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By Department of Chemistry, Sriwijaya University

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Indonesian Journal of Fundamental and Applied Chemistry (IJFAC) is an an international research journal and invites contributions of original research articles as well as review articles in several areas of chemistry. The journal aims to publish refereed, high-quality research papers with significant novelty and short communications in all branches of chemistry. Papers which describe novel theory and its application to practice are welcome, as are those which illustrate the transfer of techniques from other disciplines.

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Biochemistry field received articles in a range of scientific disciplines, including genetics, microbiology, forensics, plant science and medicine which was studying components like proteins, lipids and organelles.

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The submitted article must have been substantially revised, expanded and rewritten so that it is significantly different from the conference paper or presentation on which it is based. The article must be sufficiently different to make it a new, original work. As a guide, you should aim to have more than 50% new material. This is a matter of judgment and will be based on a comparison of the submitted article with the original conference paper.

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Application of Nanosilica from Rice Husk Ash as Iron Metal (Fe) Adsorbent in Textile Wastewater

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Abstract

Heavy metals are considered hazardous to health if they accumulate excessively in the human body. The adsorption process using adsorbents can remove iron metal ions. In this study, removal of heavy metals such as ferrous metal (Fe) was done by adsorption with nanosilica. Nanosilica is made from rice husk ash contains 80-90% silica so that it can be utilized as a raw material in nanosilica synthesis. Synthesis of nanosilica carried out using a KOH solution with varied concentration (1.5; 2.5; 3; 3.5 and 4 M). The five types of nanosilica applied to textile waste containing ferrous metal (Fe) in five different duration ((5, 10, 15, 20 and 25 minutes). Textile waste containing iron metal that was analyzed by AAS to see the remaining iron content. From the analysis of AAS produced the smallest iron concentration of 0.186 ppm in nanosilica with 1.5 M KOH solvent. Nanosilica with 1.5 M KOH solvent was characterized using a SEM-EDX tool to obtain a surface morphological size of 0.44 µm with 28.95% SiO_2 content.

Keywords: nanosilica, adsorbent, rice husk ash, textile wastewater

Abstrak (Indonesian)

Logam berat dianggap berbahaya bagi kesehatan bila terakumulasi secara berlebihan di dalam tubuh manusia. Ion logam besi dapat dihilangkan dengan proses adsorpsi menggunakan adsorben. Dalam penelitian ini, penghilangan logam berat seperti logam besi (Fe) diadsorpsi dengan menggunakan adsorben berupa nanosilika. Nanosilika dibuat dari abu sekam padi dimana abu sekam padi mengandung 80-90% silika sehingga dapat dimanfaaatkan sebagai bahan baku dalam sintesis nanosilika. Sintesis nanosilika dilakukan menggunakan pelarut berupa KOH dengan variasi konsentrasi 1,5; 2,5; 3,0; 3,5 dan 4,5 M. Kelima jenis nanosilika diaplikasikan terhadap limbah tekstil yang mengandung logam besi (Fe) dengan variasi waktu kontak 5, 10, 15, 20, dan 25 menit. Limbah yang mengandung logam besi yang telah diadsorpsi dianalisa dengan spektrofotometri serapan atom untuk melihat kadar logam besi yang tersisa. Dari analisa AAS dihasilkan konsentrasi besi terkecil yaitu 0,186 ppm pada nanosilika dengan pelarut KOH 1,5 M. Nanosilika dengan pelarut KOH 1,5 M dikarakterisasi dengan menggunakan alat SEM-EDX sehingga diperoleh ukuran morfologi permukaan 0,44 µm dengan kandungan SiO_2 28,95%.

Kata Kunci: nanosilika, adsorben, abu sekam padi, limbah tekstil

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INTRODUCTION

Nanosilica is one of nanotechnology that utilizes silica in nano size. Nanosilica is a nanomaterial that is widely used with applications of adhesive polymers, optical fiber strands, inks, paints, coatings, cosmetics, food additives, and cement-based building materials [1]. The size of silica nanoparticles that have been studied yields a size of 25-60 nm by the co-precipitation method, 13.36 to 50 nm by the sol-gel method [2,3]. Crops such as rice, sugar cane, and wheat have high silica content.

Rice husk is one of the biggest silica-producing sources after complete combustion [4,5]. Combustion of rice husk ash, which is controlled at high

temperatures (500-600 ºC) will produce silica ash that can be used for various chemical processes. Rice husk ash contains 86%-97% dry weight silica [6], from other studies showing that rice husk ash contains 90-98% dry weight silica [7,8]. Ash from burning rice husk has silica content that can reach 91% [9].

The silica content collected with rice straw is much greater than other plants because rice straw contains organic material as follows: cellulose 32-47%, hemicellulose 19.27%, lignin 5-24%, and ash 13-20%. Rice straw ash has 60% silica which, of course, is reported to be different in different climatic conditions, depending on the type of soil, rice cultivation season, weather conditions and geography [10,11]. The advantages of silica made from agricultural rice waste compared to silica obtained from rock deposits (quartz) are as follows. Firstly, silica from rice husks or rice straw adds value to agricultural waste, while silica from rocks causes environmental damage due to deposits. Secondly, silica from Amorphous and reactive rice husk or straw and does not require much energy when transformed to the critical globite structure, making it suitable for starting materials in producing silica. Finally silica obtain directly from husk or straw is a high purity powder and purifying it easily while for obtaining Pure silica powder from quartz rock requires much energy for grinding and refining [12,13].

The increasingly rapid industrial activity today, various types of heavy metal waste produced can be a serious problem for health and the environment. Textile waste is one type of waste that needs attention, because it usually contains heavy metals that are harmful to human life, such as iron (Fe). Along with the times, problems arise related to textile waste. Iron and cobalt metals are toxic metals that can poison the human body and damage the environment. According to the RI KEPMENKES No. 907/MENKES/VII/IV/ 2002 dated 29 July 2002 concerning conditions for the supervision of drinking water quality, the permissible level of Fe in consumption water is 0.3 mg/l [10].

MATERIALS AND METHODS

Materials

The materials used in this study were rice husk, chemical substances such as chloride acid (HCl), potassium hydroxide (KOH), sulfidic acid (H_2SO_4), sodium hydroxide (NaOH), and aquadest.

Methods

To make nanosilica from rice husk ash, 200 gr of rice husk as much as 25 grams was dissolved in 200 ml of KOH with a varied concentration of 1.5 M; 2.5 M; 3.0 M; 3.5 M and 4.5 M. The solution was heated and stirred using a magnetic stirrer at 85 ºC for 3 hours. The solution was cooled at room temperature, filtered and added 1.5 M HCl slowly while stirring until the pH of the solution became 7 and a gel had formed. The solution filtered to obtain pure silica. Pure silica was refluxed using 6 M HCl for 4 hours then washed to acid free using distilled water. Pure silica dissolved in 2.5 M NaOH while stirring using a magnetic stirrer for 6 hours. Concentrated H_2SO_4 was added into pH 8. The precipitate washed using distilled water until it is free of base and then dried in an oven at 60 ºC, crushed with mortar.

Five types of nanosilica with variations in solvent concentration were analyzed using an SEM-EDX (scanning electron microscopy-energy dispersive x-ray) tool to determine the characteristics of the nanosilica. The results of the SEM-EDX analysis are the pore size produced and the composition levels of the elements contained in the nanosilica.

Five types of nanosilica were applied to wastes containing ferrous metal (Fe). The waste used is textile coloring. At the time of application, 1 gram of nanosilica was used in 50 ml of textile waste, with stirring 500 rpm and the variation of contact time was 5 minutes, 10 minutes, 15 minutes, 20 minutes and 25 minutes. Artificial waste adsorbed with nanosilica was analyzed using an AAS (atomic absorption spectrophotometer) to determine the Fe metal content before and after adsorbed with nanosilica.

RESULT AND DISCUSSION

Efficiency and adsorption capacity

The adsorption capacity and absorption efficiency of nanosilica were obtained based on measurements of Fe metal concentrations in textile waste before and after absorption with nanosilica.

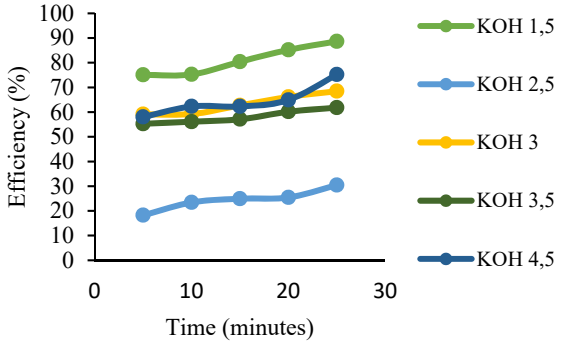

Figure 1. Effect of stirring time to adsorption efficiency

From Figure 1, it can be seen that the longer the contact time of nanosilica, the greater the adsorption

efficiency. This is influenced by the concentration, stirring speed, and time used for contacting the adsorbent with the textile waste, the smaller the concentration of iron metal (Fe) produced from the textile waste, the more the concentration of ferrous metal (Fe) is absorbed by the adsorbent which will increase the value of efficiency.

Nanosilica, which uses 1.5 M KOH solvent, has an increasing value of efficiency from 5 minutes to 25 minutes, with efficiency values successively 75.06%, 75.24%, 80.38%, 85.21% and 88, 63%. This type of nanosilica is the highest efficiency nanosilica. From Figure 2, nanosilica that uses 2.5 M KOH solvent has the lowest efficiency value than the other four types, this is due to low concentrations, lower adsorbent adsorption. The contact time remains the same, which is 5 minutes to 25 minutes, with 18.22%, 23.41%, 24.94%, 25.43%, and 30.44% respectively. The optimum contact time of nanosilica on textile waste occurs at a stirring time of 25 minutes.

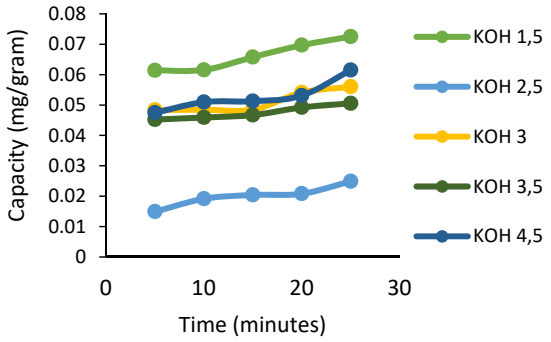

Figure 2. Effect of stirring time to adsorption capacity

From Figure 2, the resulting capacity of each type of nanosilica has increased, the longer the contact time with nanosilica, the greater the adsorption capacity. This is because the longer the contact time of nanosilica to waste, the more iron metal molecules (Fe) collide and interact with the adsorbent so that the adsorption capacity increases over time. The adsorption capacity is directly proportional to the efficiency of its adsorbent ability. Nanosilica made by 1.5 M KOH solvent shows increasing capacity from 5 minutes to 25 minutes. The capacity value are 0.0614 mg/g, 0.06155 mg/g, 0.06575 mg/g, 0.0697 mg/g respectively and 0.0725 mg/g. Nanosilica with 1.5 M KOH solvent is the highest value of nanosilica capacity, while nanosilica with 2.5 M KOH solvent is the lowest value of adsorption capacity. Nanosilica uses 2.5 M KOH solvent, has a contact time of 5-25 minutes, with values of capacity respectively 0.0149 mg/g, 0.01915 mg/g, 0.02040 mg/g, 0.0208 mg/g and 0.0249 mg/g.

From Figures 1 and 2, it can be seen that the greater capacity was produced, the greater the efficiency. Efficiency and capacity values of adsorption indicate the quality of the adsorbent. The greater the efficiency and capacity produced, the better the quality of an adsorbent and vice versa. The highest efficiency and capacity values are found in the type of nanosilica that uses 1.5 M KOH solvent. This type of nanosilica is the best quality type of nanosilica, while nanosilica that uses 2.5 M KOH solvent is the poorest quality type of nanosilica, which has the highest capacity value and lowest efficiency, this is due to the high concentration of solvents, the absorption capacity will be high but the absorption efficiency decreases.

The adsorbent porosity can affect the adsorption of an adsorbent. Adsorbents with large porosity have a higher absorption ability compared to adsorbents that have small porosity [14]. The 1.5 M KOH nanosilica has a higher adsorption power compared to the 2.5 M KOH nanosilica because the formation of greater porosity occurs in the 1.5 M KOH nanosilica solvent. The best optimum condition when using KOH solvent with a concentration of 1.5 M with a stirring time of 25 minutes.

The Langmuir and Freundlich Isotherm Model

Adsorption of the Langmuir and Freundlich Isotherms is calculated for only one of the best types of nanosilica, namely nanosilica which uses a 1.5 M KOH solvent. Nanosilica with 1.5 M KOH solvent is said to be the best, seen from the value of efficiency and capacity. The efficiency and capacity value of nanosilica using 1.5 M KOH solvent is the biggest value.

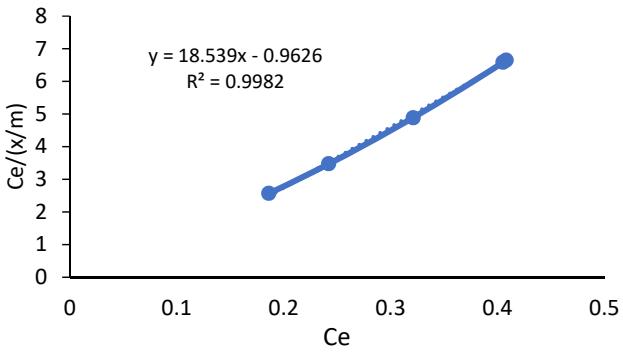

Figure 3. Isoterm model of Langmuir

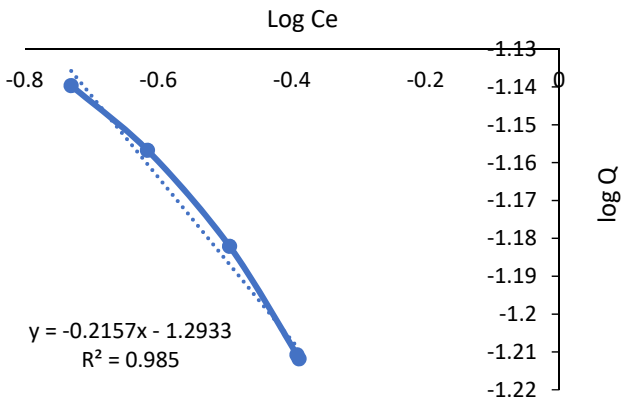

Figure 4. Effect of KOH concentration

Langmuir and Freundlich's theory reveals that the amount of substances adsorbed at a constant temperature by an adsorbent depends on the concentration and activity of the adsorbate to adsorb certain substances. From Figures 3 and 4, we get the equation of the Langmuir isotherm with y = 18.539x-0.9626 and the Freundlich isotherm with y = 0.2157x-1.2933. The R-value on the Langmuir equation obtained 0.9982 is greater than the R-value on the Freundlich equation of 0.985. Based on the value of R obtained, nanosilica using 1.5 M KOH solvent is more likely to use the Langmuir Isotherm adsorption equation because the R-value in the Langmuir equation (Figure 3) is closer to 1 than the Freundlich equation (Figure 4).

Nanosilica with 1.5 M KOH solvent uses the Langmuir isotherm equation, so it can be assumed that the adsorption of iron metal (Fe) that occurs on the surface of the nanosilica is homogeneous and adsorbate adsorbed in a single form (monolayer). Langmuir illustrates that on the surface of the adsorbent, there are a certain number of active sides, which are proportional to the surface area. On each active side, only one molecule can be adsorbed [15-17].

The line equation obtained from the Langmuir isotherm equation is calculated for the a and b values, where 1/b is the intercept, and 1/ab is the slope. The value of a is the Langmuir equilibrium constant, and b is the constant which shows the maximum number of adsorbed solutes per weight of the adsorbent to obtain the Langmuir constant of 0.05 and the maximum adsorption capacity of 1.03 mg/g.

Nanosilica Characteristic

SEM-EDX analysis is used to determine the differences in the morphology of the surface. The resulting image of nanosilica with a magnification of 5000x and 10.000x using SEM Tescan Vega3 depicted on Figure 5 and 6.

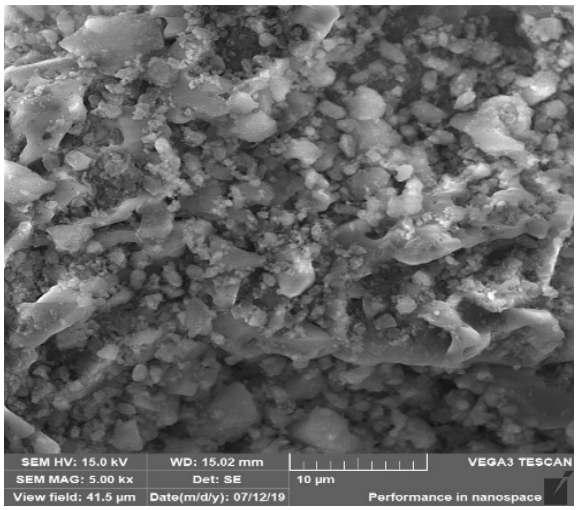

Figure 5. Surface morphology of 1.5M KOH nanosilica magnification of 5000x

It can be seen from Figure 5 that nanosilica already has a dense structure. It can be seen in the magnification of 10000x the size of the porosity obtained by 44 nm is shown in Figure 6. The smaller pore, the greater surface area of the nanosilica obtained. The surface of the sample is uneven, which consists of lumps, and some are spherical, this indicates that nanosilica particle size is quite diverse. The SEM analysis also gives the composition of elements found in nanosilica, Oxygen 49.29%, Silica 28.95%, Carbon 10.95%, Sodium 6.29%, Sulfur 3.56%, and Fluorine 0.51%, listed in Table 1.

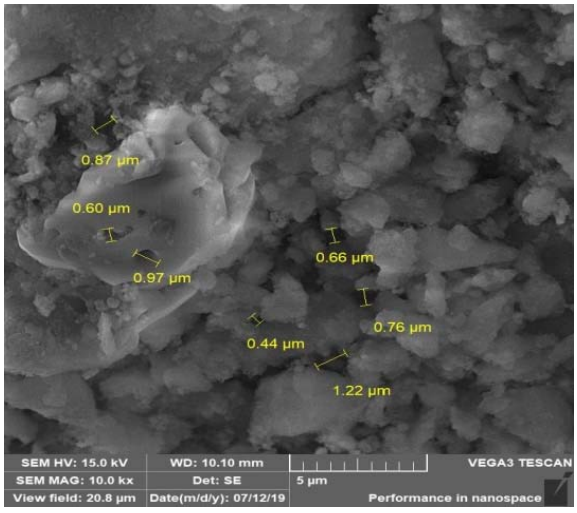

Figure 6. Surface size of 1.5M KOH nanosilica magnification of 10.000x

Table 1. Element Composition of SEM-EDX Analysis results

Element	Mass (%)	Normal Mass (%)
Oxygen	47.23	49.29
Silica	27.74	28.95
Carbon	10.49	10.95
Sodium	6.29	6.57
Sulphur	3.56	3.71
Flourine	0.51	0.53
Total	95.82	100

CONCLUSION

Applyication of nanosilica to textile waste produces the highest adsorption capacity of nanosilica with 1.5 M KOH solvent of 0.073 mg/g with a contact time of 25 minutes. The lowest adsorption capacity value of nanosilica with 2.5 M KOH solvent with a value of 0.015 mg/gr with a contact time of 5 minutes. The result proves that the longer the contact time of nanosilica for textile waste, the greater the adsorption capacity. The highest adsorption efficiency occurred in nanosilica with 1.5 M KOH solvent of 88.63% with a contact time of 25 minutes while the lowest adsorption efficiency value in nanosilica with 2.5 M KOH solvent was 18.215% with a contact time of 5 minutes. This proves that the longer the contact time, the greater the adsorption efficiency.

From the Langmuir and Freundlich isotherm equations, a regression value of approximately 1 is obtained, namely the Langmuir isotherm of 0.9982 with the equation y = 18.539x-0.9626 and the Langmuir constant is 0.05 and the maximum adsorption capacity is 1.03 mg/g.

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