Artikel 10

by Aster Rahayu

SYNTHESIS OF SILICA-PEG BY PHYSICALLY COATING WITH PEG 4000 FOR ABSORPTION OF ION NITRATE

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Abstract: All production activities will eventually produce waste, especially liquid waste. The nitrate ion (NO_3^-) is one of the substances in liquid waste that can have a significant impact on water systems and human health. Several methods can be used to treat the nitrate contained in the water, such as adsorption, phytoremediation, and wetland treatment. One of its methods was adsorption. The silica was modified with polymer due to its huge carbon chain. This research was focused on optimizing the ability of silica-based materials using PEG by physically coating them since PEG has a long carbon chain that could be transformed into a helix. In this research, the adsorbent will be prepared from modified silica gel with PEG 4000 by physical coating. Several parameters will be observed, including the effect of coating time, silica-PEG ratio, and PEG concentration. The capacity of silica-PEG to absorb NO_3^- is being studied. The characterization of functional group content in silica-PEG was carried out by Fourier Transform Infrared (FTIR). The study results show that the coating time was 10 minutes with the ratio of silica to PEG of 1:5, and the concentration of PEG used was 15%, indicating the optimum performance of silica-PEG when used as an absorbent, which achieved 84.44 % nitrate removal. An increase in the absorption capacity of silica-PEG demonstrates the ability of silica-PEG as a porous material. It was compared to silica gel without the PEG coating process, which was 9.09% to 84.44%.
Keywords: *Adsorption; Nitrates; PEGs; physically coating.*

Abstrak: Semua kegiatan produksi pada akhirnya akan menghasilkan limbah, terutama limbah cair. Limbah cair biasanya akan bermuara di sungai dan berakhir di laut. Salah satu zat dalam limbah cair adalah ion nitrat (NO_3^-) yang mana dalam jumlah banyak dapat mengganggu sistem perairan dan kesehatan. Beberapa metode dapat digunakan untuk mengolah nitrat yang terkandung dalam air, seperti adsorpsi. Salah satunya dimodifikasi dengan polimer karena memiliki rantai karbon yang besar. Penelitian ini dikonsentrasikan untuk menyelidiki optimalisasi kemampuan silika dengan PEG dengan pelapisan fisik karena PEG memiliki rantai karbon panjang yang dapat bertransformasi menjadi heliks. Dalam penelitian ini, adsorben akan dibuat dari silika gel termodifikasi dengan PEG 4000 dengan pelapisan fisik. Beberapa parameter yang akan diamati antara lain pengaruh waktu pelapisan, rasio silika-PEG, konsentrasi PEG, dan temperatur pelapisan. Kinerja silika-PEG untuk menyerap NO_3^- diselidiki.

Karakterisasi kandungan gugus fungsi pada silika-PEG dilakukan dengan Fourier Transform InfraRed (FTIR). Hasil penelitian menunjukkan bahwa waktu pelapisan adalah 10 menit dengan perbandingan silika dan PEG 1:5, dan konsentrasi PEG yang digunakan adalah 15%, menunjukkan kinerja silika-PEG yang optimum bila digunakan sebagai penyerap NO_3^- sebesar 84,44%. Kemampuan silika-PEG sebagai bahan berpori dibuktikan dengan peningkatan kapasitas penyerapan silika-PEG dibandingkan silika gel tanpa proses pelapisan PEG yaitu 9,09% menjadi 84,44%.
Kata kunci: Adsorpsi; Nitrat; PEGs; pelapisan fisik.

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Introduction

Indonesia is one of the industrial countries, contributing to more than 20% of the Indonesian economy. It makes Indonesia the top 10 in the world in the category of manufacturing value-added. The rapid development of industry in Indonesia, cannot be separated from the problem of environmental pollution, one of which is water pollution (Hamzah, 2021). With increasing industrial growth in Indonesia, it has the potential to negatively impact ecological quality, especially water, due to the imperfect processing of liquid waste produced by an industry. There is reinforced based on data collected from the Indonesian statistical agency in the environmental field, the level of water pollution in Indonesia has increased by 10% in recent years (Luh, 2017). Physical, chemical, and biological parameters become the reference for determining water quality. The physical parameters include the value of turbidity, temperature, color, discharge, Total Dissolved Solid (TDS), and Total Suspended Solid (TSS). The chemical parameters include BOD, COD, DO, pH, nitrate content, nitrite, phosphate, and heavy metals (Hamzah, 2021). As for biology, the range of microorganisms is contained. The parameter of concern in testing water quality is the nitrite content. Increasing nitrate content in water can result in increased nutrient content in water. These nutrients are beneficial for the survival of aquatic biotas such as algae and bacteria (Deswati et al., 2020). At a specific value limit, the presence of these nutrients will not have an effect, but at a high enough concentration, it will cause changes in function due to the eutrophication process.

One of the efforts to deal with water pollution due to industrial waste in Indonesia can be made with the adsorption method. The use of silica as a nitrate ion adsorbent in wastewater has been widely carried out. The use of silica is based on its properties which have a distinctive surface area and large pores. Several studies have been carried out to obtain silica as an adsorbent, heavy metal with reasonable effectiveness, such as silica to absorb heavy metal waste iron (Fe) (Kukwa et al., 2020), the maximum percentage removal of metal ions reached 99.48%. Silica has also been successfully used as an adsorbent to reduce Pb^{2+}

(Naat et al., 2021), Mn^{2+} (Darjito et al., 2019), Cd (Indriyani et al., 2019), Cr^{3+} (Choi, 2018; Rodiah, 2021), Mg^{2+} (Fahmiati et al., 2010) and Cu^{2+} (Fathurrahman, 2020; Naat, 2021), wastewater and nitrate levels in drinking water (Erdoo et al., 2020). Silica, a silicon oxide with a chemical formula (SiO_2), is an oxide that does not have an active group. Several studies reported silica-based optimization for adsorbing heavy metals, such as using the sulfonate functional group (Fahmiati et al., 2010). However, less reported optimizing the potential of silica with polymer, specifically polyethylene glycol (PEG), by physical coating (Onn et al., 2020; Suhono et al., 2019) and chemically (Niizeki et al., 2016). As a polymer, PEG has a primary hydroxyl group at the end of the polyether chain containing oxyethylene. It can form a double helix with intermolecular interactions (Catauro et al., 2018). The functional groups in PEG can provide several other interactions, such as hydrogen bonding and dipole-dipole interactions (Niizeki et al., 2016). This study aims to increase the effectiveness of the silica-based by using a physically coating method with PEG in a single and simple step. The absorption ability of silica PEG coating will be used as an adsorbent for the absorption of nitrate ions (NO_3^-). The ability of silica-PEG will be proven by comparing it with silica gel without going through a coating process for the absorption of nitrate ions.

Material And Methods

Materials

The materials used in this study included silica gel, PEG 4000 (Nacalai Tesque, Kyoto, Japan), $NaNO_3$ (Nacalai Tesque, Kyoto, Japan), Brucine Sulfate (Nacalai Tesque, Kyoto, Japan), Ethanol (Wako 1st Grade), Sulfuric Acid (Wako 1st Grade), NaCl (Nacalai Tesque, Kyoto, Japan) and Aquadest.

Apparatus

The detection of NO_3^- in terms of concentration, Spectrophotometry UV-Vis (Thermo Scientific-Genesys) was carried out. Furthermore, identifying the functional group in the polymer was carried out using Fourier Transform InfraRed (FTIR) Spectrum 400 Series Perkin Elmer.

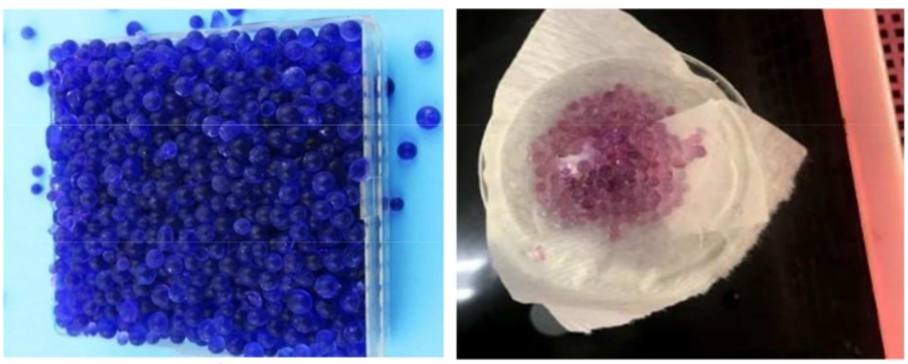
Synthesis of Silica-PEG

Silica gel was modified with PEG and was carried out by weighing 3 grams of PEG 4000, dissolved in 20 ml of 98% ethanol, and then mixed with 5 grams of silica. The coating was carried out with a stirrer for a predetermined time. Then, the separated silica with PEG for removing the residual solution. The performance of Silica-PEG was investigated to absorb NO_3^- using UV-Vis spectrophotometry based on Standard Methods for the Examination of Water and Waste Water Indonesia (SNI 06-6989.9-2004).

Discussion

Modification and characterization of Silica-PEG 4000

Modification of silica gel using PEG 4000 through the method of physical coating. It was conducted by observing several factors that affect the silica modification process. It was started by coating the silica gel with PEG 4000, such as the contact time factor between PEG 4000 and silica, the ratio between silica and PEG 4000, the concentration of PEG 4000 used, and a temperature ongoing physical coating. Afterwards, the effectiveness of silica-PEG 4000 will be observed. The initial test of the performance of silica-PEG 4000 was by adsorbing the ion 15 ppm of NO_3^-. The coating process lasts only on surface silica. Physically coating occurs based on the Van Der Waals Force interface between the silica and PEG surfaces. Silica has site active group silanol (Si-OH) and siloxane (Si-O-Si), a cluster -OH on the surface active site of silica gel with low polarizability. According to (An et al., 2020), PEGylated on the surface of silica microparticles would improve the ability of silica microparticles for controlled pulmonary drug delivery. The presence of polarizability dramatically affects the movement or displacement of electrons on the silica surface. In theory, a vast surface of nano-silica interacts with the polymer chains, thereby reducing the freedom of movement of the PEG 4000 polymer chains (Linda et al., 2021). Visually there is a colour change in the silica gel used, shown in Figure 1.



A B

Figure 1. Visual silica gel. A. Silica gel before the coating process with PEG. B. Silica gel after processing coating with PEG.

The silica-PEG was characterized using Fourier Transform Infra-Red (FTIR) to determine and confirm the type of bonding of the organic compound groups formed on Si-PEG 4000. FTIR characterization was carried out using the mid-infrared wavelength range, which is 4000-400 cm^{-1}, as shown in Figure 2. The characteristic peaks of silica-based (Si-OH) just consist of Si-O-Si stretching that indicated around 1130-800 cm^{-1}. Besides that, silica-PEG appeared to peak from 3000 to 3700 cm^{-1}. Specifically, the broad peak at 3250 cm^{-1} and 1647.21 cm^{-1} were assigned to the –OH stretching vibrations of silica coating PEG (Saravanan et al., 2020). The bounding of silica and PEG was indicated by the dipole-dipole interactions (Linda et al., 2021). The ions between the atoms of oxygen from the long chain in PEG and silica coordinated and formed a double helix. Hence acts as an active site of the ion exchange at a wavelength of 3250 cm^{-1} and is identified as an alcohol group (-OH). Thus the wavelength of 1647.27 was determined as a -OH group attached to silica. The silica itself was detected at 628 cm^{-1} as silica gel. The strong bands at 1076,28 cm^{-1} and 337.54 cm^{-1} were associated with the asymmetric and symmetric Si-O-Si stretching vibration bonding (Shaiful et al., 2014). Furthermore, the wavelength of 1647.27 was determined as C=C bounding and 1215.15 cm^{-1} was detected as C-C bounding from PEG (Tran et al., 2013). The spectrum that appears is adjusted concerning the wavelength of the infrared spectrum, as shown in Table 1

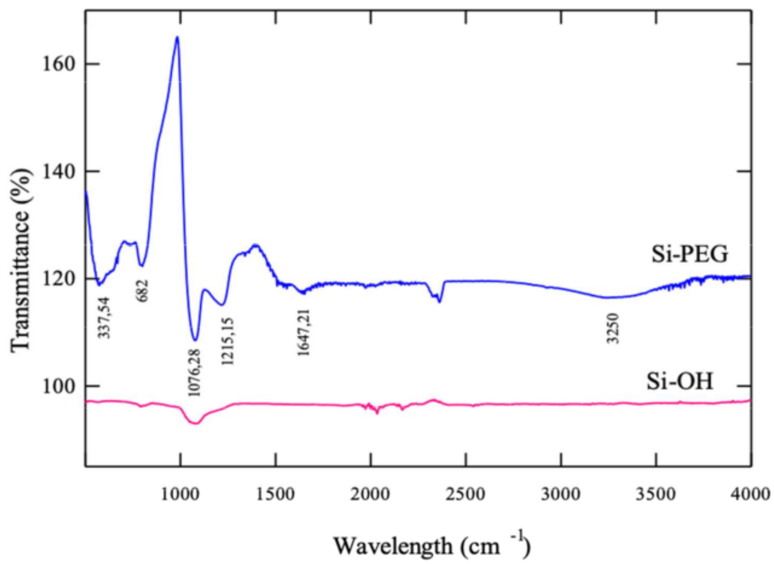


Figure 2. Fourier Transform Infrared (FTIR) spectrum of Si-PEG 4000.

Table 1. Comparison of IR spectrum wavelengths between references with Silica-PEG

Type Bond	**Wavelength (cm^{-1})**	
	Reference (Saravanan et al., 2020)	**Silica-PEG**
O-H ***stretching***	3000-3700	3250
C-**C** ***stretching***	1500-800	1215.15
C=C stretching	1650-1900	1647.27
Si-O-Si ***asym.stretching***	1130-800	1076.28; 628; 337.54

The effectiveness of Silica-PEG 4000 for nitrate adsorption

Several things affect the silica modification process with PEG as an adsorbent in terms of coating time, silica-PEG ratio and PEG concentration. Each of these parameters affects the performance of silica-PEG in NO_3^- absorption. The effect of coating time on the effectiveness of silica-PEG was carried out on the absorption of anions. The anion used is NO_3^- ion with a concentration of 15 ppm. Absorption was carried out for 10 minutes at room temperature. The absorption results can be seen in Figure 3. It can be seen that the longer the coating time there was decreased the effectiveness of the silica-PEG, which was indicated by an increase in the removal of nitrate (%). These can be caused by a fall layer of PEG that coats the silica surface due to prolonged contact between silica and PEG. The interaction between silica and PEG for 10 minutes in this coating time parameter shows a better capacity value than the other coating times. Due to weak silanol and siloxane bonds on silica gel, the removal of nitrate increases from 10 minutes with an adsorption capacity, and nitrate removal is 0.22 mg/g and 29.8 %, sequentially. Then the coating time for adsorption of NO_3^- will be accrued at 10 minutes. This is in line with research on preparing tetraethyl orthosilicate (TEOS) and polyethylene glycol (PEG). Methanol (CH_3OH) was used as the solvent and nitric acid (HNO_3) as the catalyst. TEOS was first partially hydrolysed with deionized water, methanol, and PEG for 10 minutes (Suzaimi et al., 2019).

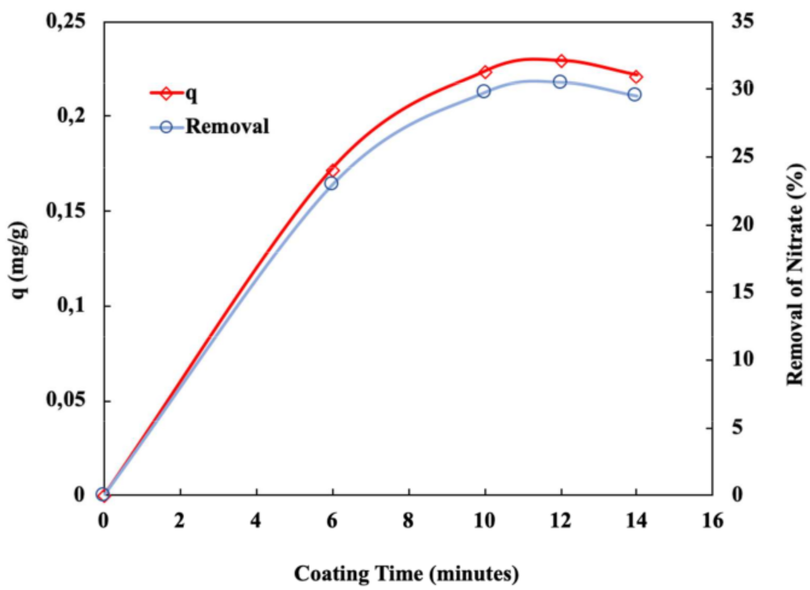


Figure 3. Effect of coating time on silica-PEG in absorption NO_3^- 15 ppm at room temperature.

The ratio weight between silica and PEG was observed to investigate the heavy influence of silica-PEG's effectiveness. It was conducted with 1:3; 1:5; 1:7; 1;8 (w:w) of variation. Figure 4 shows the effect of the ratio silica-PEG on the absorption of NO_3^- 15 ppm in a temperature room. There is a difference of approximately 5% between each observed ratio variation. This matter caused an existing difference in the interaction between PEG and NO_3^-. Increasing the amount of PEG potentially introduces more carbon chains, Carbon chains that could transfer to double helix with dipole-dipole interaction with a silanol. due to this potential, it will improve the ability of silica-PEG to adsorb the nitrate ion. Nitrate ions as anions will be trapped in double helix cavities (Indriyani et al., 2019; Linda et al., 2021). The ratio Optimum coating process silica-PEG is at 1:5 with adsorption capacity, and nitrate removal is 0.33 mg/g and 44.2 % sequentially.

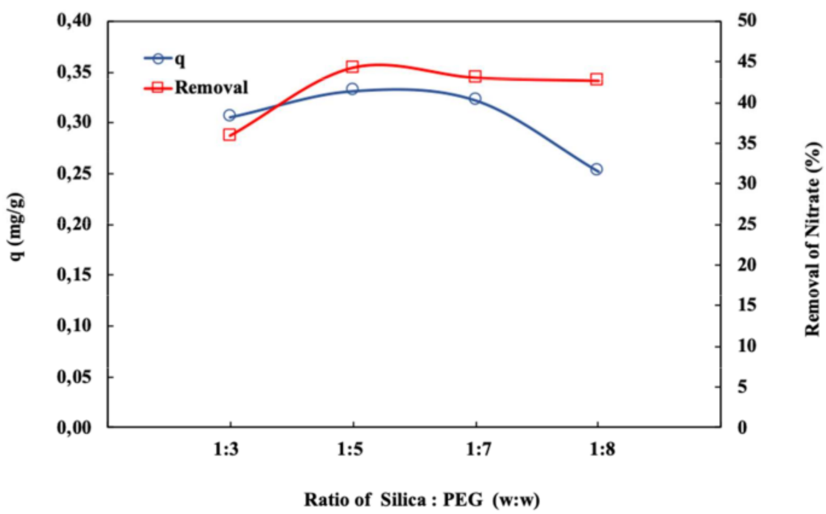


Figure 4. Effect of ratio of silica: PEG on effectiveness silica-PEG in absorption NO_3^- 15 ppm at room temperature

The effect of variations in the concentration of PEG used in the silica coating process can be seen in Figure 5. It can be seen that there is an actual significant difference using PEG 15% compared to the others. While using 10 % of PEG as started point, it was shown the lowest value of capacity and percentage of nitrate removal: 0.58 mg/g and 78.42 %, sequentially. The high concentration of PEG can accelerate the saturation of the active groups of silica and slow down the adsorption process. Meanwhile, if the concentration is too low, the functional group that acts as the active site in the NO_3^- absorption process will not be maximally located on the silica surface (Ahmadi, 2019; Zhu, 2018). Based on Figure 5, the optimum concentration of PEG is at 15 %, with the capacity value and percentage removal of nitrate reaching 0.63 mg/g and 84.44 %, sequentially.

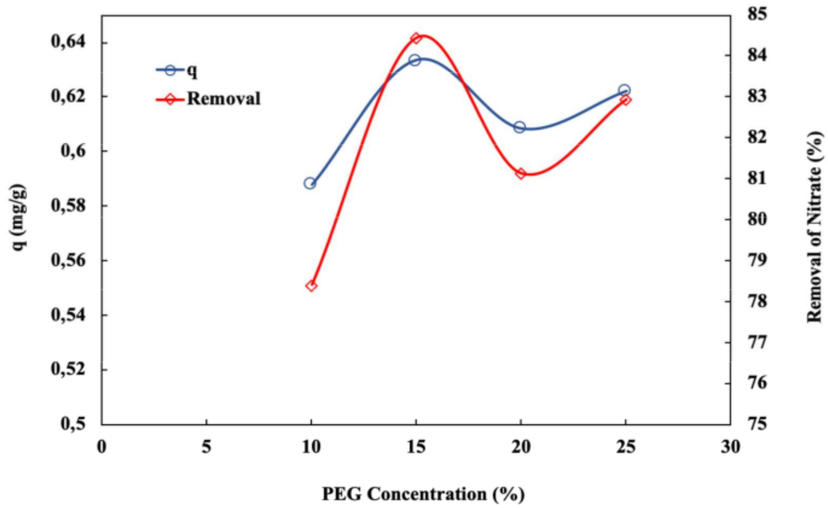


Figure 5. Effect of PEG concentration on effectiveness silica-PEG in absorption NO_3^- 15 ppm at room temperature.

Elongation at break is the ratio between increased length and initial length after breakage of the tested specimen at a controlled temperature. In general, the combination of ultimate tensile strength and elongation at break can be a good estimation of the toughness of a polymeric material (Ahmadi-Khaneghah et al., 2019). Figure 5 shows the effect of the silica-PEG ratio on nitrate removal. There is a difference in the interaction between PEG and NO_3^- for each variety that affected the percentage of nitrates removal. The amount of PEG has the potential to introduce more carbon chains that can move to the double helix by dipole-dipole interactions with silanols. because of this potential, it will increase the ability of silica-PEG to adsorb nitrate ions. Nitrate ions as anions will be trapped in the cavity of the double helix (Linda et al., 2021; Tran et al., 2013).

Comparison of the effectiveness of Silica before and after modification with PEG 4000 for nitrate adsorption

Silica has a silanol group that is the potential to introduce and be attached to other functional groups according to the purpose of application. Firstly the carbonyl group from PEG could form a double helix and act as an active site for adsorbing ion nitrate. Since the silica-based could work as an absorber for nitrate removal, silica-based effectiveness will be observed with and without attaching any PEG groups. Table 2 shows the comparison of capacity value and percentage removal of nitrate. it can be seen that the percentage removal of nitrate significantly improves the adsorbent's performance by almost ten times, from 9.09 % to 84.45 %. In tune with the percentage of removal nitrate, attaching the PEG group to the silica-based surface also improves the capacity value from 0.07 mg/g to 0.63 mg/g. It could be concluded that modifying using PEG by the physical coating of silica-based improves the performance removal of nitrate.

Table 2. Comparison of Silica with and without attaching PEG groups.

Material	Q (mg/g)	Removal of Nitrate (%)
Silica-based	0.07	9.09
Silica-PEG	0.63	84.45

Conclusions

Modifying silica with PEG in this study showed an increase in the performance and activity of silica that is seen from the value of anion absorption capacity and percentage of NO_3^- removal. the attachment of PEG groups into the surface of silica as an adsorbent improves the performance for nitrate removal almost ten times in terms of capacity value (q) and removal percentage. The optimum condition for the coating process was at 10 minutes of coating time, the ratio of silica: PEG was 1:5, and the concentration of PEG was 15%, achieving 84.44% of nitrate removal. Silica-PEG has the potential to be used as an alternative adsorbent for the absorption of NO_3^- ions and optimizing the removal of NO_3^- from the outlet of secondary treatment with the following investigation.

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