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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 body. One way to remove iron metal ions by adsorption process uses adsorbents. In this study, removal of heavy metals such as ferrous metal (Fe) was adsorbed using an adsorbent in the form of nanosilica. Nanosilica is made from rice husk ash where rice husk ash contains 80-90% silica so that it can be utilized as a raw material in nanosilica synthesis. Synthesis of nanosilica was carried out using a solvent in the form of KOH with a variation of 1.5M concentration; 2.5M; 3.0M; 3.5M and 4.5M. The five types of nanosilica were applied to textile waste containing ferrous metal (Fe) with variations in a contact time of 5 minutes, 10 minutes, 15 minutes, 20 minutes, and 25 minutes. Waste containing iron metal that has been adsorbed was analyzed by atomic absorption spectrophotometry (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 1.5M KOH solvent was characterized using a Scanning electron microscopy (SEM) -Energy Dispersive X-ray (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 waste.

Abstrak (Indonesian)

Logam berat dianggap berbahaya bagi kesehatan bila terakumulasi secara berlebihan di dalam tubuh. Salah satu cara penghilangan ion logam besi 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 dimanfaatkan sebagai bahan baku dalam sintesis nanosilika. Sintesis nanosilika dilakukan menggunakan pelarut berupa KOH dengan variasi konsentrasi 1,5M; 2,5M; 3,0M; 3,5M dan 4,5M. Kelima jenis nanosilika diaplikasikan terhadap limbah tekstil yang mengandung logam besi (Fe) dengan variasi waktu kontak 5 menit, 10 menit, 15 menit, 20 menit, dan 25 menit. Limbah yang mengandung logam besi yang telah diadsorpsi dianalisa dengan spektrofotometri serapan atom (AAS) untuk melihat kadar logam besi yang tersisa. Dari analisa AAS dihasilkan konsentrasi besi terkecil yaitu 0,186 ppm pada nanosilika dengan pelarut KOH 1,5M. Nanosilika dengan pelarut KOH 1,5M dikarakterisasi dengan menggunakan alat *Scanning electron microscopy* (SEM)-*Energy Dispersive X-ray* (EDX) sehingga diperoleh ukuran morfologi permukaan 0,44µm dengan kandungan SiO_2 28,95%.

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

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 coprecipitation 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]. Combustion of rice husk ash, which is controlled at high temperatures (500-600°C) will produce silica ash which can be used for various chemical processes. Rice husk ash contains 86%-97% dry weight silica [5], from other studies showing that rice husk ash contains 90-98% dry weight silica [6]. Ash from burning rice husk has silica content that can reach 91% [7].

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. The advantages of silica made from agricultural rice waste compared to silica obtained from rock deposits (quartz) are (1) silica from rice husks or rice straw adds value to agricultural waste, while silica from rocks causes environmental damage due to deposits, (2) 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, and (3) silica from direct husk or straw can be in the form of high purity powder and purifying it easily while for obtaining Pure silica powder from quartz rock requires much energy for grinding and refining [8].

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 [9].

MATERIALS AND METHODS

Materials

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

Methods

In the process of making nanosilica from rice husk ash, the concentration of the solvent was varied. The solvent used was KOH with a concentration of 1.5M; 2.5M; 3.0M; 3.5M and 4.5M. 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 that has been adsorbed with nanosilica was analyzed using an AAS (atomic absorption spectrophotometer) to determine the Fe metal content before and after adsorbed with nanosilica.

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.

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.

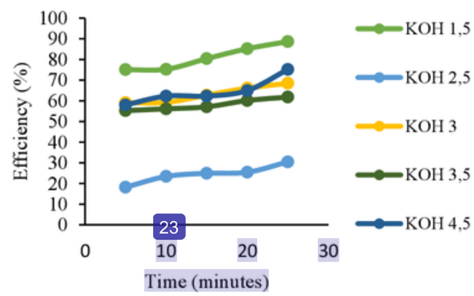

Figure 1. Graph of adsorption efficiency with stirring time

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 1.5M 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 4 types. 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.

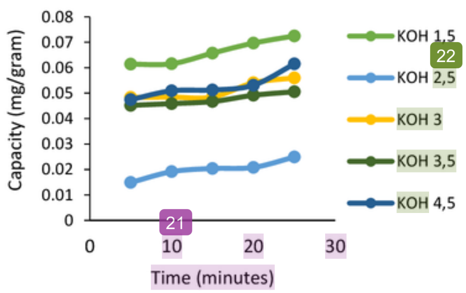

Figure 2. Graph of adsorption capacity to stirring time

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 (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 that uses 1.5M KOH solvent has a value of increasing capacity from 5 minutes to 25 minutes, with the capacity value of 0.0614 mg/g, 0.06155 mg/g, 0.06575 mg/g, 0.0697 mg/g respectively and 0.0725 mg/g. Nanosilica with 1.5M KOH solvent is the highest value of nanosilica capacity, while nanosilica with 2.5M KOH solvent is the lowest value of adsorption capacity. Nanosilica which uses 2.5M 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 the capacity 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, so this type of nanosilica is the best quality type of nanosilica while nanosilica that uses 2.5M KOH solvent is the worst quality type of nanosilica which has the highest capacity value and lowest efficiency.

The adsorbent porosity can affect the adsorption power of an adsorbent. Adsorbents with large porosity have a higher absorption ability compared to adsorbents that have small porosity [10]. The 1.5M KOH nanosilica has a higher adsorption power compared to the 2.5M KOH nanosilica because the formation of greater porosity occurs in the 1.5M 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 1.5M 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.

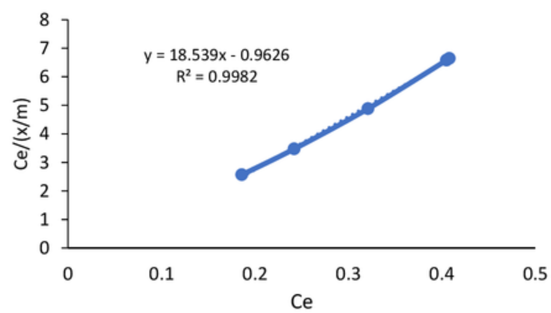

Figure 3. 1.5M KOH Langmuir Isotherm graph

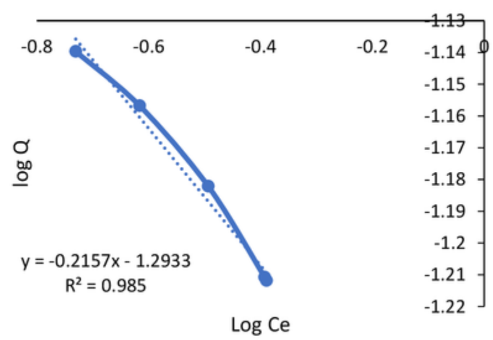

Figure 4. 1.5 M KOH Freundlich isotherm graph

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 1.5M 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, there is only one molecule that can be adsorbed [9].

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/gr.

Nanosilica Characteristic

SEM-EDX analysis is used to determine the differences in the morphology of the surface of nanosilica with a magnification of 5000x and 10,000x using SEM Tescan Vega3.

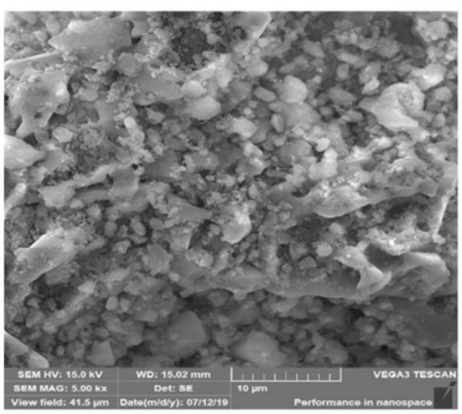

Figure 5. Surface morphology of KOH Nanosilica 1.5M magnification of 5000x

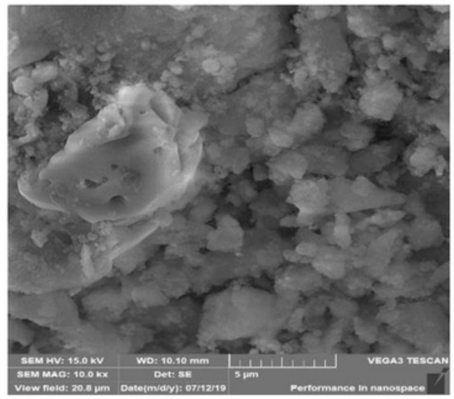

Figure 6. Surface morphology of 1.5M KOH Nanosilica 10,000x magnification

From Figure 5, it can be seen that nanosilica already has a dense structure. Can be seen in the magnification of 10000x the size of the porosity obtained by 44nm is shown in Figure 6. The smaller the pore value obtained in nanosilica results in, 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%.

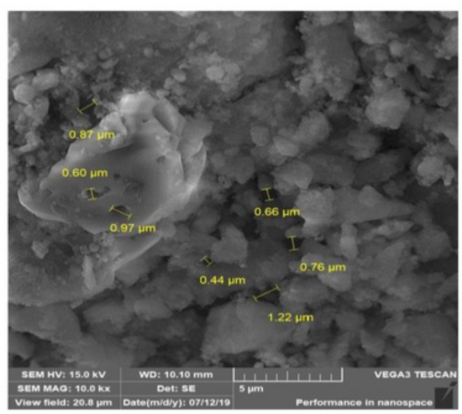

Figure 7. Surface size of 1.5M KOH Nanosilica magnification of 10,000x

The results of this SEM analysis also give the composition of elements contained in nanosilica, namely Oxygen 49.29%, Silica 28.95%, Carbon

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10.95%, Sodium 6.29%, Sulfur 3.56% and Fluorine 0.51% listed in Table 1.

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
Sulfur	3,56	3,71
Flourine	0,51	0,53
Total	95,82	100

CONCLUSION

When applying nanosilica to textile waste, it 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, while the lowest adsorption capacity value of nanosilica with 2.5 M KOH solvent with a value of 0.015 mg/g 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/gr.

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