



The Efficiency of Polyaluminum Silicate Chloride Coagulants in Nickel Removal from Aqueous Solutions

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Abstract

Background & Aims: Nickel is one of the toxic heavy metals naturally released into the environment through industrial activities. Coagulation and flocculation are one of the available heavy metal removal methods, but experience has shown that high pH and the addition of coagulant aid are usually required to remove nickel by coagulation and flocculation process. This study aimed to examine the efficiency of polyaluminum silicate chloride (PASiC) coagulant in nickel removal from aqueous solutions.

Materials and Methods: This study was conducted experimentally on a laboratory scale using the standard jar testing method. We investigated the effect of pH parameters (4-11), amount of coagulant (7.5-75 mg/L based on aluminum), initial metal concentration (10-400 mg/L), and settling time (15-90 minutes). Nickel concentration was measured by flame atomic absorption spectroscopy. In this study, *t* test, between-groups ANOVA, and regression were used for analysis.

Results: Results showed that the removal efficiency decreased at higher concentrations of nickel metal. Furthermore, increasing the settling time beyond 30 minutes did not significantly change the removal efficiency. PASiC had a removal efficiency of over 99% at an optimum pH of 8, an optimum PASiC concentration of 15 mg/L, an optimum time of 30 minutes, and a nickel concentration of 10-100 mg/L.

Conclusion: According to the results, the use of PASiC as a coagulant is a suitable option for removing nickel from polluted wastewater.

Keywords: Water purification, Metals, Heavy, Coagulation, Flocculation, Nickel, Polyaluminum silicate chloride

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1. Introduction

Minor amounts of heavy metals such as nickel, manganese, lead, chromium, cadmium, zinc, copper, iron, and mercury are important components of most wastewater [1]. The presence of high amounts of each of these metals interferes with the beneficial uses of water in terms of toxicity [2]. Industrial effluents typically contain high concentrations of contaminants such as organics, heavy metals, and toxic compounds [2]. Unlike organic substances, heavy metals are non-biodegradable, and most of them are known to be toxic and carcinogenic metals, causing numerous environmental and health problems [3,4]. Therefore, wastewater contaminated with heavy metals must be treated prior to discharge [5].

Nickel is a hard silvery-white metallic element that is ductile. Its chemical symbol is Ni, its atomic weight is 58.96, its density is 8.9, and its valence is 0, 1, 2, and 3 [6]. Nickel is one of the toxic heavy metals that can cause headache, nausea, dry cough, chest pain, shortness of breath, and cyanosis in high concentrations [7]. It usually enters drinking water through pipes and fittings. In addition, nickel can penetrate into underground water

through dissolution and rock erosion. It is used as an alloy in combination with other metals and non-metals. One of the properties of nickel alloys is strength and resistance to corrosion and heat [8]. Although inhalation of nickel metal compounds is carcinogenic, there is little evidence of carcinogenicity through ingestion. Perinatal mortality (senile) seems to be a major risk in drinking water containing 20 µg/L Ni as determined by the World Health Organization [9,10]. The adverse health effects of nickel are related to the heart and liver, and the European community has set a maximum value of 0.05 mg/L [6]. Although there is no information about the effects of nickel deficiency, nickel is essential for humans [8,11]. Divalent nickel compounds at the concentrations found around us are minimally toxic to humans. Adverse side effects caused by nickel exposure have been reported in cases of skin contact (causing contact dermatitis) and inhalation exposure (causing ductal irritation and asthma) in people exposed to this contaminant [12,13].

Various physicochemical processes such as chemical precipitation, flotation, membrane methods, ion exchange, electrochemical methods, and adsorption



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have been used to remove heavy metals from wastewater [8]. Heavy metal removal methods have limitations such as high cost, incomplete removal of metals, the need for reactive materials, large amounts of energy, and generation of toxic substances or secondary sludge that requires special treatment [9,10]. Coagulation and flocculation are one of the main processes in water and wastewater treatment. Coagulation works based on two mechanisms: charge neutralization of colloidal particles by hydrolyzed cationic coagulants and removal of impurities by irregular hydroxide compounds [14,15].

Polyaluminum silicate chloride (PASiC) belongs to a new generation of inorganic polymer coagulants. Polyaluminum chloride and polysilicate (PSi) are combined to form PASiC, which polymerizes trivalent aluminum under special conditions to improve the coagulation effect [16]. Considering the health and environmental hazards of nickel and the strengths and weaknesses of different heavy metal removal methods, as well as reviewing study records, it seems necessary to identify and use coagulant materials that can function over a wider pH range without the need for the addition of coagulant aid. Given the high efficiency of PASiC, this study investigated the efficiency of this coagulant in removing nickel from aqueous solutions. It also strived to determine the effect of pH, amount of coagulant, initial metal concentration, and settling time on the removal efficiency by coagulation and flocculation methods through PASiC coagulant and to determine the volume of produced sludge.

2. Materials and Methods

This is an experimental study conducted on a laboratory scale. PASiC coagulant concentration, pH, divalent nickel metal concentration, and settling time were considered in this study. Double distilled water was used for the preparation of stock solutions and dilution. Chemicals included nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), water glass (SiO_2), sodium carbonate (Na_2CO_3), and hydrochloric acid 37%. Moreover, sodium hydroxide from Merck and aluminum chloride (AlCl_3) from Activechem with laboratory grade were prepared. To make the nickel stock solution, 4.95 grams of ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was reached to 1 liter with twice-distilled water. This solution contained 1000 mg/L of divalent nickel ions. The current study used pH meter devices (EUTECH - pH 1500), a digital scale with an accuracy of 0.0001 (Sartorius TE214S), a 6-beaker test jar (Phipps and Bird), a flame atomic absorption device (AAS Vario 6), and a magnetic stirring device.

2.1. Synthesis of PASiC coagulant

PASiC was made according to the method proposed by Gao et al using the co-polymerization technique at room temperature with a molar ratio of Al/Si = 5 and OH/Al = 2 as follows:

First, 10.75 mL of water glass with a concentration of 3 M silicon dioxide was added slowly (1 mL/min) to 10 mL of 2 M hydrochloric acid under mixing conditions, and the solution was called PSi. It has the properties of silicon dioxide 1.5 M and pH = 2-2.5. In the second step, 2.5 M AlCl_3 was mixed with 10 mL of fresh PSi solution (about 2 hours old) to obtain a molar ratio of aluminum to silicate equal to 5. Then, 1.5 M sodium carbonate Na_2CO_3 solution was added slowly (0.1 mL/min) under mixing conditions (700-800 rpm) to obtain a molar ratio of hydroxide to aluminum equal to 2 and prepare PASiC coagulant, which is a milky liquid [17]. Figure 1 shows how to make and prepare PASiC.

2.2. Coagulation and flocculation tests

Experiments related to the process of coagulation and flocculation were carried out by the Phipps and Bird jar machine at a rapid mixing time of 1 minute with 200 revolutions per minute and a slow mixing period of 15 minutes with 45 revolutions per minute. Then, the solution was allowed to settle for 30 minutes to remove divalent nickel [18]. In the first phase of the first stage, a solution containing nickel with a concentration of 10 mg/L was prepared from the stock solution to determine the effect of pH on the efficiency of nickel removal, and the pH of the solution was adjusted using hydrochloric acid. Then, one-tenth normal sodium hydroxide [4-11] was set at various values, and PASiC coagulant was added to nickel-containing beakers at the rate of 75 mg/L (in terms of aluminum). In the second phase, 15 mg/L coagulant was added to the solution containing 100 mg/L nickel, and the jar test was performed according to the first phase. After the jar test, a sample was taken from a height of 2 cm below the water surface of each beaker, and the samples were filtered by a membrane. After preparing the samples, the residual nickel was read using a flame atomic absorption device. In the second stage, the optimum amount of PASiC coagulant was determined using the one-factor-at-a-time method. The solution containing nickel with a concentration of 100 mg/L was prepared from the stock solution, and the pH was adjusted to the optimum values obtained from the previous stage and neutral pH. Then, different concentrations of the coagulant in the values of 0, 7.5, 15, 37.5, 56, and 75 mg/L were added in terms of aluminum, respectively. After performing the jar test according to the previous stage, the remaining nickel was measured, and the best amount of coagulant was determined.

In the third stage, the effect of the initial concentration of nickel in 10, 25, 50, 100, 200, and 400 mg/L on the removal efficiency was evaluated under optimal pH and neutral pH conditions, and the optimal coagulant concentration obtained from the previous steps was tested and measured. In the fourth stage, to investigate the effect of settling time on the removal efficiency,

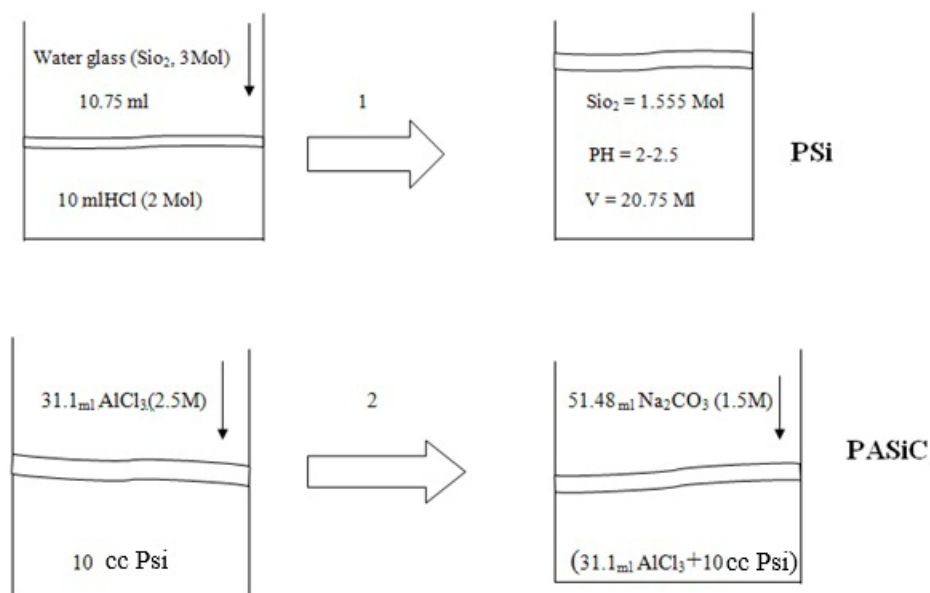


Figure 1. Production of PASiC With a Molar Ratio of 5 = Al/Si, 2 = OH/Al. Note. PASiC: Polyaluminum silicate chloride; AL: Aluminum; Si: Silicate; OH: Hydroxide

beakers containing the optimal concentration of PASiC and nickel obtained from the previous stages were tested at the pH rate of 7, and sampling was performed at settling times 15, 30, 45, 60, and 90 minutes. Finally, the amount of produced sludge with different amounts of PASiC coagulant was investigated.

A flame atomic absorption device (AAS Vario 6) at a wavelength of 232 nm was used to measure the concentration of divalent nickel ions. Nickel concentration was measured after the coagulation and flocculation process according to the acetylene-air direct blowing method (B)-3111) of the standard method of water and wastewater tests [19,20]. Further, 0.1 M solution of hydrochloric acid and sodium hydroxide was used to adjust the pH of the desired samples. The amount of metal removal was calculated according to the following formula:

$$Removal\ efficiency\ (\%) = \frac{C_i - C_f}{C_i} * 100 \quad (1)$$

where C_i and C_f are the initial and final concentration of the metal, respectively. All tests were conducted at room temperature, each stage of the test was measured twice, and finally the average results were reported.

2.3. Statistical data analysis

The graphs were drawn using Microsoft Excel 2007. In this study, SPSS 16 was used to perform statistical tests, including between-groups ANOVA, regression, and *t* test.

3. Results

3.1. Determining the effect of pH

The effect of pH on the removal efficiency of nickel from synthetic samples is shown in Figures 2a and 2b. In

this stage, 75 mg/L of PASiC was added to the solution containing 10 mg/L of nickel as a coagulant according to Figures 2a, and the effect of pH in the range of 4-11 was investigated based on the removal efficiency. The settling time at this stage was 30 minutes. Results indicated that the nickel removal efficiency increased from 4 to 7 pH and increased from 5.06% to 58.63%. Moreover, in the pH range between 8 and 11, the nickel removal efficiency was always a constant value of 99%.

Then, according to Figure 2b, 15 mg/L of PASiC was added to the solution containing 100 mg/L of nickel, and the effect of pH in the range of 4-11 was investigated based on the removal efficiency. As can be seen in Figure 2b, the nickel removal efficiency increased with the change in pH from 4 to 7 and increased from 20.13% to 52.54%, then in the pH between 8 and 11, the nickel removal efficiency was always a constant value of 99%. As a result, pH equal to 8 with 99% removal efficiency was chosen as the optimal pH for nickel removal.

3.2. Determining the effect of PASiC coagulant dose

Figures 3a and 3b present the effect of PASiC coagulant dose on nickel removal efficiency from synthetic samples at neutral pH and optimal pH. In this step, according to Figure 3a, first, under neutral pH conditions, different coagulant concentrations of 0, 7.5, 15, 37.5, 56, and 75 mg/L were added to the solution containing nickel with a concentration of 100 mg/L in terms of aluminum. The highest and lowest observed removal efficiencies were 52.54% and 21.85% in doses of 15 and 75 mg/L, respectively. Further, the results of the statistical test comparing the mean of the single variable *t* test showed a significant difference between the concentration of nickel ions contacted and not contacted with the coagulant in the process of coagulation and flocculation ($P < 0.05$).

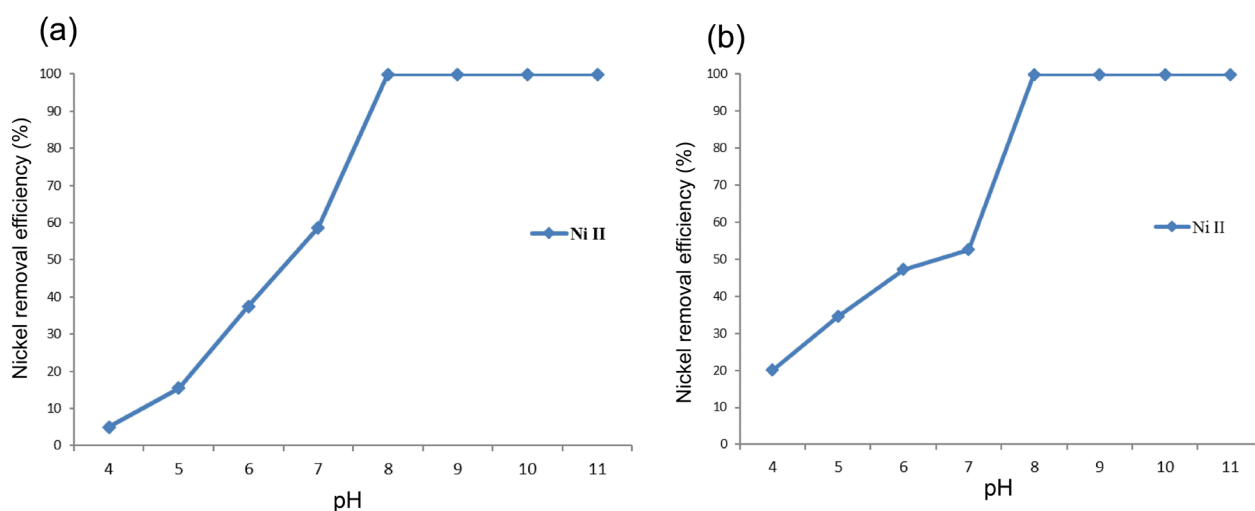


Figure 2. Effect of pH on nickel removal efficiency (a: 10 mg/L Ni = 75 mg/L PASiC, settling time 30 minutes and b: 100 mg/L Ni = 15 mg/L PASiC, settling time 30 minutes). Note. PASiC: Polyaluminum silicate chloride; Ni: Nickel

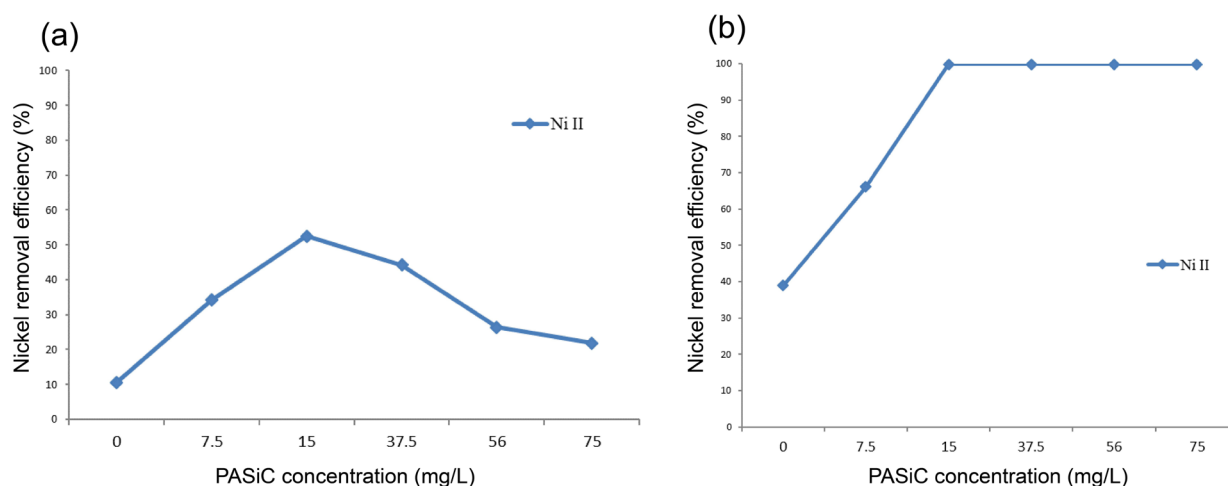


Figure 3. Effect of PASiC concentration on nickel removal efficiency (a: mg/L = 100 Ni, pH = 7, settling time 30 minutes and b: mg/L = 100 Ni, pH = 8, settling time 30 minutes). Note. PASiC: Polyaluminum silicate chloride; Ni: Nickel

According to Figure 3b, in this stage, under the optimal pH conditions obtained from the previous step (pH = 8), different coagulant concentrations of 0, 7.5, 15, 37.5, 56, and 75 mg/L were added to the solution containing nickel with a concentration of 100 mg/L in terms of aluminum. The results revealed that the highest and lowest observed removal efficiencies of 99% and 66.12% were obtained at doses of 15 and 7.5 mg/L, respectively. Moreover, the results of the statistical test comparing the mean of the single variable *t* test demonstrated a significant difference between the concentration of nickel ions contacted and not contacted with the coagulant in the process of coagulation and flocculation ($P < 0.05$). Based on this, the coagulant dose of 15 mg/L was chosen as the optimal amount of nickel removal.

3.3. Determining the effect of initial metal concentration

At this stage, the effect of the initial concentration of nickel was studied in values of 10, 25, 50, 100, 200, and 400 mg/L at neutral pH and the dose of PASiC equal to

15 mg/L. The highest and lowest observed nickel removal efficiencies were obtained at 71.22% and 34.08% in initial concentrations of 10 and 400 mg/L, respectively. Figure 4a illustrates the trend of changes in nickel removal efficiency parallel to the increase of initial concentration at a pH rate of 7.

Then, the effect of the initial concentration of nickel of 10, 25, 50, 100, 200, and 400 mg/L was studied at the optimal pH of 8 and the dose of PASiC equal to 15 mg/L. The results indicated that the highest removal efficiency is obtained in the concentration range of 10-100 mg/L, which is equal to 99%, and the removal efficiency was 53.48% and 30.84% in metal concentrations of 200 and 400 mg/L, respectively. Figure 4b depicts the trend of nickel removal efficiency changes in parallel to increasing initial concentration at an optimal pH of 8.

3.4. determining the effect of settling time

The effect of settling time on the amount of nickel removal using PASiC is presented in Figure 5. At this

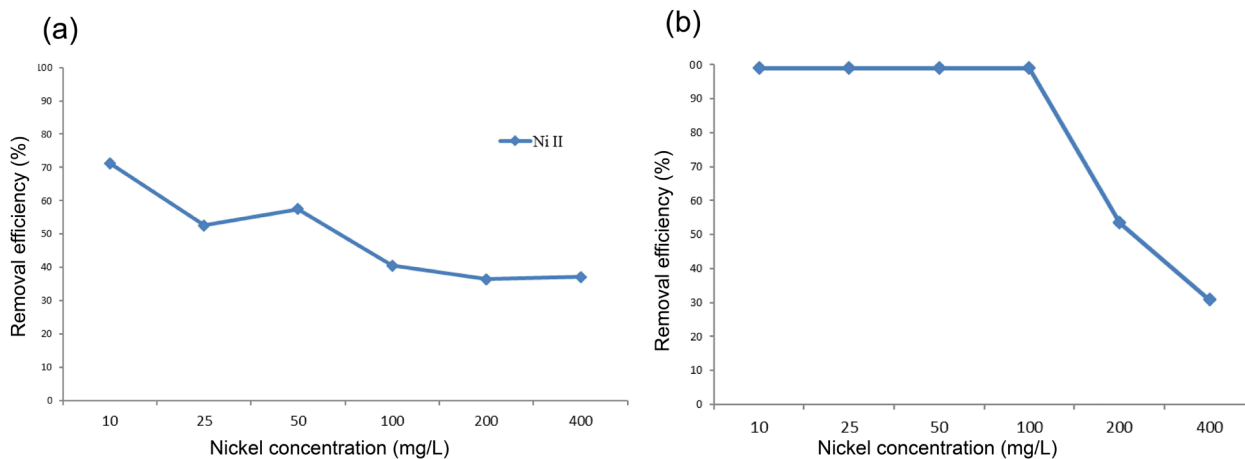


Figure 4. The effect of initial metal concentration on nickel removal efficiency (a: mg/L=15PASiC, pH=7, settling time 30 minutes and b: mg/L=15PASiC, pH=8, settling time 30 minutes). Note. PASiC: Polyaluminum silicate chloride

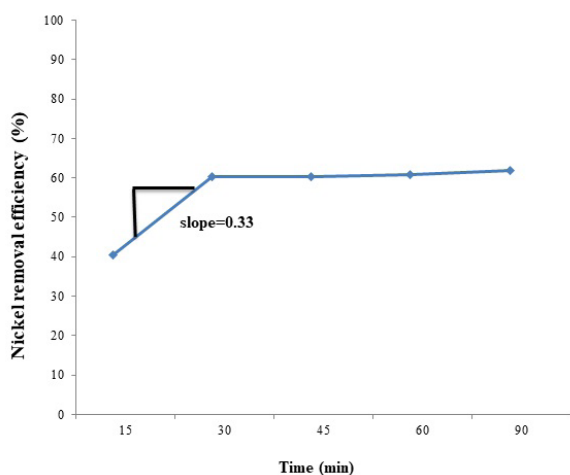


Figure 5. The effect of the settling time on nickel removal efficiency (100 Ni=mg/L, PASiC =15 mg/L, pH=7). Note. Ni: Nickel; PASiC: Polyaluminum silicate chloride

stage, the coagulant concentration was 15 mg/L, the nickel concentration was 100 mg/L, and the pH was 7. The highest and lowest nickel removal efficiencies were obtained at 54.98% and 43.26% in 15 and 90 minutes, respectively. According to the obtained results, by increasing the settling time up to 30 minutes, the removal efficiency increased dramatically, the nickel removal efficiency changed from 30 to 90 minutes, the slope of the graph was almost constant, and no significant change was observed in the removal rate. Accordingly, 30 minutes was considered as the optimal settling time for nickel removal.

3.5. Determining the volume of produced sludge

In this phase, the volume of sludge formed at the optimum pH of 8 and 100 mg/L of nickel was studied with PASiC doses of 15, 37.5, 56, and 75 mg/L, respectively. As can be observed in Figure 6, the highest and lowest amounts of produced sludge were 80 and 52 mL/L at coagulant doses

of 75 and 15 mg/L, respectively.

4. Discussion

4.1. Determining the effect of pH

In order to determine the optimal pH of the coagulation and flocculation processes in the removal of divalent nickel from aqueous solutions by PASiC coagulant, this stage was carried out in two phases. In the first phase, 10 mg/L nickel was added to the aqueous solutions, and after adjusting the pH in the range of 4-11, 75 mg/L PASiC was added to each beaker. Next, after conducting the jar test, the residual nickel was measured. As can be seen in Figure 2a, with increasing pH, the amount of nickel removal increased, and at pH equal to and above 8, the nickel removal efficiency was 99%. In the second phase of determining the optimum pH, 100 mg/L of nickel and 15 mg/L of PASiC were added to beakers. According to Figure 2b, the nickel removal efficiency increased with increasing pH, and at pH equal to and above 8, the nickel removal efficiency was 99%. Therefore, the optimal pH for nickel removal by PASiC coagulant was obtained to be 8.

Regarding the relationship between pH and hydrolyzed groups of aluminum, Yang et al reported that species of aluminum hydrolysis at pH less than 5 include monomeric, dimeric, and Al₃ aluminum. At pH between 5 and 6, medium and large aluminum polymer species with Al₁₃ cores were dominant, and the flocs formed in this case were relatively small. Moreover, the dominant form of aluminum hydrolysis at pH between 6 and 7 consisted of amorphous Al(OH)₃ clots, and in this case, colloids and natural organic substances were easily removed by the mechanism of absorption and co-precipitation by cationic species, aluminum hydroxide with low solubility, and the top level. Further, at pHs higher than 7, Al(OH)₄-hydrolyzed species were dominant, and the stability of the suspension increased [18]. Bakar and Halim found that the removal rate of nickel from industrial wastewater by polyaluminum chloride and coagulant aid at neutral pH

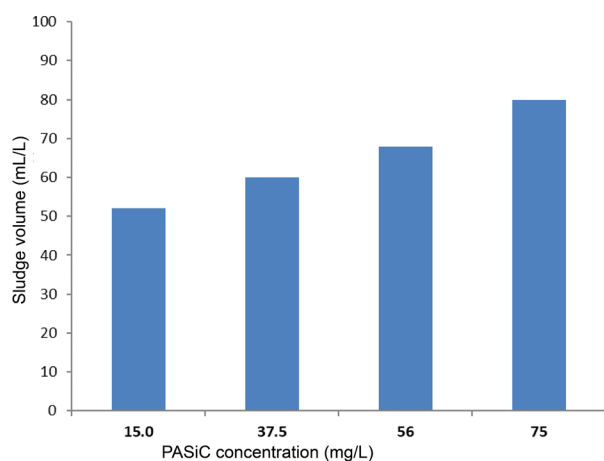


Figure 6. The Amount of Produced Sludge (mg/L: Ni = 100, pH = 8). Note. Ni: Nickel

was 63%. In the present study, the nickel removal rate at neutral pH was less than 60%, indicating that high pH is required to achieve high efficiency in nickel removal [21]. The results of Hu et al demonstrated that the removal efficiency of cadmium increased with increasing pH, and the removal rate at a pH of 8.5 was 93% [22]. Additionally, in the study by Liu et al, the highest removal efficiency of nickel from industrial wastewater was obtained by polyaluminum chloride coagulant at a pH of 10 [23]. The results of Jaafarzadeh et al showed that the removal efficiency increased with the increase of pH to 9 due to the reduction of the positive charge of chitosan coagulant and the cationic properties of nickel [24].

4.2. Determining the effect of PASiC coagulant dose

In the second stage of the study, the effect of different dosages of coagulant on nickel removal efficiency was investigated under neutral pH conditions and optimal pH obtained from the first stage. As seen in Figures 3a and 3b, the removal efficiency increased with the increase of coagulant dose. According to Figure 3a, the highest nickel removal efficiency at neutral pH was obtained from PASiC at a concentration of 15 mg/L, which is equivalent to 52.54%. According to Figure 3b, the highest nickel removal efficiency was obtained at the optimal pH of 8 for PASiC at a concentration of 15 mg/L, and the removal efficiency was above 99%. The results of Gyawali and Rajbhandari showed that increasing the dosage of PASiC coagulant increased the amount of turbidity removal. It was also observed that by decreasing the ratio of aluminum to silicate, the molecular weight of the coagulant increased, and by increasing the amount of silicate in the structure of the coagulant, the growth and development of flocs were faster due to the bridging mechanism. Further, by increasing the ratio of hydroxide to aluminum, the removal efficiency increased [25]. Therefore, given that in this study, a coagulant with a high ratio of silicate to aluminum was used, nickel removal was

complete at alkaline pH. In a study, Jaafarzadeh et al found that the removal efficiency of nickel increased from 40% to 88% by changing the amount of chitosan from 10 to 100 mg/L [24]. In another study, Akbal and Camci reported that by changing the amount of alum and ferric chloride coagulants from 100 to 2000 mg/L, the nickel removal efficiency increased from 24.5% to 99% [26].

4.3. Determining the effect of initial metal concentration

A test was done to determine the effect of the initial concentration on the removal efficiency at the optimal dosage of coagulant of 15 mg/L, optimal pH of 8, and neutral pH. As depicted in Figure 4a, under neutral pH conditions, the removal efficiency decreased with the increase in nickel concentration. The results of Jaafarzadeh et al showed that the efficiency of nickel removal by chitosan coagulant increased with the increase of the initial concentration of nickel, which was attributed to the insufficient amount of coagulant to absorb the pollutant surface on the coagulant [24]. Further, the results of a study by Xu et al showed that the removal efficiency decreased with the increase of the initial concentration of cadmium due to the insufficient absorption sites [27]. Moreover, according to Figure 4b and at a pH of 8, it was observed that the amount of nickel removal decreased to 200 mg/L with the increase in the amount of nickel.

4.4. Determining the effect of settling time

This test was performed in the optimal concentration of PASiC of 15 mg/L, nickel concentration of 100 mg/L, and pH of 7. According to Figure 5, the removal efficiency at settling times of 15, 30, 45, 60, and 90 minutes was 43.72%, 52.54%, 53.6%, 54.5%, and 54.98%, respectively. In Liu and colleagues' study entitled "Removal of phosphorus and nickel from automobile industry wastewater by coagulation and flocculation and combination with manganite, it was found that the amount of removal increases by increasing the settling time to 30 minutes [23].

5. Conclusion

In general, the results obtained from this study indicated that the presence of active PSi compounds in the coagulant polymer structure increased the negative charge of PASiC in comparison with common coagulants such as polyaluminum chloride, and this negative charge facilitated the nickel removal, which had cationic properties. The results also showed that in the alkaline pH range, the removal efficiency increased due to the production of $Al(OH)_4^-$ hydrolytic species and the decrease of nickel ion solubility. The results suggested that PASiC at pH of 8, PASiC concentration of 15 mg/L in terms of aluminum, settling time of 30 minutes, and a concentration range between 10-100 mg/L related to nickel had removal efficiency of above 99%. Therefore,

the coagulation and flocculation processes using cadmium-nickel were effective, and PASiC could be used as an effective chemical method to remove nickel.

It is also recommended to study the use of PASiC coagulant in industrial wastewater treatment and to check its effectiveness in removing parameters such as turbidity, suspended solids, biological oxygen demand, chemical oxygen demand, oil, heavy metals, and the like.

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Author Contributions

M-JM, AT, MZ, and A-BN were principal investigators of the study and drafted the manuscript. M-JM and AT were advisors of the study. AT, MZ, and A-BN performed the statistical analysis. All authors contributed to the design and data analysis and assisted in the preparation of the final version of the manuscript. All authors read and approved the final version of the manuscript.

Conflict of Interests

There is no conflict of interests according to the authors of this study.

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