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Effect of Mass and Contact Time of Coffee Grounds in Nickel Metal Ion Adsorption on Liquid Waste of Chemistry Laboratory

Inez Trinanda*, Yussi Pratiwi, Tritiyatma Hadinugrahaningsih

Department of Chemistry, Faculty of Mathematics and Naturals Sciences, Universitas Negeri Jakarta, Jl. Rawamangun Muka, Jakarta 13220, Indonesia

*Corresponding author: ineztagustus@gmail.com

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Abstract

Based on Indonesian Government Regulation PP No. 22 of 2021 concerning the implementation of environmental protection and management, the quality standard for nickel in waters is 0.05 mg/L. Nickel waste is very harmful to aquatic and terrestrial ecosystems and poses risks to human health. In this study, nickel waste was treated with coffee grounds using the adsorption method. The purpose of this study was to determine the effect of variations in the mass of coffee grounds and contact time on the adsorption of nickel metal in the liquid waste generated by the UNJ Chemistry laboratory. Coffee grounds are activated with NaOH 0.01 M to increase their adsorption ability. Two tests were conducted, namely, the variation of adsorbent mass and the variation of contact time. Based on the results of testing the variation of adsorbent mass and contact time, the highest percentage of removal was obtained at 91.70% at a mass of 0.05 g with a contact time of 15 minutes. The lowest percentage of removal was 39.56% in a mass of 1.5 g with a contact time of 90 minutes. This research provides a novel approach to heavy metal waste treatment especially nickel metal ion, which can be adapted to enhance waste treatment technologies in the future.

Keywords: coffee grounds, adsorption, nickel, liquid waste

1. Introduction

Laboratory liquid waste is waste produced from practical and research activities. The substances contained in laboratory liquid waste can range from harmless metals to heavy metals that have a negative impact on the environment [1]. Chemical waste from the chemical laboratory, especially waste containing nickel metal, is currently collected in a single container and remains unprocessed. Nickel is a metal with moderate toxicity so the waste cannot be disposed of directly into the environment because it is still toxic to the surrounding ecosystem and needs treatment [2]. Therefore, to ensure safe disposal into the environment, it must meet the quality standards. Based on Government Regulation PP No. 22 of 2021 concerning the implementation of environmental protection and management the quality standard of nickel in water is 0.05 mg/L [3].

One effective way that can be done to reduce metal levels in liquid waste from the Chemistry laboratory is through the adsorption method. The adsorption method has several advantages, namely it is a simple process, the energy required is low, and it the cost-effective [4]. If compared to other methods, adsorption is the most preferred method because of its high efficiency in removing heavy metal ions [5].

Adsorbents derived from biological materials are referred to as biosorbents. Biosorbents and their derivatives contain various functional groups capable of binding heavy metal ions. Typically, biosorbents are produced from agricultural waste such as chitin and chitosan, peat, yeast, biomass, fruit peels, eggshells, bagasse, tea waste, coffee grounds, and others. Agricultural waste-based biomass is preferred due to its lignocellulosic structure, which includes hemicellulose, cellulose, and lignin [5–7]. Biosorbents are known as adsorbents that can remove heavy metals from wastewater and have good potential to remove contaminants [8]. Biosorbents have characteristics that support good adsorption affinity for analytes, such as large surface areas, have various pore sizes, contain many

oxygenated functional groups, and are easy to modify to achieve the desired material properties [9].

Coffee grounds contain various functional groups, such as lignin, cellulose, fatty acids, and hemicellulose [10,11]. The adsorption ability of coffee grounds can be improved by activating it. The principle of activation is to expand the surface of the pores and present more oxygenated functional groups, as a result of which more molecules are adsorbed, so that the adsorbent absorption capacity increases [12,13]. The hydroxyl groups in cellulose play a significant role in ion exchange capacity. However, these groups exhibit weak ion exchange capacity due to the weak nature of the OH bonds. Untreated coffee grounds contain 23.69% hemicellulose and 21.06% cellulose. Coffee grounds treated with hexane contain 26.52% hemicellulose and 23.12% cellulose. Coffee grounds treated with ethanol exhibit 30.21% hemicellulose and 25.11% cellulose. Coffee grounds treated with sulfuric acid show 0.43% hemicellulose and 28.90% cellulose [14].

Temperature conditions, contact time, pH, initial substance concentration, and adsorbent dosage can affect adsorption efficiency [15]. Previous research investigated the adsorption of nickel using physically activated coffee grounds with varying masses ranging from 0.02 g to 0.12 g. The study revealed that higher doses of coffee grounds resulted in a decrease in adsorption capacity [16]. Another study examined the adsorption of nickel using chemically activated coffee grounds treated with NaOH. This research explored variations in particle size, pH, and adsorbent mass. It was found that differences in particle size had no significant effect, while an increase in adsorbent dosage led to a reduction in adsorption capacity [17]. In this study, coffee grounds were used to reduce nickel metal levels in laboratory liquid waste by testing the effect of variations in adsorbent mass and adsorbent contact time. The research problem addressed in this article is how the variation in mass and contact time of coffee grounds affects the percentage of nickel adsorption from liquid waste generated by Chemistry laboratory.

2. Materials and Method

2.1 Materials

The materials used consist of coffee grounds obtained from the espresso brewing process, deionized water (Waterone), $NiSO_4 \cdot 6H_2O$ (Merck), 0.01 M NaOH (Merck), 65% HNO_3 (Merck), filter paper (Whatman No. 42), and nickel waste collected from the UNJ Chemistry Laboratory.

2.2 Methods

2.2.1 Preparation of coffee grounds

The preparation of coffee grounds begins with activation. Activation of coffee grounds is carried out by chemical activation by soaking coffee grounds into a 0.01 M NaOH activator solution using a ratio of 1:10 (g/mL) for 30 minutes and repeated until the filtered solution is no longer yellow. Furthermore, the coffee grounds are washed with deionized water to pH ≈ 6. pH buffers were excluded as they could potentially act as ligands, competing with the metal cations (Ni^{2+}). Then the coffee grounds are dried using an oven with a temperature of 100°C for 2 hours and cooled at room temperature. After that, the coffee grounds are sifted using a 100-mesh sieve and weighed.

2.2.2 Characterizations

Characterization of coffee grounds is carried out with Fourier transform infrared (FTIR). In this study, FTIR aims to determine the hydroxyl functional group (OH^-) of cellulose in activated coffee grounds. The OH^- is generally in the range of 3200 cm^{-1} to 3600 cm^{-1} with a widened absorption form.

2.2.3 Preparation of standard solution

The nickel standard solution is obtained from the dilution of the nickel parent solution. A 1000 ppm nickel master solution is made by diluting $NiSO_4.6H_2O$ to 10 ppm. The parent solution is diluted to 10 ppm by inserting 1 mL of the parent solution into a 100 mL measuring flask, then diluted with HNO_3 0.10 M. It is then diluted again to make a standard solution, to 0.50, 1, 2, 3, and 4 ppm by inserting 2.5 mL, 5 mL, 10 mL, 15 mL, and 20 mL of 10 ppm parent solution into a 50 mL measuring flask.

2.2.4 Determination of nickel concentration

Nickel content measurement was carried out using an atomic absorption spectrometer (AAS, Shimadzu AA-7000 series). The wavelength used is 232 nm with a limit detection of 6 µg/L.

2.2.5 Adsorption process

In the adsorption process, nickel waste from the UNJ Chemistry laboratory is used. Table 1 shows the variation in mass and the variation in time.

Table 1. Variation in mass and contact time of adsorbent used in adsorption process by batch method.

Batch	Contact time (min.)	Mass of adsorbent (g)				
1	15	0.05	0.10	0.50	1	1.50
2	30	0.05	0.10	0.50	1	1.50
3	60	0.05	0.10	0.50	1	1.50
4	90	0.05	0.10	0.50	1	1.50

2.2.6 Data collection and analysis techniques

The data collection and analysis technique was carried out by calculating the removal efficiency and adsorption capacity. Removal efficiency was carried out to determine the comparison of concentrations between before adsorption and after adsorption. Removal efficiency can be calculated using equation (1) [18].

$$\text{removal efficiency (\%)} = \frac{(A-B)}{(A)} \times 100\% \quad (1)$$

Where:
A = initial concentration (mg/L)
B = final concentration (mg/L)

The adsorption capacity in this study aims to determine the amount of adsorbates accumulated on the surface of coffee grounds. The determination of adsorption capacity is expressed in the equation (2) [19]

$$qe = \frac{(C_o - C_e)}{m} \times V \quad (2)$$

Where:
qe = adsorption capacity (mg/g)
C_o = initial concentration of solution (mg/L)
C_e = final concentration of solution (mg/L)
m = mass of adsorbent (g)
V = sample volume (L)

3. Results and Discussion

Coffee grounds are used as a biosorbent to remove various contaminants, including heavy metals, due to their cellulose content and porous surface structure [20]. Biosorbents derived from coffee grounds exhibit superior adsorption capacity compared to biosorbents made from other lignocellulosic materials [21,22]. Coffee grounds are prepared by activating. This activation process is carried out to increase the ability of coffee grounds to adsorb nickel metal. One of the things that affects the adsorbent absorption of coffee grounds is the presence of OH^-. Activation with NaOH can increase the OH^- in the adsorbent.

The initial pH of the activated filtrate is 12.2. It was then rinsed with deionized water until the pH reached approximately 6 to remove impurities, such as chemical residues attached to the adsorbent. The coffee grounds washing process was carried out 13 times. In this study, deionized water was used because of its high purity and free from ions that could interfere with the results. Coffee grounds are sifted using a 100-mesh sieve. A 100-mesh sieve has pores of 150 micrometers, so the adsorbent has a finer particle size. This sifting process can also separate the fine dust contained in the adsorbent.

Figure 1. Filtrate from activation and washing process.

Previous research explained that inactivated biosorbents show a smaller surface area and pore volume compared to NaOH-activated biosorbents [23]. This shows that activating coffee grounds with NaOH can significantly increase the surface area and total pore volume of coffee grounds. The increase in surface area and pore volume causes higher metal ions to bind to the active site of adsorbents.

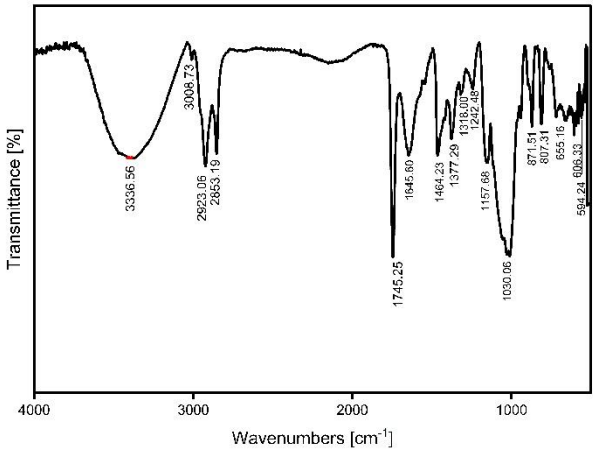

Figure 2. Results of characterization of activated coffee grounds functional clusters.

Figure 2 shows the results of the characterization of activated coffee grounds. In the activated coffee grounds, an absorption band was found at 3336.56 cm^{-1} with a widened absorption shape indicating the presence of a stretching O-H group. The peak is in accordance with previous research, where the range of 3200 cm^{-1} to 3600 cm^{-1} is the range of hydroxyl groups located [24]. The absorption band with strong intensity at 1745.25 cm^{-1} indicates the presence of a stretching C=O group. The

absorption bands with strong intensity at 2923.05 cm^{-1} and 2853.19 cm^{-1} indicate the presence of a stretching C-H group associated with an alkyl group. The absorption band with strong intensity at 1030.06 cm^{-1} and the absorption band with medium intensity at 1157.68 cm^{-1} showed the presence of C-O stretching bonds from the alcohol group. The absorption bands with medium intensity at 1645.60 cm^{-1} and 1464.23 cm^{-1} showed the presence of a C=C stretching group from the benzene group. The absorption band with a medium intensity of 1377.29 cm^{-1} indicates the presence of a C-H group from the methyl group [24].

Table 2. Characterization analysis of FTIR test results.

Wavenumbers (cm^{-1})		Functional Group
Research results	Reference	
3336.56 (*broad*)	3200 – 3600 (*broad*) [15]	O-H
1745.25 (*strong*)	1650-1750 (*strong*) [20]	C=O
2923.06 & 2853.19 (*strong*)	2850-3000 (*strong*) [20]	C-H
1030.06 (*strong*) & 1157.68 (*medium*)	1125-1205 (*medium to strong*) [20]	C-O
1645.60 & 1464.23 (*medium*)	~1600 & 1500-1430 (*strong to weak*) [20]	C=C
1377.29 (*medium*)	1370-1390 (*medium*) [20]	C-H

From previous research, inactivated coffee grounds have a widened absorption peak of 3300 cm^{-1} which indicates the presence of hydroxyl groups. It was found that the results of FTIR characterization for coffee grounds without activation and NaOH activated coffee grounds had similar functional groups, namely O-H bonds, C-H bonds, C=O bonds, and C=C bonds. The difference can only be seen in the percentage of transmission. The percentage of hydroxyl group transmittance in coffee grounds without activation is around 78%, in activated coffee grounds is about 25%. This shows that the hydroxyl group in NaOH activated coffee grounds has a stronger absorption peak than coffee grounds without activation. Meanwhile, coffee grounds that have adsorbed Ni^{2+} have a transmittance percentage of around 83%. A larger percent of transmittance indicates less IR radiation passing through the sample at a given wavelength, in this case the O-H wavelength. The high percentage of transmittance in coffee grounds that have adsorbed Ni^{2+} is estimated to be due to the obstruction of hydroxyl groups by Ni^{2+} due to steric effects [25].

A standard calibration curve is used to determine the concentration of nickel contained in waste. In making the standard calibration curve, 5 standard solutions were used, namely 0.5 ppm; 1 ppm; 2 ppm; 3 ppm; and 4 ppm. Standard solution and sample measurements, both before and after adsorption, use AAS. Figure 3 shows the curve of calibration. Based on the curve, it is known that the linear equation is y = 0.0801x + 0.0247 with a linear regression of 0.9955. Regression close to 1 shows a strong positive relationship between variables and shows a good predictive model fit [26].

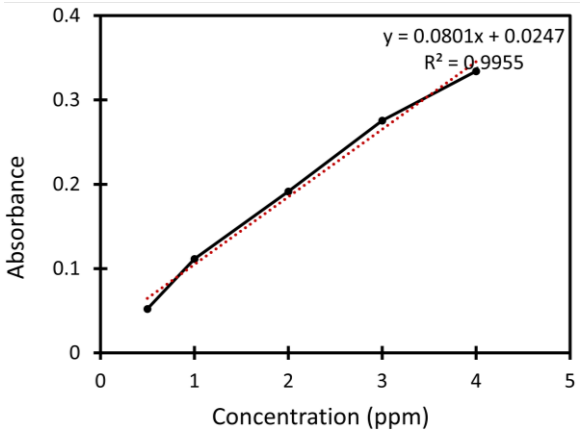

Figure 3. Standard calibration curve of nickel solution.

The adsorption pose begins by filtering the nickel liquid waste to be adsorbed. Nickel waste needs to be filtered first to avoid the presence of solids in the sample. The adsorption process is carried out by adding adsorbents which in this study are coffee grounds according to the variation in mass into 50 mL of nickel waste and stirred using a stirrer at a speed of 120 rpm. Screening uses Whatman No.42 filter paper so that fine particles remain filtered, so as not to interfere with the measurement process.

The destruction carried out in this study is wet destruction by adding a concentrated acid solution to the sample. The concentrated acid solution used in this study was HNO_3 65%. HNO_3 acts as a strong oxidizing agent. The heating process will cause the breaking of organometallic bonds to become inorganic [27]. Heating the sample is carried out until the volume of the solution reaches 15-20 mL to obtain a pure sample free of impurities. Then the sample was redissolved using HNO_3 with the aim of assisting in neutralizing any traces of concentrated nitric acid that may still be present after the destructive process.

The OH^- contained in coffee grounds has a partial negative charge on oxygen, which will later interact with the partial positive charge of Ni^{2+}. Oxygen from the hydroxyl group has a pair of free electrons that can bind to

Ni^{2+} ions. Ni^{2+} ions have an orbital void that can receive electron pairs from oxygen, resulting in the formation of a coordinating bond with Ni^{2+} ions, as demonstrated in Fig. 4. The bond will trap the nickel from the solution. This covalent bond enhances the interaction between the hydroxyl group and the surface of the coffee grounds, leading to chemical and physical alterations in the surface structure of the activated coffee ground.

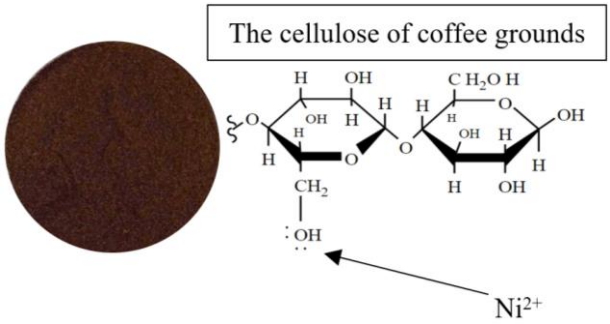

Figure 4. Illustration of the formation and breaking of bonds in the adsorption process [31].

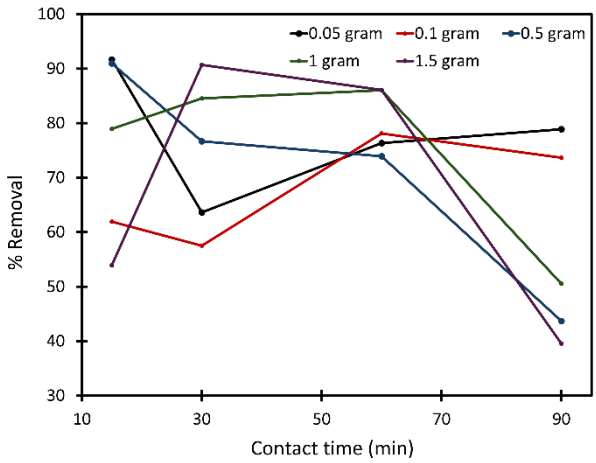

Figure 5. Graph of adsorbent mass relationship to nickel metal removal percentage.

The effect of coffee grounds adsorbent on the percentage of nickel metal removal can be seen in Fig. 5. The concentration of nickel waste before adsorption was known to be 0.51 ppm. In batch 1, which has a contact time of 15 minutes, the highest percentage of removal was found in the use of coffee grounds mass of 0.05 g with a removal percentage of 91.70%. In batch 2, which had a contact time of 30 minutes, the highest percentage of removal was found in the use of coffee grounds mass of 1.50 g with a removal percentage of 90.69%. In batch 3, which has a contact time of 60 minutes, the highest percentage of removal was found in the use of coffee grounds mass of 1.50 g with a removal percentage of 86.09%. In batch 4, which has a contact time of 90 minutes, the highest percentage of removal was found in the use of coffee grounds of 0.05 g with a removal percentage of 73.65%.

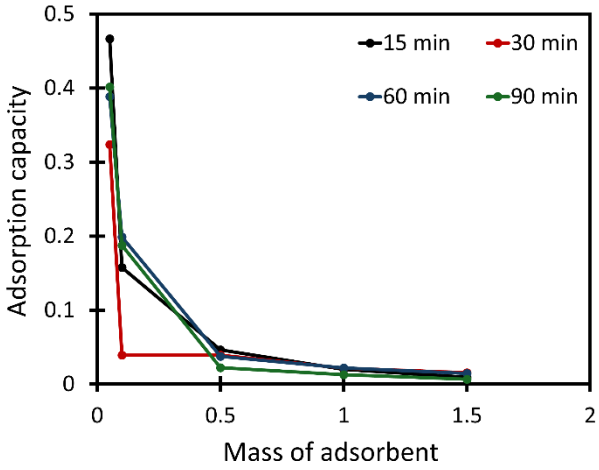

Figure 6. Graph of adsorbent mass relationship to nickel metal removal percentage.

Based on Fig. 5, it can be seen that the highest percentage of removals in each batch as a whole is decreasing. Meanwhile, the addition of the mass of coffee grounds to the percentage of removal is not directly proportional. This can happen because there is no longer a place available for binding, so the adsorption process stops. This can be attributed to the availability of active sites for adsorption in the early stages and after the interval of time, the remaining sites are difficult to occupy due to the repulsion-repulsive force [28]. Between the adsorbate (Ni^{2+}) molecule and the Ni^{2+} ions that have been entangled on the adsorbent surface because they have the same charge. Contact time of more than 1 hour can also allow desorption to occur which indicates that adsorption is reversible [29].

Figure 6 shows the adsorption capacity curve, which is obtained by plotting the mass of coffee grounds against the calculated adsorption capacity. The curve indicates that a coffee groundmass of 0.05 g achieves the highest adsorption capacity: 0.47 mg/g at 15 minutes, 0.32 mg/g at 30 minutes, 0.39 mg/g at 60 minutes, and 0.40 mg/g at 90 minutes. The adsorption capacity decreases as the adsorbent mass increases, differing from previous research, which found that higher adsorbent mass leads to greater adsorption capacity [30]. This discrepancy may occur because increasing adsorbent mass does not necessarily expand the surface area. Moreover, prior studies suggest that higher adsorbent mass can lead to biosorbent aggregation, reducing available adsorption

sites [17]. Such aggregation decreases the total surface area and can cause weakly bound adsorbate desorption from the adsorbent surface [31].

4. Conclusion

Coffee grounds adsorbents with masses of 0.50 g, 1 g, and 1.50 g showed a decreasing percentage of removal with the length of contact time because the availability of active sites for adsorption after intervals was difficult to occupy. As the mass of the adsorbent increases at 15 minutes and 90 minutes of contact time, the percentage of removal decreases. As the mass of adsorbents increases at 30 minutes & 60 minutes of contact time, the percentage of removal increases. However, the longer the contact time, the lower the average percentage of removal. This condition may occur due to biosorbent aggregation which causes a decrease in total surface area. Based on the findings, the optimum condition for coffee grounds to adsorb nickel is at 0.05 g for 15 minutes, achieving 91.70% removal and a final concentration of 0.04 ppm.

Author contributions

Inez Trinanda: Methodology, Investigation, Formal Analysis, Writing-Original Draft, Funding Acquisition. Yussi Pratiwi: Conceptualization, Supervision, Analyzed and Writing-Review & Editing. Tritiyatma Hadinugrahaningsih: Conceptualization, Supervision, Analyzed and Writing-Review & Editing.

Conflicts of interest

There are no conflicts to declare.

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