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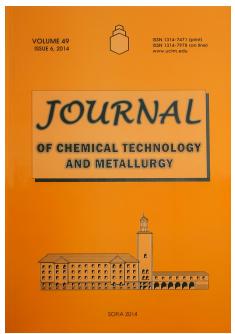
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AN ISOTHERM MODEL OF THE ADSORPTION OF NITRATE IONS ON THE SURFACE OF SILICA FROM SUGARCANE BAGASSE

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ABSTRACT

The abundant sugarcane bagasse from the sugar mills can be a waste that endangers the environment. In fact the sugarcane bagasse contains silica that is useful as an adsorbent. This research is focused on the synthesis of silica from sugarcane bagasse and its application as an adsorbent of nitrate ions in view of the binding process between silica and nitrate ions. The silica from the baggase is characterized by FTIR and XRD. It is compared to Kiesel gel type 60 (Merck). The adsorption data is collected by varying the nitrate ions concentration. The proton stoichiometry (χ) and the amount of the added H^+ or OH^- required to maintain a constant pH value of 7.0 during the adsorption experiments are calculated. All experiments are conducted at a room temperature within an equilibrium time of 30 min. The results obtained are described by the isotherm equations of Langmuir, Freundlich, and Dubinin-Radushkevich. It is found that they fit very well the Freundlich isotherm, which in turn indicates the formation of a multilayer coverage of the silica surface sites by nitrate ions. The energy involved in the adsorption process calculated by the Dubinin-Radushkevich model amounts to 5.61 kJ/mole. The χ value obtained shows that the adsorption process does not involve a significant amount of protons. The three models used in this research provide the conlcusion that the nitrate ions adsorption is a physical one.

<u>Keywords</u>: sugarcane bagasse, adsorption of NO₃, gel silica, physisorption.

INTRODUCTION

The sugarcane bagasse is a solid mixture of residues of the sugarcane processing industry. Most of the sugarcane bagasse is not optimally used. In fact, the sugarcane bagasse contains fiber (cellulose, pentosan, and lignin), ash, and water [1] that can be treated and used as a biomass [2], aerogel silica [3], and an adsorbent [4]. This study is intended to synthesize a gel silica from sugarcane bagasse, and to use it as an adsorbent of nitrate anions.

The synthesis of SiO₂ from sugarcane bagasse is carried out by soaking the bagasse in NaOH solution to produce Na₂SiO₃ solution and adding HCl solution to reach pH of 7 and gel formation [5]. The gel silica is an inorganic solid which has a fair thermal and mechanical

stability. It does not swell in organic solvents [6].

The adsorption of nitrate anions by various adsorbents is reported. The use of modified silica from Lapindo mud is reported by Oktaviana [7]. The adsorption of nitrates contained in in fertilizer industry wastewater on treated zeolite is conducted by Wahyuni [8]. The adsorption of NO₃- by rice husk ash is reported by Widomulyo [9], while that of NO₃- present in wastewater in case of using biomass derived activated carbon is studied by Nunell et al. [10]. However, the use of gel silica in this respect has not been previously reported. The adsorption process can be studied by modeling the experimental data and proton stoichiometry measurement as done by Ikhsan [11] in case of adsorption of aspartic acid by kaolinite.

The nitrates can be a hazardous pollutant that can

endanger the surface and the ground water. For instance, the high concentrations of nitrates in the drinking water may produce nitrosamines which cause cancer and increase the risk of other diseases [12]. The sources of free nitrates in the environment refer to the waste of the abundant usage of nitrogen fertilizers without a careful stoichiometric control [13]. The use of such fertilizers by the farmers in Indonesia is usually higher than that required by the plants, so the excess of free nitrates in the environment cannot be ignored. This results in soil and water pollution, because the anions are highly soluble in water. Nitrates are also wastes of the industries producing fertilizers, food and beverage packaging.

The understanding of the process of nitrates adsorption can be an effort to overcome the abundant hazardous waste of soluble nitrate anions in the ground water. A study of anions adsorption is reported in previous works, such as that of Ikhsan et al. [14] who model the data of Ca(II) and Mg(II) adsorption by sulfonate-modified silica from sugarcane bagasse aiming to determine the process rate. The present investigation is focused on the removal of nitrate ions from the environment and the ground water through their adsorption by sugarcane bagasse silica.

EXPERIMENTAL

Materials

All chemicals used in this research were p.a. grade from Merck. The sugarcane bagasse was collected from Kulon Progo, Yogyakarta, Indonesia. HCl and NaOH were used for the separation of silica and for pH adjustment, while KNO₃ acted as a nitrate (NO₃) source. Distilled water free from minerals was used in this study. The glassware and the tools used included a muffle furnace, a Buchner filter device, a magnetic stirrer and a heater, a shaker, 200 mesh sieve, a mortar and a porcelain pestle. Rigaku Miniflex600 Benchtop and Shimadzu UV-Vis 2450 spectrophotometers were used. The X-Ray Diffraction (XRD) study was carried out with Thermo Nicolet Avatar 360 FTIR.

Methods

The sugarcane bagasse was dried under sun light and then burnt. The ash obtained was further calcinated at 600°C for 5 h and sieved. 20 g of the ash were dissolved in 1 L of 0.1 M HCl. The suspension was stirred with a magnetic stirrer for 2 h, filtered with a Buchner

filter, and washed with distilled water till a neutral reaction. The ash was put in an oven and kept at 80°C until a constant mass was reached. Then it was transferred to a polytetrafluoroethylene oven and was heated with continuous stirring at 90°C for 1 h. After that it was filtered. The filtrate obtained referred to a sodium silicate solution. It reacted with 1 M HCl under stirring until the medium pH reached a constant value of 7.0 and a gel was formed. Filtering with a Buchner funnel was applied to give hydrogel silica as a residue. The latter was washed with distilled water until a neutral pH value and dried in an oven at 80°C. The product obtained was further characterized by XRD study to identify silica crystalline structure and the results obtained were compared to those of Kiesel gel type 60 Merck serving as a standard. FTIR spectroscopy was applied to determine the functional groups present in the synthesized product.

The adsorption experiments were carried out as follows. 1 mL of 0.00715 M NO₃ stock solution was added into 100 mL suspension containing 0.1 g gel silica in distilled water, and stirred continuously with a magnetic stirrer. The system pH value was kept constant at 7.0 by adding H⁺ or OH⁻. The concentrations of H⁺ and OH were carefully noted for the proton stoichiometry measurement. Sample of 1 mL were taken at the equilibrium time of 30 min. The sample was centrifuged. The solution obtained was 100 times diluted. 1 mL of 1 M HCl was added as an indicator. UV-Vis spectroscopy was then applied. The experiments were continued as the above mentioned procedures by adding another volume of 3 mL, 4 mL, 5 mL, 5 mL and 5 mL of the 0.00715 M NO₂ stock solution was added in the adsorption system, respectively.

The other variables such like the temperature, the stirring duration, and the adsorbent mass were kept constant. The temperature of the experiments was kept equal to 25°C during the whole experiment.

RESULTS AND DISCUSSION

The X-Ray Diffraction (XRD) of sugarcane bagasse silica

The X-ray diffraction characterization shows amorphous silica obtained on the ground of sugarcane baggase as illustrated by Fig. 1.

It is evident that the gel silica from the bagasse has a sloping peak at an angle of $2\theta = 21.7^{\circ}$. According to Kalapathy et al. [15], this sloping peak indicates that the bagasse structure is amorphous.

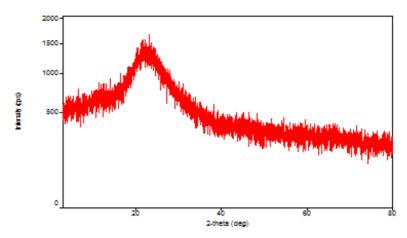


Fig. 1. An XRD diffractogram of gel silica from sugarcane bagasse.

FTIR spectra of sugarcane bagasse silica

Fig. 2 presents the FTIR spectrum of sugarcane bagasse silica compared to that of Kiesel gel type 60 Merck. Similar functional groups are identified in both spectra. This verifies that the synthesis carried out produced standard silica. Table 1 presents the functional groups of sugarcane bagasse silica compared to those of Kiesel gel 60. Table 1 shows that the bagasse silica and Kiesel gel have similar functional groups. The absorption bands of both spectra are at very close wavenumbers. The peak at 3460.21 cm⁻¹ indicates the presence of -OH functional groups of silanol. The wide peaks at 3460.21 cm⁻¹ can be attributed to -OH functional groups of water adsorbed by silica surface through hydrogen

bonds. The absorption bands at 1094.90 cm⁻¹ refer to the asymmetric stretching vibrations of -Si-O groups of siloxane, while the absorption bands at 797.82 cm⁻¹ are connected with the symmetry-shifting vibrations of -Si-O of siloxane. The presence of a band at 964.41 cm⁻¹ indicates the vibration of Si-O in the silanol group. The -OH group bending vibration of water molecules is outlined at 1641.25 cm⁻¹, while the bending vibration of siloxane (Si-O-Si) groups are recorded at 466.63 cm⁻¹ [16]. In general, the functional groups of silica refer to silanol (Si-OH) and siloxane (Si-O-Si). The similarity of the absorption pattern of both samples indicates that the synthesis product on the ground of bagasse refers to silica meeting the standards of the commercial one.

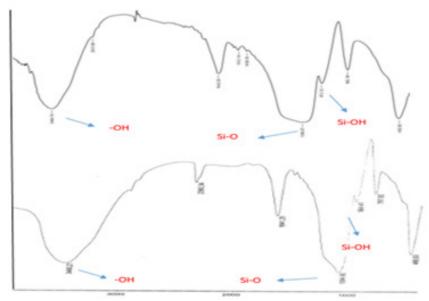


Fig. 2. FTIR spectra of : (a) Kiesel gel 60 silica Merck; (b) gel silica from sugarcane bagasse.

	Wavenumbers (cm ⁻¹)		
Functional Groups	Silica Kiesel Gel 60 Merck	Gel Silica from synthesis	
Stretching vibration –OH from Si-OH	3464.15	3460.21	
Asymmetric stretching vibration of ≡Si-O on ≡Si-O-Si≡	1095.57	1094.90	
Symmetrical stretching vibration of ≡Si-O from ≡Si-O-Si≡	802.39	797.82	
Bending vibration of –OH from water molecules	1643.35	1641.25	
Stretching vibration Si-O from Si-OH	972.12	964.41	
Bending vibration Si-O-Si	462.92	466.63	

Table 1. Silica functional groups derived from the FTIR spectra recorded.

NO₃- adsorption equilibrium by gel silica at various sorbate concentrations

Fig. 3 illustrates the effect of nitrate ions concentration on their adsorption. The graph presents the relationship between the adsorption capacity of the gel silica adsorbent and the equilibrium concentration of NO₃. Fig. 3 shows that the greater equilibrium concentration of NO₃ results in a greater amount of adsorbed NO₃.

Isotherm model for the adsorption of NO₃⁻ by gel silica

The experimental data obtained is modelled using the isotherm models of Langmuir, Freundlich, and Dubinin-Radushkevich. The Langmuir isotherm is presented by Eq.1:

$$\frac{C_e}{q_e} = \frac{1}{K_{L,q_{max}}} + \frac{C_e}{q_{max}} \tag{1}$$

where q_e is the amount of NO_3^- adsorbed by one gram of silica (mol g^{-1}), q_{max} is the monolayer capacity of silica (mol g^{-1}), K_L is the adsorption constant (L mol⁻¹), while C_e is the equilibrium NO_3^- concentration in the solution (mol L⁻¹). The plot of C_e versus C_e/q_e should be linear in accordance with Eq. 1. The experimental data obtained in this study do not fit well the Langmuir equation (Fig. 4) as indicated by very small value of the R, coefficient of correlation of 0.3565. This means that the adsorption of NO_3^- does not result in monolayer coverage of silica surface.

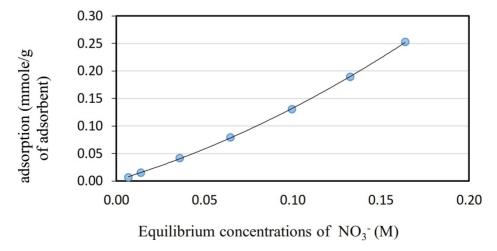


Fig. 3. NO₃ - adsorption by silica from bagasse at various adsorbate concentrations.

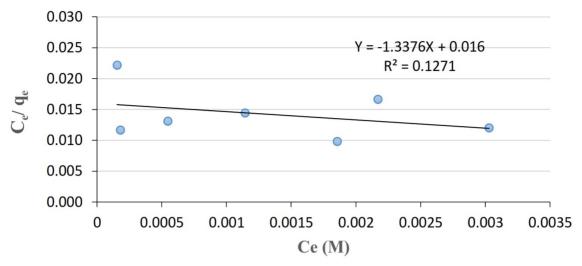


Fig. 4. A poor fit of the experimental data to the Langmuir isotherm model.

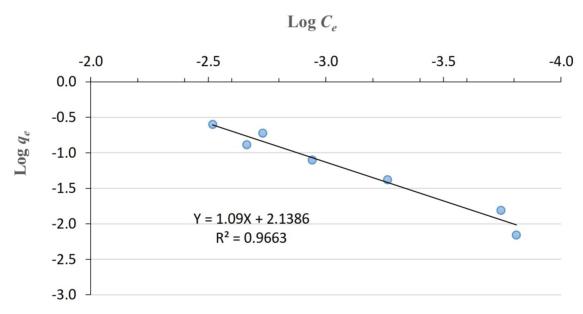


Fig. 5. A good fit of the experimental data to Freundlich isotherm equation.

The fitting of the experimental data to the Freundlich equation (Eq. 2) is carried out in correspondence with Eq. 2:

$$\log q_e = \log K_F + 1/n \log C_e \tag{2}$$

where K_F and n are Freundlich adsorption isotherm constants. An illustration is presented in Fig. 5.

Fig. 5 shows that the calculated line fits very well to the data. The value of the R refers to 0.983. This model indicates that the adsorption of NO₃⁻ by silica tends to follow the assumption of the Freundlich equation, i.e. the surface of silica is heterogeneous, while the adsorption proceeds through the formation of multilayer coverage.

The Dubinin-Radushkevich isotherm model is described by Eq. (3):

$$\ln q_e = \ln q_{max} - \beta \epsilon^2 \tag{3}$$

where ε is the value of [RT ln(1+(1/Ce))], c is a constant related to the adsorption energy (mole²/kJ²), while q_{max} is the maximum adsorption capacity of silica (mole g^{-1}). The plot of ε^2 versus ln q_a (equation 3) should be linear.

The average free energy of adsorption (E) is calculated on the ground of Eq. 4:

$$E = (-2\beta)^{-0.5}$$
 (4)

The value of E is used to determine the type of the

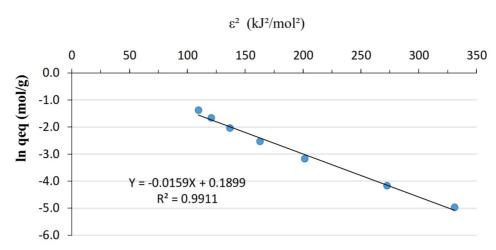


Fig. 6. A good fit of the experimental data to the Dubinin-Radushkevich equation.

adsorption process. It proceeds through a cation exchange when the value obtained is between 8 kJ/mole and 16 kJ/mole, while physisorption takes place when it is less than 8 kJ/mole [17]. The parameters obtained by data modelling are listed in the Table 2. The formation of a complex does not follow the Langmuir model. It corresponds to the isoterm of Freundlich and Dubinin-Radushkevich. The model fit is verified by the correlation coefficient values obtained. They refer to 0.983 and 0.996 in case of Freundlich and Dubinin-Radushkevich models, correspondingly (Table 2).

The value of n from the Freundlich isotherm equation indicates the adsorption intensity of the adsorbate in respect to the adsorbent. A greater than 1 shows that the adsorption runs perfectly. Raji and Anirudhan [18] report their finding that the value of n higher than 1.00 indicates that the adsorption of Cr (VI) by polyacrylamide proceeds through an anion exchange. The present study findas a value of n lower than 1.00, i.e. 0.917.

This implicates that the bonds on silica surface are weak. However, it can not be stated that these bonds results from a physical adsorption. The analysis carried out with the application of the Dubinin-Radushkevich isotherm model provides finding the answer of this question. The energy value found with this equation application amounts to 5.607 kJ mol⁻¹. It indicates that the bonds between the nitrate anions and the gel silica can be attributed to physisorption [19]. The measurement of proton stoichiometry referring to the number of the protons (H⁺ ion) consumed or released by the system during the adsorption of NO₃ is conducted at pH of 7.00. The value obtained is also used as a verification of the adsorption model investigated [20]. The proton stoichiometry found in this investigation is very small (3.9 x 10⁻⁹ mol of proton/mol of nitrate adsorbed). It supports the fact that the adsorption studied proceeds predominantly through a mass transfer which did not involve a significant number of protons.

Table 2. Isotherm adsorption parameters.

Isotherm adsorption parameters	Langmuir	Freundlich	Dubinin-Radushkevich
q_{max} (mole/g)	-0.747	-	1.209
K_L (L/mole)	-83.600	-	-
$K_F(L/g)$	-	0.0137	-
1/n	-	1.090	-
N	-	0.917	-
\mathbb{R}^2	0.357	0.983	0.996
R	0.597	0.991	0.998
E (kJ/mole)	-	-	5.607

CONCLUSIONS

The silica obtained from sugarcane bagasse is amorphous and contains siloxane and silanol groups. The adsorption experimental data fits very well to the Freundlich isotherm equation indicating the formation of multilayer coverage on the silica surface. The low value of the proton stoichimetry (χ) indicates that adsorption process does not involve many protons. The energy involved in the adsorption process is calculated on the ground of the Dubinin-Radushkevich model. It is found equal to 5.61 kJ/mole verifying that physical adsorption of nitrate ions proceeds on the surface of sugarcane bagasse silica.

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