carbohydrate Polymers***, Vol. 196, (2018), 126-134, doi: 10.1016/j.carbpol.2018.04.107.

20. Vali. K. S, Murugan. B. S, Reddy. S. K, Farsangi. E. N, "Eco-friendly hybrid concrete using pozzolanic binder and glass fibers", ***International Journal of Engineering Transactions A : Basics***, Vol. 33, No. 7, (2020), 1183-1191, doi: 10.5829/ije.2020.33.07a.03.

21. Yakushkin. S. S, Balaev. D. A, Dubrovskiy. A. A, Semenov. S. V, Knyazev. Y. V, Bayukov. O. A, Kirillov. V. L, Ivantsov. R. D, Edelman. I. S, and Martyanova. O. N, "ε-Fe_2O_3 nanoparticles embedded in silica xerogel - Magnetic metamaterial", ***Ceramics International***, Vol. 44, No. 15, (2018), 17852-17857, doi: 10.1016/j.ceramint.2018.06.254.

22. Saputra. R. E, Astuti. Y, and Darmawan. A, "Hydrophobicity of silica thin films: The deconvolution and interpretation by Fourier-transform infrared spectroscopy", ***Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy***, Vol. 199, (2018), 12-20, doi: 10.1016/j.saa.2018.03.037.

23. Nuryono and Narsito, "Pengaruh Konsentrasi Asam Terhadap Karakter Silika Gel Hasil Sintesis dari Natrium Silikat", ***Indonesian Journal of Chemistry***, Vol. 5, No. 1, (2005), 23-30.

24. Guo. Q, Huang. D, Kou. X, Cao. W, Li. L, Ge. L, and Li. J,"Synthesis of disperse amorphous SiO_2 nanoparticles via sol-gel process," ***Ceramics International***, Vol. 43, No. 1, (2017), 192-196, doi: 10.1016/j.ceramint.2016.09.133.

25. Munasir, Triwikantoro, Zainuri. M, and Darminto, "Synthesis of SiO_2 nanopowders containing quartz and cristobalite phases from silica sands" , ***Materials Science- Poland***, Vol. 33, No. 1, (2015), 47-55, doi: 10.1515/msp-2015-0008.

26. Mujiyanti. D. R, Nuryono, and Kunart. E. S, "Sintesis dan karakterisasi silika gel dari abu sekam padi yang diimobilisasi dengan 3-(trimetoksisilil)-1-propantiol," ***Sains dan Terapan Kimia***, Vol. 4, (2010), 150-167

27. Utami. W. S, Herdianita. N. R, and Atmaja. R. W,"The Effect of Temperature and pH on the Formation of Silica Scaling of Dieng Geothermal Field, Central Java, Indonesia," ***Thirty-Ninth Workshop on Geothermal Reservoir Engineering Stanford University***, (2014), 24-26.

28. Lion. M, Maache. M, Lavalley. J. C, Ramis. G, Busca. G, Rossi. P. F, and Lorenzelli. V, "FT-IR study of the Brønsted acidity of phosphated and sulphated silica catalysts", ***Journal of Molecular Structure***, Vol. 218, (1990), 417-422.

29. Muljani. S, Setyawan. H, Wibawa. G and Altway. A, "A facile method for the production of high-surface-area mesoporous silica gels from geothermal sludge", ***Advanced Powder Technology***, Vol. 25, No. 5, (2014), 1593-1599. doi: 10.1016/j.apt.2014.05.012.

30. Jung. I. K, Gurav. J. L, Bangi. U. K. H, Baek. S, Park. H. H, "Silica xerogel films hybridized with carbon nanotubes by single step sol-gel processing", ***Journal of Non-Crystalline Solids***, Vol. 358, No. 3, (2012), 550-556, doi: 10.1016/j.jnoncrysol.2011.11.009.

31. Witoon. T, Tatan. N, Rattanavichian. P, Chareonpanich. M, "Preparation of silica xerogel with high silanol content from sodium silicate and its application as CO_2 adsorbent", ***Ceramics International***, Vol. 37, No. 7, (2011), 2297-2303, doi: 10.1016/j.ceramint.2011.03.020.

32. Lee. J, Kim. J, Lee. B. J, Lee. J, Lee. H. W, Hong. M, Park. H, Shim. D. I, Cho. H. H, Kwon. K, "Characterization of mesoporous silica thin films for application to thermal isolation layer", ***Thin Solid Films***, Vol. 660, (2018), 715-719, doi: 10.1016/j.tsf.2018.04.001.

33. Zhuang. Q, and Miller. J. M, “One-pot sol-gel synthesis of sulfated ZrO2-SiO_2 catalysts for alcohol dehydration,” ***Canadian Journal of Chemistry***, Vol. 79, No. 8, (2001), 1220-1223, doi: 10.1139/cjc-79-8-1220.

34. Bleam. W. F, “Adsorption and Surface Chemistry”, ***Soil and Environmental Chemistry***, (2012), 371-407, doi: 10.1016/b978-0-12-415797-2.00009-1.

35. Liu. C, and Komarneni. S, “Carbon-silica xerogel and aerogel composites”, ***Journal of Porous Materials***, Vol. 1, No. 1, (1995), 75-84, doi: 10.1007/BF00486526.

36. Mosquera. M, J, Santos. D, d. los. M, Valdez-Castro. L, Esquivias. L, “New route for producing crack-free xerogels: Obtaining uniform pore size”, ***Journal of Non-Crystalline Solids***, Vol. 354, No. 2-9, (2008), 645-650, doi: 10.1016/j.jnoncrysol.2007.07.095.

37. Yu. H, Tong. Z, Yue. S, Li. X, Su. D, and Ji. H, “Effect of SiO_2 deposition on thermal stability of Al_2O_3-SiO_2 aerogel,” ***Journal of the European Ceramic Society***, Vol. 41, No. 1, (2021), 580-589, doi: 10.1016/j.jeurceramsoc.2020.09.015.

Persian Abstract

چکیده

مقدار زیادی لجن توسط نمک زمین گرمایی در نیروگاه زمین گرمایی Dieng تولید می شود که بیش از ۱۶۵ تن در ماه است. این لجن معمولاً استفاده نمی شود ، مگر اینکه در محل دفن زباله استفاده شود. رسوب (لجن) در درجه اول از سیلیس تشکیل شده است. هدف از این تحقیق سنتز سیلیس مزوپور xerogel (SiO2) از لجن زمین گرمایی (GS) و بررسی اثرات pH به عنوان تلاشی برای بالا بردن ارزش اقتصادی لجن از طریق استخراج قلیایی و به دنبال آن اسیدی شدن است. xerogel SiO2 با استخراج GS تهیه شد تا به سیلیکات سدیم تبدیل شود (Na2SiO3) با کمک NaOH پایه و با استفاده از H2SO4 به عنوان عامل ژل سازی رسوب می کند. تجزیه و تحلیل FTIR از xerogel SiO2 گروهی از سیلانول (Si-OH) و سیلوکسان (Si-O-Si) را نشان داد. تجزیه و تحلیل XRD نشان داد که xerogel SiO2 بی شکل است. علاوه بر این ، با استفاده از آزمون BET (Breneur-Emmet-Teller) از آزمون جذب–دفع نیتروژن مشاهده شد که pH کاهش می یابد و به سطح خاص افزایش می یابد و اندازه منافذ کاهش می یابد. بزرگترین سطح اختصاصی مشاهده شده در SiO2 xerogel تهیه شده با pH 5.5 به ۴۰۰.۱۰ مترمربع در گرم با اندازه منافذ ۴.۵ نانومتر رسید. اندازه منافذ برای همه موارد در محدوده ۴ ~ ۱۲ نانومتر بود ، نشان می دهد که xerogels SiO2 مزوپور بودند. اندازه منافذ سیلیس آماده شده خاصیت پایداری حرارتی نمونه را تحت تأثیر قرار می دهد.