

DITIZHONE-IMMOBILIZED NICKEL SLAG FOR THE ADSORPTION OF SILVER(I) ION

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ABSTRACT

Nickel Slag (NS) is a byproduct generated from nickel ore processing, which mainly contains silica and alumina. This study has developed an adsorbent of dithizone-immobilized nickel slag (NSD) for Ag(I) removal in batch mode. Characterization of the adsorbent was carried out using SEM-EDX, XRD, FTIR, and DSC/TGA to confirm dithizone immobilization on the surface of activated nickel slag (NSA). The optimum condition for Ag(I) ion adsorption was achieved at pH 5 with a contact time of 90 minutes. The adsorption capacity of NSD towards Ag(I) was better than that of NSA. The adsorption kinetics of Ag(I) complied with the pseudo-second-order reaction, while the adsorption isotherms followed Langmuir isotherm models. Sequential desorption results indicated that Ag(I) adsorption in NSD was dominated by hydrogen bond interactions and complexation.

Keywords: Nickel Slag, Immobilization, Dithizone, Adsorption, Silver Ion.

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INTRODUCTION

Heavy metal pollution in water bodies affects water quality and threatens human life, health, and the surroundings. Silver is a heavy metal that is commonly utilized in many industries due to its superior flexibility, electrical and thermal conductivity, photosensitivity, ductility, and antimicrobial characteristic.¹⁻⁵ Adsorption is an excellent method for heavy metals removal because it is easy in an experimental setup and has a wide availability of various inexpensive adsorbents.⁶ Material containing SiO₂ is often used as a solid adsorbent due to the functional groups on the porous solid structure and possess excellent mechanical and thermal stability.⁷ Nickel slag (NS) is a solid waste produced by smelting nickel metal or nickel-iron alloy processing. The chemical composition of NS varies significantly depending on the ore source and the smelting process. It is frequently composed of SiO₂, MgO, Fe₂O₃, Al₂O₃, CaO, and others.⁸ There are 6-16 tons of nickel slag waste generated per ton of nickel produced.⁹ The increasing waste amount causes a decrease in soil, water, and air quality around the disposal area. In recent years, slag has begun to be used as a concrete mixture, road construction, cement mixture, as well as a raw material to fabricate Portland cement clinker for highway construction.⁹⁻¹² Abd El-Azim et al. reported that water-spray electric arc furnace slag could be an effective adsorbent for several heavy metals, e.g., Zn, Fe, Ni, Pb, and Co in aqueous solutions and wastewater.¹³ However, natural slag material as an adsorbent usually has a low adsorption capacity. Slag can be used as an adsorbent for heavy metals by adding a chelating agent and can be modified using a suitable ligand.¹⁴ Modifying the adsorbent can be done by coating the adsorbent's surface with a ligand that has active groups such as N, -NH, and -SH.¹⁵ Immobilizing dithizone can increase the adsorption capacity of some adsorbents such as zeolite, silica gel, and bottom ash.¹⁵⁻¹⁷ This study was conducted to synthesize dithizone-immobilized nickel slag (NSD) and analyze the adsorption parameters of this material, namely adsorption capacity, adsorption kinetics, and adsorption isotherm models towards Ag(I).

EXPERIMENTAL

Nickel slag was obtained from PT Aneka Tambang (ANTAM) Company located in Sulawesi, Indonesia. Dithizone (1,5-diphenylthiocarbazone), silver nitrate (AgNO₃), hydrochloric acid (HCl), toluene (C₇H₈), ethanol (C₂H₅OH), citric acid (C₆H₁₀O₇), disodium hydrogen phosphate (Na₂HPO₄), sodium citrate (C₆H₅Na₃O₇·2H₂O), sodium dihydrogen phosphate (NaH₂PO₄·H₂O), potassium nitrate (KNO₃), hydroxylamine hydrochloride (NH₂HO·HCl), acetic acid (CH₃COOH), and ethylenediaminetetraacetic

acid (Na_2EDTA) were obtained from Merck, Germany. The adsorbent synthesis was initiated by preparing activated nickel slag (NSA) from nickel slag. The activation process in NSA was done using 6 M HCl.¹⁷ The synthesis of dithizone-immobilized nickel slag (NSD) was carried out using NSA and dithizone with the assistance of toluene and ethanol. The immobilization process was done by immersing 4.0 g NSA into 80 mL of toluene. Then 1.0311 g of dithizone was added to it and then stirred for 4 h at 50 °C.¹⁸ The mixture was rinsed with toluene, ethanol, and water, respectively, and then dried at 50°C for 6 h. The characterization of adsorbents was done using FTIR (8201 PC Shimadzu), SEM-EDX (JSM-6510LA, 15 kV), XRD (Philips XRD X'Pert MS), and DSC/TGA (Linseis sta pt 1600). The Ag(I) concentration was identified using flame AAS (Atomic Absorption Spectroscopy, Perkin Elmer) at Integrated Research and Testing Laboratory, Universitas Gadjah Mada. The following adsorption studies were all conducted in a batch system. To study the pH effect, 100 g each of NSA and NSD were separately reacted with 10 mL of Ag(I) solution (10 mg/L). The solution pH was adjusted from 3.0 to 8.0 by the addition of buffer solution. Meanwhile, the optimal mass adsorption was determined by varying 10, 30, 50, 100, 200, 300, and 400 mg of adsorbent mass at the optimum pH. The adsorption kinetics of Ag(I) was investigated for both NSA and NSD. The adsorption contact time was set from 5 to 240 min at the optimum parameter condition. The data obtained were determined using the first and second-order pseudo kinetic model equation.¹⁹ Moreover, the isothermal adsorption model was investigated from the initial concentrations of Ag(I) ion on NSA and NSD. The initial concentration was varied to 10, 20, 30, 40, 50, 60, 70, and 80 mg/L. The data obtained were applied in Langmuir and Freundlich adsorption isotherm model equation to determine the adsorption capacity (q_{max}) and equilibrium constant (K_L) & (K_F).²⁰ Eventually, sequential desorption studies were carried out in 1 hour using water, 1 M KNO_3 , 0.3 M $\text{NH}_2\text{HO-HCl}$ in 25% CH_3COOH , and 0.1 M Na_2EDTA . Those four types of solvents were used to identify physical interactions, electrostatic, hydrogen bonding, and complexation between adsorbent and Ag(I) ion.

RESULTS AND DISCUSSION

SEM-EDX Characterization

Elemental and morphology analyses of the adsorbents were carried out using SEM-EDX. Table-1 shows EDX elemental analysis for NS, NSA, and NSD. It suggests that the activation process using acid has reduced various impurities in natural nickel slag and increased the % mass of Si and Al. Furthermore, immobilization of dithizone on NSA was successfully done. It is proven by increasing % mass of S, N, and C as characteristic elements of dithizone. Meanwhile, the SEM image in Fig.-1 (left) demonstrates that the NSA has a messy structure with silica as the main composition (Table-1). Besides, the NSD in Fig.-1 (right) has a neater structure and pointed shape, indicating the presence of dithizone on the NSA.

FTIR Characterization

The dithizone immobilization significantly affected the wavenumber changes on the FTIR spectra from the functional groups of NSA, NSD, and dithizone (Fig.-2). NSD (Fig.-2b) has the same vibrational peak as the characteristic functional group of dithizone (Fig.-2c) at wavenumbers of 1219, 1319, 1442, and 1496 cm^{-1} , which were associated with the vibration of C=S, CN, N=N, and C=C aromatics, respectively. The peak shift occurs in several wavenumbers in the NSD from the NSA, such as 756, 1627, and 3433 cm^{-1} .

Table-1: The EDX Elemental Analysis for NS, NSA, and NSD

Composition (% mass)	Adsorbent		
	NS	NSA	NSD
Si	17.14	28.16	5.74
Al	1.53	2.47	0.17
O	47.05	46.92	16.54
C	5.24	9.06	45.79
N	nd	nd	26.60
S	nd	nd	3.92
Fe	9.75	8.08	0.28
Mg	14.75	2.42	0.73
Other	4.54	2.89	0.23

*nd = not detected

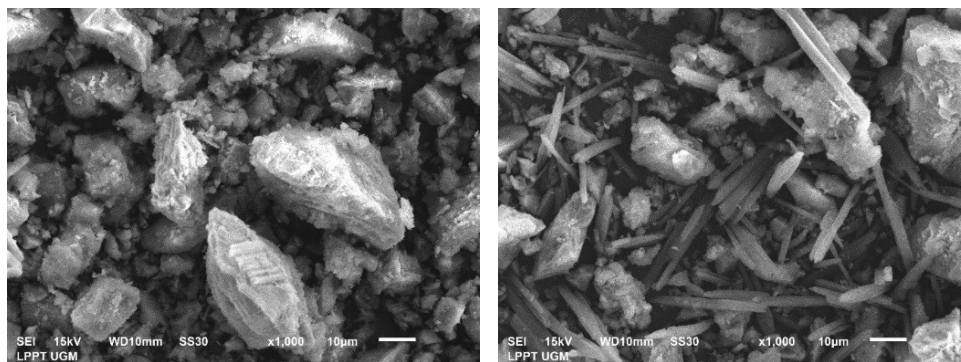


Fig.-1: Comparison of SEM Images between NSA (left) and NSD (right)

The wavenumber shift from 3448 to 3433 cm^{-1} (OH vibration) indicates that there were hydrogen bond interactions between the silanol groups of nickel slag and dithizone¹⁹, as well as between nickel slag silanol groups and water molecules in the NSD.¹⁷

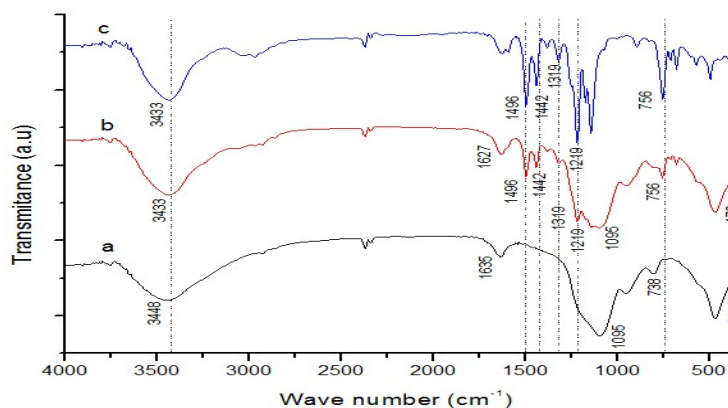


Fig.-2: Infrared Spectra of (a) NSA, (b) NSD, and (c) Dithizone

XRD Pattern

According to the database of the Joint Committee on Powder Diffraction Standards (JCPDS), the diffraction peaks of some minerals found in NSA (Figure-3a) are forsterite ($(\text{MgFe})_2\text{SiO}_4$) (number 00-031-0795), olivine ($\text{Mg}_2(\text{SiO}_4)$) (number 01-080-0944), quartz (SiO_2) (number 01-078-2315), and clinoenstatite (MgSiO_3) (number 00-013-0415). Several peaks that demonstrate characteristics of nickel slag and dithizone can be found in NSD (Figure-3b). Based on the results, the existence of peak characteristics of dithizone in NSD demonstrates that dithizone was successfully immobilized on the NSA surface. These results are supported by SEM-EDX and FTIR characterization data.

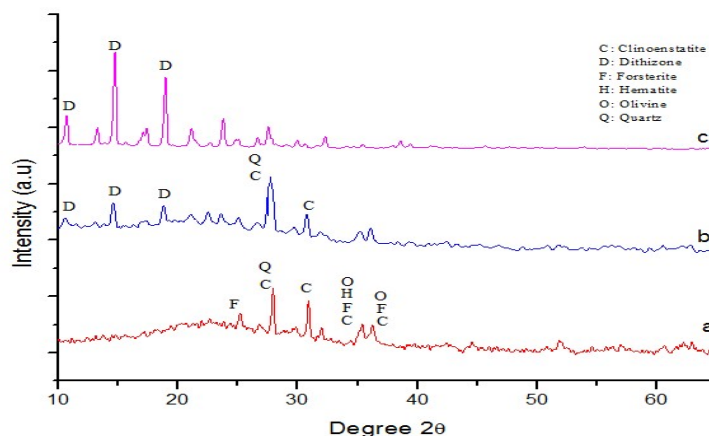


Fig.3: X-Ray Diffraction Pattern for (a) NSA (b) NSD, and (c) Dithizone

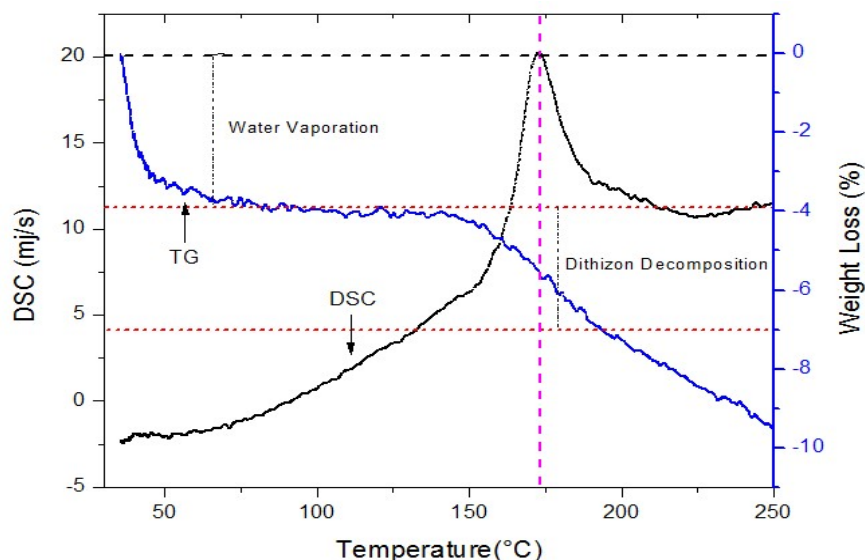


Fig.-4: DSC/TGA Curve for NSD

DSC/TGA Analysis

DSC/TGA analysis aimed to determine the heat required for the complete vaporization of each sample.²² Characterization using DSC/TGA on NSD has been carried out by heating this adsorbent up to 500°C (heating rate = 10°C/min). This procedure was done to measure the evaporation temperature of dithizone in NSD. The decrease in weight at 100°C (Figure-4) was due to the evaporation of the water molecules adsorbed by physical interactions. In addition, the decrease in weight between 150-200°C with an exothermic peak at 175°C was due to the molecular degradation of dithizone on the NSD. This is in correlation with research by Lumanyano *et al*²³, which stated that the degradation of dithizone initiates at 169°C, while other literature stated that dithizone is thermally decomposed at 130°C and possesses an exothermic peak at 181°C. Additionally, the weight loss of about 3% was due to the decomposition of dithizone in nickel slag. This is comparable to the literature that reported the decay of dithizone by 5%.²⁴

Effect of pH

Based on the results, the adsorption of Ag(I) by NSA and NSD reached an optimum condition at pH 5 with the adsorption capacities of 0.80 and 0.87 mg/g, respectively (Figure-5). At pH less than 5, the acid active sites tend to be protonated and have a partial positive charge. In this situation, the adsorption ability towards Ag(I) ions is low owing to the possible electrostatic repulsion between Ag(I) ions and the active group in NSA and NSD. In addition, the abundance of H⁺ concentrations at low pH causes competition between positively charged metal ions and H₃O⁺ ions to interact with the adsorbent.

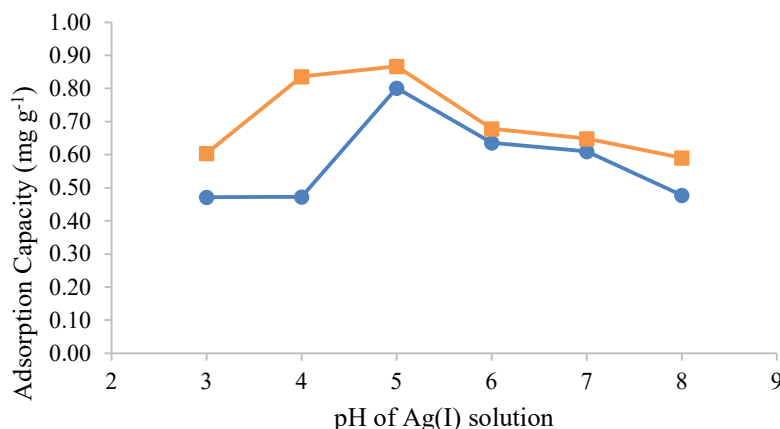


Fig.-5: Effect of pH on Ag(I) Adsorption by NSA (blue) and NSD (orange)

In a pH higher than 5, the adsorption capacity of the Ag(I) ion will drop. The higher the pH value in a solution, the higher the presence of OH^- ions. As a result, the possibility of precipitation of ion solutions is greater. According to the distribution diagram by Puigdomenech, the Ag(I) ion species tend to form a precipitate of AgOH at pH 10.34.²⁵ In general, the adsorption capacity of NSD towards Ag(I) is greater than that of NSA because there are additional active groups of N, -NH, and -SH, placed on the surface of the NSD. The functional groups in dithizone are more specific to interacting with soft and intermediate acids, such as Ag(I).²⁵ Referring to Pearson's HSAB (Hard Soft Acids and Bases) concept, Ag(I) is a soft acid that can interact with either soft or borderline bases. At the same time, the active groups in dithizone (N, -NH, and -SH) are soft bases as well as intermediate bases (borderline). Therefore, the number of metal ions adsorbed on NSD is higher than that on NSA.

Effect of Adsorbent Mass

The effect of NSA and NSD mass on Ag(I) ion adsorption resulted in the optimum mass of 0.2 g for both adsorbents (Figure-6). After reaching the optimum condition, the adsorbed Ag(I) mass tends to remain or slightly decrease. Adding a more considerable amount of adsorbent generally causes the adsorbed metal ions to tend to remain or slightly decrease. This was possible because metal ions do not fully occupy the active sites on the adsorbent.²⁶ Another possibility is that the interaction between particulates formed a large aggregate adsorbent that covers the active site and causes a reduction in the adsorbent's total surface area.

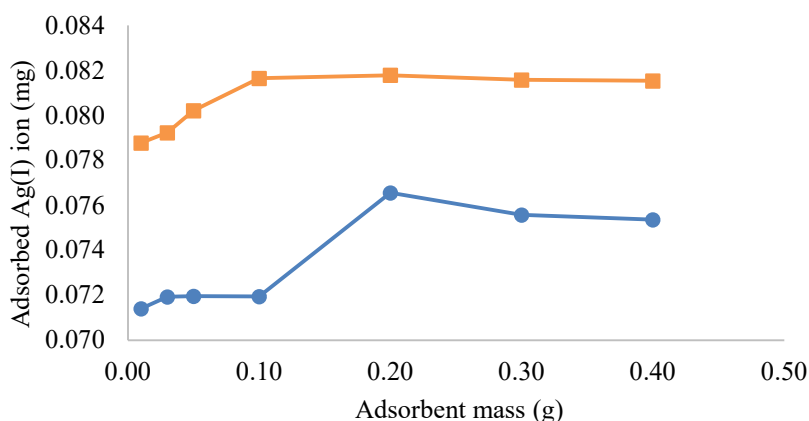


Fig.-6: Effect of NSA (blue) and NSD (orange) Mass on Ag(I) Adsorption

Adsorption Kinetics of Ag(I)

The equilibrium of Ag(I) ion adsorption on both NSA and NSD was reached at 90 minutes of contact time (Fig.-7). The adsorption kinetics in this study was determined based on the most suitable reaction kinetics equation approach. Table-2 presents various types of kinetic reaction orders. Based on the table, the adsorption of Ag(I) followed the pseudo-second-order kinetic models of Ho and McKay with an R^2 value of 0.99.

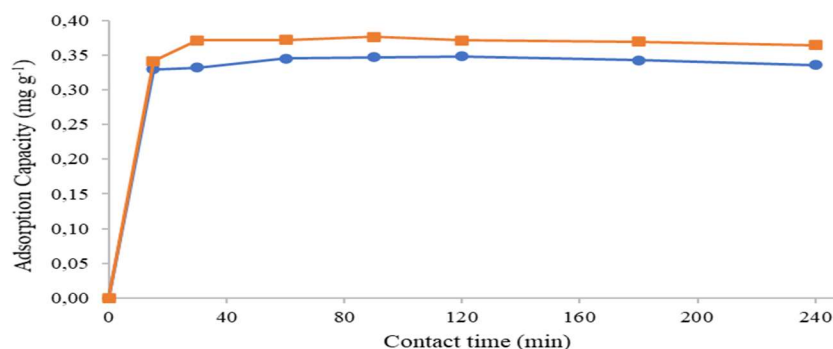


Fig.-7: Effect of Contact Time on Ag(I) Adsorption using NSA (blue) and NSD (orange)

Table-2: Adsorption Kinetics of Ag(I) on NSA and NSD

Reaction Order	R ² , Linearity	
	Ag(I)	
	NSA	NSD
Zero	0.4288	0.2267
First	0.4314	0.2291
Second	0.4340	0.2314
Third	0.3466	0.2337
Pseudo-first	0.0084	0.0073
Pseudo-second	0.9998	0.9998

Based on the results, the adsorption kinetics parameters show that the rate constant value (k_2) on the Ag(I) ion by NSD was higher than that by NSA with 13.7758 and 8.3330 g mg⁻¹ min⁻¹, respectively (Table-3). Meanwhile, the adsorption capacities (q_e) were 0.4155 and 0.3819 mg g⁻¹, respectively. This condition indicated that the metal ion adsorption process on the NSD was faster with a higher adsorption capacity than that on the NSA. The presence of dithizone on the NSD increases the type of active groups. Additionally, this caused the adsorbent was able to attract more Ag(I) ions and enhanced the adsorption rate constant.¹⁷

Table-3: Kinetic Parameters of Ag(I) Adsorption

Metal ion	Adsorbent	Adsorption Rate Constant*, k_2 (g mg ⁻¹ min ⁻¹)	Adsorption Capacity, q_e (mg/g)	Adsorbed of Adsorbate, h (mg g ⁻¹ min ⁻¹)
Ag(I)	NSA	8.3330	0.3819	1.2150
	NSD	13.7758	0.4155	2.3788

*Pseudo-second-order

Adsorption Isotherm of Ag(I)

Adsorption isotherm was studied by altering the initial concentration of Ag(I) while maintaining a constant temperature. The adsorption isotherm is the equilibrium correlation between the adsorbed ions and the adsorbent particles at a particular and constant temperature. Figure-8 shows the determination of the R² value as depicted in Table-4. According to the table, the R² value of Ag(I) adsorption is more suitable with the isotherm model of Langmuir than that of Freundlich. After the Langmuir isotherm model was chosen, the model was used to determine $q_{\max}$, K_L , and adsorption energy (Table-5). The NSD has higher values of $q_{\max}$, K_L , and adsorption energy than those of NSA. Because the adsorption energy value was greater than 20.92 kJ mol⁻¹ for both Ag(I) adsorption on NSA and NSD, this means that Ag(I) adsorption by NDS and NSA are chemical adsorption.

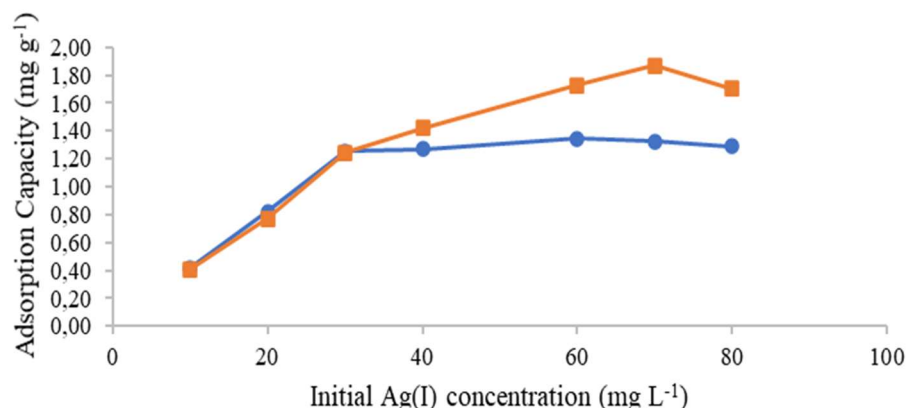


Fig.-8: Effect of Ag(I) Concentration on Adsorption Capacity of NSA (blue) and NSD (orange)

Table-4: Linearity Value (R^2) of Langmuir and Freundlich Adsorption Isotherm Models for Ag(I) Adsorption on NSA and NSD

Metal ions	Adsorbent	Linearity (R^2)	
		Langmuir	Freundlich
Ag(I)	NSA	0.9803	0.5423
	NSD	0.9886	0.9118

Table-5: Parameters of Langmuir Adsorption Isotherm Model for Ag(I) Adsorption on NSA and NSD

Metal Ions	Adsorbent	$q_{\max}$ (mol g ⁻¹)	K_L (L mol ⁻¹)	Adsorption energy (kJ mol ⁻¹)
Ag(I)	NSA	1.256×10^{-5}	5.07×10^4	26.842
	NSD	1.698×10^{-5}	9.64×10^4	28.434

Desorption Study of Ag(I)

In this study, the sequential desorption of the Ag (I) ion from the NSA and NSD was conducted to describe the interaction mechanism between Ag(I) ion and adsorbent. Before the desorption method was carried out, EDX elemental analysis was recorded for NSA and NSD adsorbents that had adsorbed Ag(I) ions. This elemental data after adsorption is presented in Table-6.

Table-6: Elemental Analysis Results of NSA and NSD During Adsorption of Ag(I)

Composition (% mass)	Nickel Slag Adsorbent	
	Ag(I) NSA	Ag(I) NSD
Si	23.11	28.48
Al	2.22	0.69
O	57.71	47.56
C	12.98	17.19
N	-	5.23
S	-	0.46
Ag	0.19	0.39
Co	-	-
Other	3.79	-

The desorption method used in this study referred to the previous experiment which applied double distilled water (physical interaction), potassium nitrate (electrostatic interaction), hydroxylamine hydrochloric acid (hydrogen bonding), and sodium EDTA (complexation) as desorbing agents.²⁷ The findings of the sequential desorption study are illustrated in Fig.-9.

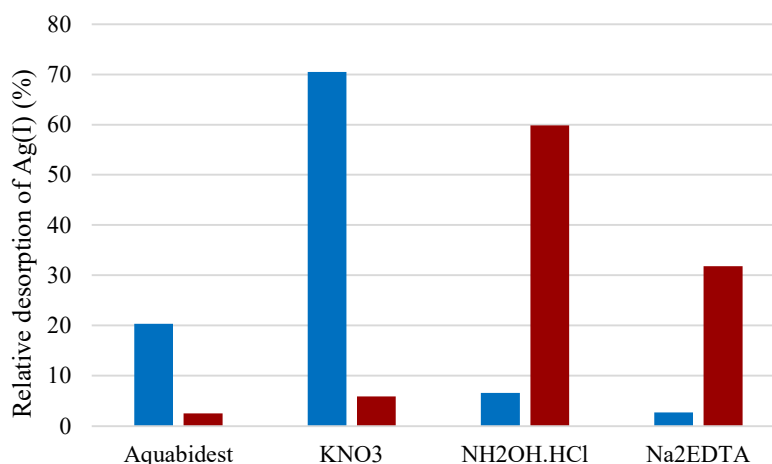


Fig.-9: Desorption Results of Ag(I) from NSA (blue) and NSD (orange) using Many Solvents

The results show that the interaction between Ag(I) and NSA was dominated by electrostatic/cation exchange interactions (70%) and physical interactions (20%). Meanwhile, the adsorption of Ag(I) in NSD was dominated by hydrogen bond interactions (60%) and complex formation (30%). The dithizone

immobilization on nickel slag has significantly increased the number of hydrogen bonds and complexations. On the other hand, it decreased the ion exchange/electrostatic and physical interactions. The adsorption mechanism through hydrogen bonds was possible between the hydrating complex of Ag(I) in the form of $[\text{Ag}(\text{H}_2\text{O})_2]^+$ and the active sites on NSD. Hydrogen bonds were formed by bonding the O atom in the H_2O molecule of the hydrated metal complex with the H atom from the surface of the nickel slag active group or between the N atom in the dithizone with the H atom of the H_2O molecule.

CONCLUSION

Immobilization of dithizone on nickel slag has been successfully carried out using toluene solvent and confirmed using FTIR, XRD, SEM-EDX, and DSC/TGA. The adsorption capacity of NSD towards Ag(I) is greater than that of NSA. The adsorption process in this study conforms to a pseudo-second-order kinetics and Langmuir isotherm model. Sequential desorption results suggest that the adsorption of Ag(I) in NSA is dominated by electrostatic and physical interactions, while in NSD is dominated by hydrogen bonding and complex formation.

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