ISSN: 1410-8917
Jurnal Kimia Sains & Aplikasi
e-ISSN: 2597-9914

Jurnal Kimia Sains dan Aplikasi 23 (11) (2020): 396–402
Jurnal Kimia Sains dan Aplikasi
Journal of Scientific and Applied Chemistry
Journal homepage: http://ejournal.undip.ac.id/index.php/ksa

Synthesis of Sodium Lauryl Sulfate (SLS) and Hexadecyltrimethylammonium Bromide (HDTMA-Br) Surfactant-Modified Activated Carbon as Adsorbent for Pb^{2+} and NO_3^-

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https://doi.org/10.14710/jksa.23.11.396-402

Article Info

Article history:

Received: 27th May 2020
Revised: 1st November 2020
Accepted: 12th November 2020
Online: 30th November 2020

Keywords:
Surfactant Modified Activated Carbon; Adsorption; adsorbate

Abstract

The adsorption efficiency and selectivity of activated carbon as an adsorbent for ions can be improved. One way is to convert activated carbon into surfactant modified activated carbon (SMAC). The surfactants used in this study were the anionic surfactant Sodium Lauryl Sulfate (SLS) and the cationic surfactant hexadecyltrimethylammonium bromide (HDTMA-Br). This research aims to synthesize SMAC to obtain a material with a surface charge and absorb ions better than activated carbon. This research consisted of four stages. The first step was the carbonization of rice husks using a pyrolysis reactor at 400°C for 1 hour. The second stage was carbon activation using 30% $ZnCl_2$ and microwave radiation for 5 minutes and 400 W. The third stage was the modification of activated carbon and characterization by FTIR, SEM, SAA. The fourth stage was the adsorption of Pb cations and nitrate anions by carbon, activated carbon, and SMAC. Several variables were applied, such as the type of surfactant, time, and method of modification. There are three ways of modification: (1) method A, in which activated carbon is brought into contact with SLS then HDTMA-Br. (2) Method B in which activated carbon was contacted with HDTMA-Br then SLS. (3) Method C in which activated carbon was brought into contact with SLS together with HDTMA-Br. All variables were investigated. The results showed that the optimum time for making SMAC for both surfactants was 4 hours, the optimum concentrations of SLS and HDTMA-Br were 60 and 300 ppm, respectively. SMAC made by the C method was the most effective at adsorbing Pb^{2+} and NO_3^- with adsorption capacities of 1.376 and 0.896 mg/g, respectively. The success of SMAC synthesis was evidenced by the S=O and $(CH_3)_3N^+$ groups in the FTIR spectra. The SMAC surface area is smaller than activated carbon, 14.472 m^2/g, but the surface morphology is smoother and more homogeneous.

1. Introduction

Activated carbon derived from coconut shells is more profitable than other agricultural wastes because it has a high density and purity, is harder, and is almost ash-free [1]. Coconut shell contributes to the world's pollution problem, reaching 3.18 million tons per year and representing more than 60% of the national waste volume [2]. Therefore, the use of coconut shells to become activated carbon is a means to reduce coconut shell waste. In general, activated carbon is made in 2 steps: carbonization and activation. Activation is carried out to build porosity and increase the carbon material's surface area [3].

Activator $ZnCl_2$ functions as a Lewis acid, which increases the polymerization reaction and inhibits volatile compounds' formation, thereby increasing the acquisition of activated carbon [4]. This results in a higher yield of activated carbon in the synthesis using the $ZnCl_2$ activator. The resulting activated carbon pores depend on the ratio of $ZnCl_2$ addition during activation.

Previous research [5] reported that at a concentration ratio of $ZnCl_2$: biomass of 1: 5, most pores of activated carbon were classified as micropore structures (<2 nm), while at a ratio of 2:5 and 3:5, the structure became mesoporous (2-50 nm). Meanwhile, the production of activated carbon from pumpkin seed shells with $ZnCl_2$ activator resulted in a surface area of 1,564 m^2/g at an optimum temperature of 500°C [6].

Many studies have currently used the microwave heating method because it has many advantages compared to furnace heating. Heating with microwaves requires a relatively short time, directly imparting energy to the carbon via molecular interactions, making it more energy-efficient [7]. Activated carbon from spruce seeds was prepared using microwave radiation in as little as 5 minutes [8].

Research on modification of activated carbon with surfactants has been carried out. Agustinus *et al.* [9] reported that activated carbon's adsorption power prepared from coal to Cr(VI) metal ions increased twofold due to modification with cationic surfactant ethylenediamine from 52.09% to 100%. Lee *et al.* [10] also proved that modified activated carbon using anionic surfactants, which are sodium dodecyl sulfate (SDS), sodium dodecylbenzene sulfonate (SDBS), and sodium octanoate (SO), increased the efficiency and absorption capacity of the ammonium ion cation compared to carbon.

The synthesis of activated carbon from coconut shells was carried out using $ZnCl_2$ activator and microwave radiation. Then, activated carbon was modified with anionic surfactant Sodium Lauryl Sulfate (SLS) and cationic surfactant hexadecyltrimethylammonium bromide (HDTMA-Br) to obtain a better SMAC than activated carbon. The synthesized SMAC was used to adsorb Pb^{2+} and NO_3^-. FTIR characterized materials to determine functional groups, SEM to determine surface morphology, and Gas Sorption Analysis (GSA) to determine specific surface area, total pore volume, and pore diameter. The concentration of Pb^{2+} before and after being adsorbed by SMAC was determined by atomic absorption spectrophotometer (AAS), while the nitrate groups were analyzed using UV-Vis. The purpose of this study was to determine the optimum contact time and concentration in the adsorption process of SLS anionic surfactant and HDTMA-Br cationic surfactants, to determine the most effective method of modification with SLS and HDTMA-Br to absorb Pb^{2+} and NO_3^-.

2. Methodology

2.1. Materials and Equipment

The materials used were coconut shell, $ZnCl_2$, Sodium Lauryl Sulfate (SLS) surfactant, HDTMA-Br surfactant, pH 8 phosphate buffer, distilled water, methylene blue, Bromophenol Blue (BPB), concentrated H_2SO_4, NaOH, $NaH_2PO_4.H_2O$, $Pb(NO_3)_2$, phenolphthalein indicator, chloroform, Whatman No. 42 filter paper. While, the equipment used was standard laboratory glasses, analytical scale, magnetic stirrer, pH indicator, pyrolysis reactor, Mettler AT 200 scale, Isotemp 630F oven, microwave, UV-Vis Shimadzu UV-1201 spectrophotometer, 100 mesh sieve, Perkin Elmer's FTIR. Spectrum Version 10.4.00, Atomic Absorption Spectrophotometer (SSA) and Surface Area Analyzer (SAA).

2.2. Procedure

Carbon was obtained from coconut shell carbonization through the pyrolysis process at 400°C for 1 hour. The carbon obtained was put into 100 mL of $ZnCl_2$ 30% and irradiated with microwaves for 5 minutes with a power of 400 W. The activated carbon was then washed with distilled water until a constant pH was obtained. Then, the activated carbon was dried at 105°C for 1 hour. The activated carbon was then contacted with SLS surfactant at concentrations of 40, 50, 60, 70, and 80 ppm with a contact time of 2-10 hours, with HDTMA-Br at concentrations of 200, 250, 300, 350, and 400 ppm with a contact time of 3-7 hours, to obtain the optimum surfactant concentration and time. Synthesis of SMAC was carried out by three methods. Method A was that activated carbon was contacted with SLS at the optimum concentration and time and then contacted with HDTMA-Br. Method B was that activated carbon was contacted with HDTMA-Br at the optimum concentration and time then contacted with SLS. Whereas method C was carried out in which the two surfactants were mixed then contacted with activated carbon. The amount of SLS absorbed by activated carbon was determined by the MBAS method, while the amount of HDTMA-Br absorbed was determined by the bromine phenol blue method. The formed SMAC was characterized using FTIR, SEM, SAA and kinetic data to determine the rate constants of the reaction, then SMAC was applied to the adsorbent Pb^{2+} and NO_3^-.

3. Results and Discussion

3.1. Coconut Shell Carbonization

Coconut shells were carbonized at 400°C for 1 hour in a pyrolysis reactor [11]. Carbonization is carried out to produce carbon, where in this process, volatile compounds are removed so that the initial pore structure is formed. The resulting heat energy breaks down the coconut shell's carbon complex molecules into carbon, gas, and tar. The results of this study obtained carbon of 25.11%. The reactions that occur during carbonization are presented in Figure 1.

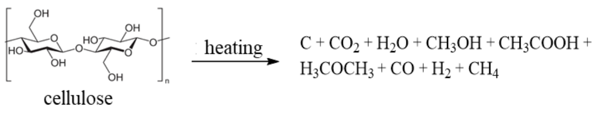

Figure 1. Carbonization reaction [12]

3.2. Carbon activation

$ZnCl_2$ activator is Lewis acid, which acts to bind impurities, hydrocarbons and open the pore structure formed from the carbonization. Activation can increase the carbon structure's lattice structure so that the more activated carbon structure is formed, the pore volume increases [13]. $ZnCl_2$ reagent is widely used as an active reagent because it produces a high surface area and a high yield of activated carbon [14]. The mechanism of activation with microwaves can be explained by dipolar polarization and ionic conduction. Some solvents have permanent dipoles. Dipoles rotate when exposed to an electric field, which causes friction and heat in the material. Ion-containing solutions can also generate heat by ionic conduction [15]. As a result, heating using microwaves, the time required is shorter. The characteristics of the activated carbon produced are also different from conventional heating. Acquisition of activated carbon in this study was 89.8% of carbon.

3.3. Synthesis of SLS and HDTMA-Br surfactant - modified activated carbon.

Activated carbon is hydrophobic, so surfactants can easily interact on its surface through hydrophobic attraction [16]. Surfactants change the surface characteristics of activated carbon from slightly polar to polar. The hydrophobic alkyl chains of the anionic surfactant (SLS) and long HDTMA-Br are oriented to the activated carbon's surface. Meanwhile, the SLS hydrophilic groups, which are negatively charged, and the positively charged hydrophilic groups of HDTMA-Br are oriented to the aqueous solution. As a result, the SMAC-SLS surface is negatively charged, while the SMAC-HDTMA-Br surface is positively charged. Some important control parameters in the adsorption of SLS and HDTMA-Br on activated carbon are the concentration of SLS and HDTMA-Br and contact time. This study determines the optimum conditions for SLS and HDTMA-Br adsorption on activated carbon to form the optimum SMAC. The surfactant adsorption mechanism on the activated carbon surface is presented in Figures 2.

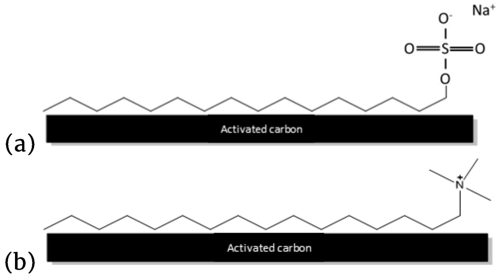

Figure 2. Mechanism of surfactant adsorption on activated carbon (a) SLS [17] and (b) HDTMA-Br [18]

3.4. Determination of the optimum contact time for surfactant adsorption

The relationship between contact time and adsorption efficiency is presented in Figure 3.

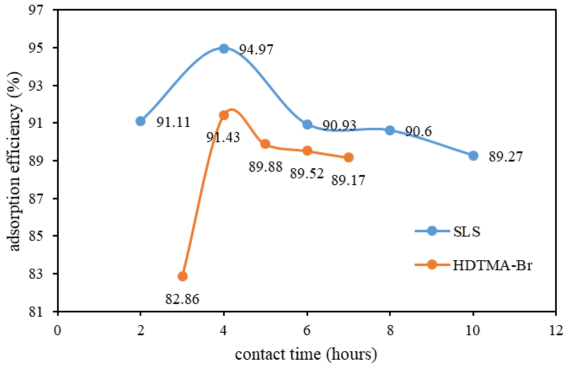

Figure 3. The relationship between contact time and the adsorption efficiency of SLS and HDTMA-Br

Figure 3 shows that SLS and HDTMA-Br surfactants' adsorption efficiency increases with increasing contact time until it reaches adsorption equilibrium, where the amount of adsorbed SLS and HDTMA-Br surfactants has covered the surface of the activated carbon. Four hours is the optimum contact time. At the contact time of 4 hours, activated carbon adsorbed SLS surfactant with an initial concentration of 60 ppm of 53.96 ppm with an efficiency of 94.97%. This result is better than previous studies with different base materials and activators, which showed an efficiency of only 90.87% [19]. At contact time after 4 hours to 10 hours, activated carbon's efficiency to absorb the SLS surfactant decreased by about 4.04%. This occurs because the SLS hydrophobic group's interaction with the surface of the activated carbon is weak. After all, it is only physical adsorption, so that the adsorbate is easily released. It is also thought that the pore corresponding to the molecular size of the SLS has been covered, resulting in decreased adsorption capacity.

Modification with HDTMA-Br showed that during the contact time between 3 to 4 hours, there was an increase in the number of HDTMA-Br molecules adsorbed by activated carbon. However, after 4 hours to 7 hours of contact time, the adsorption efficiency decreased, which means that the optimum contact time for HDTMA-Br adsorption on activated carbon was 4 hours. At 4 hours of contact time, activated carbon can adsorb HDTMA-Br with an adsorption efficiency of 89.04%. However, this result is smaller than previous studies, with different bases and activators, which showed up to 99.7% adsorption [20]. It can be explained that at the contact time of 4 hours, the pores of activated carbon corresponding to the size of HDTMA-Br were covered by HDTMA-Br surfactants. At the contact time after 4 hours, the efficiency of activated carbon in adsorbing HDTMA-Br molecules decreased by about 1.6%. This is because the functional group bonds formed between the activated carbon and the HDTMA-Br molecule are weak (physical adsorption), so they are easily released.

3.5. Determination of the optimum surfactant concentration

The optimum concentration of the surfactant adsorbed by activated carbon is shown in Figure 4.

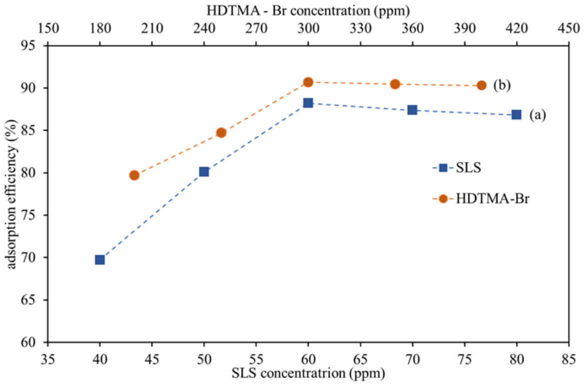

Figure 4. Effect of concentration on surfactant adsorption efficiency (a) SLS (b) HDTMA-Br

Figure 4 (a) shows that the greater the concentration of SLS surfactants, the greater the activated carbon's adsorption efficiency. The optimum concentration that can be adsorbed 1 gram of activated carbon is 60 ppm with 52.92 ppm of SLS surfactant and 88.21% efficiency. Previous studies using different basic ingredients and activators reached 91.09% [19]. SLS surfactants' adsorption tends to increase in the concentration range of 40 to 60 ppm, with the adsorption efficiency also increasing. The curve tends to flatten out after 60 ppm concentration. This shows that the activated carbon surface is starting to saturate because the pores on the surface of the activated carbon, which correspond to the surfactant molecules, have been covered by the surfactant molecules. The curve in Figure 4 (b) shows that the amount of HDTMA-Br adsorbed on activated carbon increases in the 200 to 300 ppm area. At concentrations after 300 ppm, the adsorption tends to flatten out, which indicates that HDTMA-Br molecules have covered the carbon surface. When the curve starts to flatten out, which is called the optimum concentration of HDTMA-Br adsorption, the efficiency is 90.69%. This efficiency is lower than the previous study, which used different basic materials and activators, which reached 99.7% [20].

3.6. Fourier transform infrared (FT-IR) spectroscopy

Figure 5 shows the analysis of SMAC spectra, which have been saturated by HDTMA-Br and SLS. A new peak appears at wave number 2918 cm^{-1}, which shows the vibrations of the aliphatic strain sp3 C-H from HDTMA-Br and SLS. The presence of an absorption peak at wave number 1460 cm^{-1} indicates a strain vibration from $(CH_3)_3N^+$ from the HDTMA-Br surfactant. There is a relatively weak absorption peak at wave number 1097 cm^{-1}, which indicates the S = O functional group derived from the surfactant Sodium Lauryl Sulfate (SLS). The FTIR spectrum of carbon activated carbon and SMAC samples is presented in Figure 5.

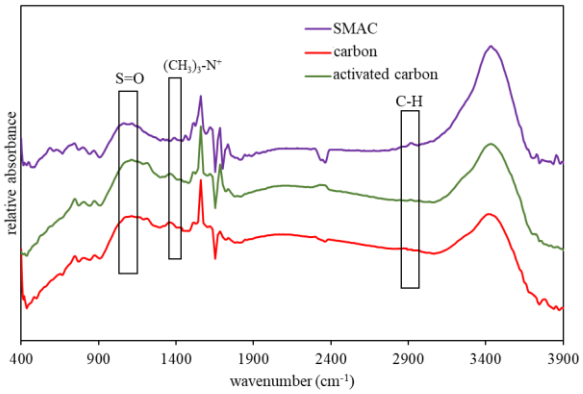

Figure 5. FTIR spectra of carbon, activated carbon, and SMAC

3.7. Scanning Electron Microscopy (SEM)

The surface morphology of carbon activated carbon and surfactant modified activated carbon were obtained from Scanning Electron Microscopy (SEM). Sample micrographs with magnifications of 2500 x and 5000 x, respectively, are shown in Figure 6. Comparison of carbon and activated carbon micrograph shows that the cavities and pores of activated carbon are more homogeneous and well developed on the external surface. The pores are seen to be uniformly developed in a mixture of oval and circular patterns. The pores formed have a large enough diameter that ranges from 2.19-6.39 μm. This indicates that the carbon activation was successful because the inactivated carbon did not show any pores. This indicates that the activation process, which involves $ZnCl_2$ as an activating agent, helps create more pores in the activated carbon, increasing surface area, and pore volume [4].

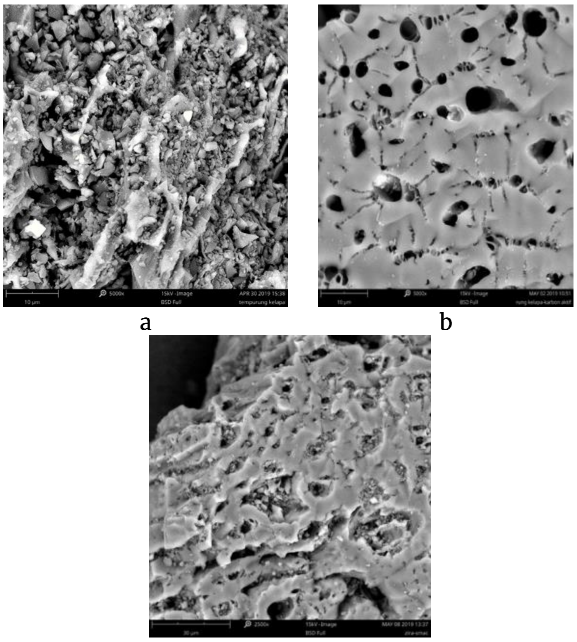
a
b
c

Figure 6. SEM results of (a) carbon, (b) activated carbon, (c) SMAC

Figure 6 shows that the micrograph of SMAC has pores between 2.65-5.22 μm. The pore size becomes smaller, and the particles in the pore activated carbon are visible. This indicates that the surfactant has been adsorbed by activated carbon. SEM images also show that SMAC's surface morphology is more homogeneous and smoother than that of carbon and activated carbon. Theoretically, the presence of surfactant interactions with the activated carbon surface provides more sites for adsorbing heavy metals or organic compounds [21].

3.8. Gas Sorption Analysis (GSA)

The pore and surface characters obtained from the Gas Sorption Analyzer (GSA) are presented in Table 1.

Table 1. Pore structure parameters of carbon, activated carbon, and surfactant modified activated carbon (SMAC)

Samples	BET Surface area (m^2/g)	Pore volume (cm^3/g)	Pore radius (Å)
Carbon	7.765	0.008	15.029
Activated carbon	47.512	0.033	16.019
SMAC	14.472	0.014	14.930

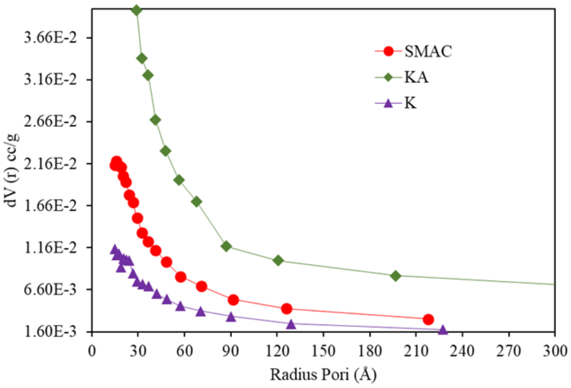

Figure 7. Pore size distribution

Table 1 shows that activated carbon activated by $ZnCl_2$ produces a larger surface area, volume, and pore radius than carbon. This shows that the preparation of activated carbon using $ZnCl_2$ activator using microwaves for 5 minutes with 400-Watt power is a fast and effective activation method. Surfactant-modified activated carbon shows a reduction in the BET-specific surface area or pore volume. In line with SEM analysis, Figure 7 shows that activated carbon pores are lower because it contains SLS and HDTMA-Br surfactants. According to the definition by IUPAC, the adsorbent pores are classified into three groups: micropores (<2 nm diameter), mesoporous (2-50 nm), and macropores (> 50 nm). Figure 7 shows that the pore size distribution of carbon, activated carbon, and SMAC is mostly at diameters of more than 20 Å or mesopore.

3.9. Adsorption Mechanism of Cationic and Anionic Surfactants by Activated Carbon

Figure 8 shows that the activated carbon modified by methods A and B occurs electrostatic interactions between each surfactant [22]. This makes SMAC less effective at adsorbing ions. In contrast to methods A and B, there was a competition between SLS and HDTMA-Br surfactants in method C, or there was competitive adsorption as proposed by [23]. Both surfactants occupy the pores of the activated carbon so that the surfactants are spread on the surface of the activated carbon in the form of their monomers.

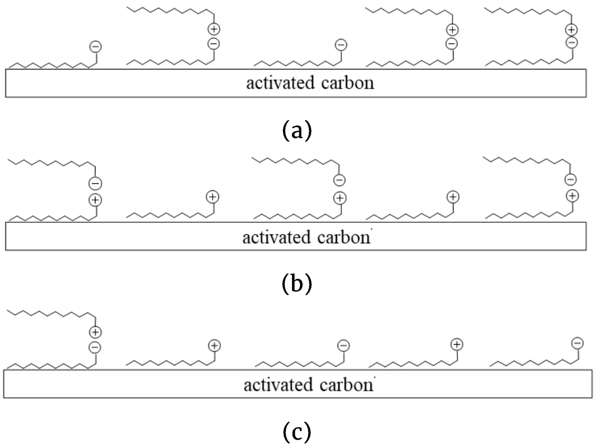

Figure 8. Prediction of activated carbon modification mechanism with SLS and HDTMA-Br: (a) Method A, (b) Method B, and (c) Method C

The direct interactions between surfactants and activated carbon can be in hydrophobic bonds, hydrogen bonds, dispersion forces, electrostatic attractions, and ion exchange. The lack of effect of head group charge on surfactant adsorption on activated carbon suggests random interactions, in which electrostatic contributions do not play an important role. The hydrophobic interaction between the surfactant hydrocarbon tails and activated carbon is the surfactant adsorption mechanism's driving force. Meanwhile, in the cationic-anionic surfactant mixture, the adsorption driving contribution is also influenced by the electrostatic attraction between the opposing surfactant headgroup charges. The adsorbed ionic surfactant can make the activated carbon surface fill, promoting the adsorption of other ionic surfactants that are otherwise charged. In other words, there is a synergy between cationic and anionic surfactants when they are adsorbed on the surface of the activated carbon.

3.10. SMAC application for Pb^{2+} and NO_3^- adsorption

This adsorption compares the specific absorption of Pb^{2+} and NO_3^- anions on activated carbon modified by anionic-cationic surfactant and activated carbon before being activated. Pb^{2+} and NO_3^- cations were adsorbed by SMAC with initial concentrations of 60 ppm and 36 ppm, respectively, with a contact time of 60 minutes. The absorption comparison test results between activated carbon, the results of the modification of activated carbon to SMAC with methods A, B, and C against Pb^{2+} and NO_3^- are presented in Figure 9.

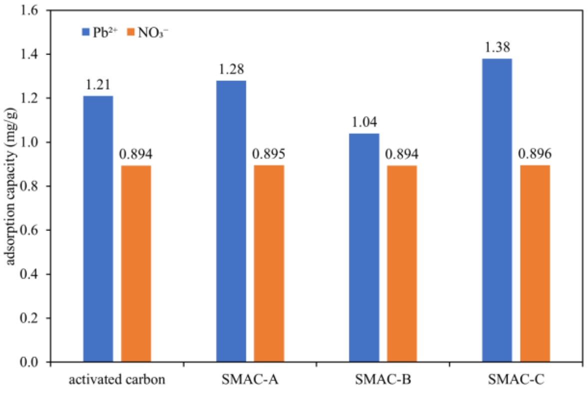

Figure 9. The capacity of Pb^{2+} ions and absorbed nitrate ions

Modification of activated carbon with HDTMA-Br and SLS surfactants causes activated carbon to have positive and negative charges on the activated carbon's surface. It can then be used as an adsorbent for Pb^{2+} and NO_3^- ions through ion exchange processes and electrostatic interactions [18]. Figure 9 shows that for all adsorbents - activated carbon, SMAC-A, SMAC-B, and SMAC-C - the adsorption capacity for the Pb^{2+} cation is always higher than the adsorption of NO_3^- anions. This is because the initial concentration of Pb^{2+} is higher than the NO_3^- ion, where the higher the initial concentration of the adsorbate, the adsorption capacity also increases [24]. Meanwhile, the adsorption capacity of nitrate ions in the four samples did not differ significantly. This is because the nitrate concentration used is too low and because the radius of NO_3^- hydrated (5.1 Å) is bigger than the Pb^{2+} cation (2.61 Å). This results in lower nitrate anions being adsorbed by activated carbon.

Figure 9 also shows that the adsorption capacities of Pb^{2+} and NO_3^- cations are higher at SMAC (C). The increase in the adsorption capacity of Pb^{2+} cations and NO_3^- anions occurs due to modification of the surfactant molecules on the activated carbon surface, which can multiply the active sites to bind or absorb ions. This is evidenced by a decrease in activated carbon's specific surface area after surfactant modification, from 47.512 m^2/g to 14.472 m^2/g [25]. The ion adsorption process does not only occur in pores but also occurs on charged surfaces. At SMAC-C, the adsorption capacity for NO_3^- anions was 1.376 mg/g and Pb^{2+} cations 0.896 mg/g. Adsorption on SMAC-C is competitive adsorption between SLS and HDTMA-Br so that both adsorbates occupy the entire surface of the activated carbon.

4. Conclusions

The optimal contact time for both SLS and HDTMA-Br adsorption on activated carbon was 4 hours with the optimum concentration of 60 ppm of SLS surfactant and 300 ppm of HDTMA-Br. The most effective method of modifying activated carbon with SLS and HDTMA-Br to absorb Pb^{2+} cations and NO_3^- anions is method C, with adsorption capacities of 1.376 mg/g, 0.896 mg/g, respectively. Surfactant modified activated carbon (method C) showed the presence of S=O and $(CH_3)_3N^+$ functional groups in the FTIR spectra with wavenumbers of 1097 cm^{-1} and 1460 cm^{-1}, respectively. SMAC's surface area decreased compared to activated carbon, which was 14,472 m^2/g with a smooth and homogeneous morphological structure.

Acknowledgment

We would like to thank the Faculty of Science and Mathematics, Diponegoro University, for the 2018 non-state budget research grant that has been given so that this research can be carried out.

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