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Cr (VI) Removal from Aqueos Solution by Coagulation – Adsorption Integrated System

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Abstract

Industrial wastewater generated by nickel mining has a high content of heavy metals, one of which is Chromium Hexavalent or Cr(VI) metal. Commonly used methods in reducing Cr(VI) metal concentration are coagulation and adsorption. This method is the most economical, simple and highly efficient method. This research uses the coagulation - adsorption integration system method, which is a combination of coagulation and adsorption methods in stages and uses the same wastewater. The purpose of this research is to see the performance of alum, $FeSO_4$ and a combination of alum: $FeSO_4$ as coagulant and fly ash as adsorbent in the removal of Cr(VI) levels in wastewater samples. Also, the efficiency of using the coagulation - adsorption integration system in the removal of heavy metal levels. The adsorption capacity value of this process can also be calculated. The resulting data were then evaluated using a spectrophotometer visible to assess the reduction of Cr(VI) concentration in wastewater. Based on the experiment, alum type coagulant with a waste and coagulant composition ratio of 2:3 and a stirring time of 60 minutes can reduce Cr(VI) levels to 14.18 mg/L from 100 mg/L. The addition of the adsorption process using fly ash helped reduce the concentration of heavy metal Cr(VI) to 12.15 mg/L and the highest efficiency value was 87.9%, and the adsorption capacity value was 0.087 mg/gram.

Limbah cair industri yang dihasilkan oleh pertambangan nikel memiliki kandungan logam berat yang tinggi, salah satu di antaranya adalah logam Cr(VI). Metode yang umum digunakan dalam menurunkan konsentrasi logam Cr(VI) yaitu koagulasi dan adsorpsi. Metode ini merupakan metode yang paling ekonomis, sederhana dan sangat efisien. Penelitian ini menggunakan metode sistem integrasi koagulasi – adsorpsi, yaitu penggabungan antara metode koagulasi dan adsorpsi secara berjenjang dan menggunakan limbah cair yang sama. Tujuan penelitian ini adalah melihat kinerja alum, $FeSO_4$ dan kombinasi alum : $FeSO_4$ sebagai koagulan dan fly ash sebagai adsorben dalam penghilangan kadar Cr(VI) dalam sampel air limbah. Serta, efisiensi penggunaan sistem integrasi koagulasi – adsorpsi dalam penghilangan kadar logam berat tersebut. Nilai kapasitas adsorpsi dari proses ini juga dapat dihitung. Data yang dihasilkan kemudian dievaluasi dengan menggunakan alat Spektrofotometer Sinar Tampak untuk menilai penurunan konsentrasi Cr(VI) dalam air limbah. Berdasarkan percobaan, koagulan jenis alum dengan perbandingan komposisi limbah dan koagulan sebesar 2 :3 dan waktu pengadukkan selama 60 menit dapat menurunkan kadar Cr(VI) hingga 14,18 mg/L dari 100 mg/L. Penambahan proses adsorpsi dengan menggunakan fly ash membantu ,menurunkan konsentrasi logam berat Cr(VI) hingga 12,15 mg/L dan nilai efisiensi tertinggi sebesar 87,9 %, serta nilai kapasitas adsorpsi sebesar 0,087 mg/gram.

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Introduction

In the last 5 years, the nickel sector in Indonesia has experienced rapid growth, evident in the establishment of many industrial estates and smelters involved in the comprehensive processing of nickel from first stages to final stages. The increasing output of liquid waste from nickel mining is precisely proportionate to this. Heavy metals are included in the liquid waste of the nickel production.

Heavy metals are non-biodegradable and potentially carcinogenic. Therefore, the presence of these metals in water at excessive levels can lead to severe health problems for living things (Nikić et al., 2023; Qasem et al., 2021a). Heavy metals accumulate throughout the environment, leading to contamination of the human body via food consumption. They induce many health consequences, such as respiratory impairment, including the development of lung cancer, gastrointestinal disturbances, hypotension, skeletal abnormalities, and many more. Moreover, the presence of metals modifies the functioning of enzymes and metabolic reactions (Costa et al., 2022). The World Health Organization (WHO) and the United States Environmental Protection Agency (USEPA) establish national and international regulations for the allowable metal content in water, measured in micrograms per liter ($\mu g\ L^{-1}$) (Costa et al., 2022). Furthermore, The heavy metals that are most widely recognized and commonly encountered are lead (Pb), zinc (Zn), mercury (Hg), nickel (Ni), cadmium (Cd), copper (Cu), chromium (Cr), and arsenic (As). While heavy metals may be identified in small amounts, they nevertheless pose a significant risk (Chow et al., 2021a; Qasem et al., 2021a; Shaker et al., 2023).

Chromium is a heavy metal that is widely disseminated in the earth's crust and usually occurs in Cr^{3+} and Cr^{6+} valence states in aqueous form (El Gaayda et al., 2023). In particular, Cr(VI) has greater solubility, and is considered as exceedingly harmful to human, whereas Cr(III) is viewed as less poisonous with a tendency to be immobilized by adsorption and precipitation across material surface locations (El Gaayda et al., 2023; Jimenez-Paz et al., 2023; Mathai et al., 2022a). Commonly utilized procedures in lowering Cr(VI) metal concentrations in wastewater are polymer resin ion exchange, coagulation-flocculation, and chemical reduction/sedimentation (Jimenez-Paz et al., 2023; Soliman & Moustafa, 2020). Adsorption and coagulation of metals in solids is the most affordable, easy and highly effective method for eliminating heavy metals present in wastewater (Costa et al., 2022; Jimenez-Paz et al., 2023).

This strategy combines the advantages of coagulation in agglomerating heavy metal particles with the advantages of adsorption in absorbing dissolved Cr(VI) (Finistyanto, 2022), and adsorption utilizing fly ash adsorbent to absorb heavy metals found in wastewater. So it is believed that this research may reduce the heavy metal content of Cr(VI) by more than 50%, the goal of the coagulation - adsorption integration system approach is expected to lower the fly ash load in absorbing heavy metal Cr(VI) once the coagulation process is complete. The research conducted by Dadebo et al., (2023)combined the chemical coagulation process and adsorption fix bed column with rice husk derived biochar as a natural adsorbent, the efficiency value of surfactant removal reached 96.6% after the coagulation - flocculation integration system. Meanwhile, research conducted by Nnaji et al, approached the coagulation - flocculation adsorption process in the removal of colour/total suspended solids (CTSS), COD and Cr(VI) metal. The efficiency values of the three were 99.2%, 90.07% and 98.29% respectively.

Therefore, in this study, the Cr (VI) metal removal process in metal industry wastewater consists of 2 (two) stages of heavy metal removal process in wastewater samples: coagulation process and adsorption process.

Method

Materials

This study uses equipment including Muffle furnace (Nabertherm b180), Spektrofotometer Visible (ICEN 350 – 1020), Oven (Cryste), Hot plate stirrer (Thermo Scientific Cimarec SP88857105), Jar test (Cyclone) and glassware in the laboratory. In addition, the materials used are fly ash, $K_2Cr_2O_7$ (Merck), HCl 2 M solution, Distilled water, $Al_2(SO_4)_3$ (Merck), $FeSO_4$ (Merck), NaOH solution, 1,5 – diphenylcarbazide (Merck), H_3PO_4 solution (Merck).

Preparation of Test Samples

The artificial waste water utilized in this investigation was generated from a solution of $K_2Cr_2O_7$ and distilled water with a total concentration of 100 mg/L.

Coagulant manufacturing

Coagulants used in this investigation included 3 (three) types such as $Al_2(SO_4)_3$, $FeSO_4$, and a combination of $Al_2(SO_4)_3$ & $FeSO_4$ in a ratio of 1:1 with a concentration of 140 mg/L.

Jar-Test Testing

The selection of the optimal coagulant is performed through the utilization of a Jar-Test apparatus, wherein different ratios of coagulant to waste composition specifically 1:2 and 2:3. The flocculation process is conducted for a duration of 60 minutes, during which the stirring speed is divided into two stages: an initial 30-minute period of rapid stirring at 100 revolutions per minute (rpm), followed by a subsequent 30-minute period of slow stirring at 60 rpm. The sedimentation duration is 30 minutes, and the pH of the solution is 9.6. The results obtained were further analyzed utilizing a Visible Spectrophotometer to determine the optimal composition of the coagulant, which would be further used in the adsorption procedure.

Adsorption process

The adsorption process incorporates system integration by selecting the optimal outcomes from the coagulation - flocculation process. Next, extract a 50 mL sample from the previous procedure and insert 5 grams of fly ash adsorbent. Two types of adsorbents were employed in this procedure: activated adsorbents and non-activated adsorbents. The time variants employed were 30 minutes, 60 minutes, 120 minutes and 150 minutes. After the adsorption procedure, the data obtained were then re-analysed using a visible spectrophotometer utilizing 1,5-diphenylcarbazide solution as a complexing solution.

Data analysis

Based on the test findings of total Cr(VI) levels that were obtained through the integration process of coagulation - flocculation and adsorption using a visible spectrophotometer instruments, the removal efficiency of heavy metal Cr(VI) may be determined using equation (1).

$$\% \, Efficiency = \frac{(C_0 - C_t)}{C_0} x \; 100\% \tag{1}$$

Where,

C_0 = initial concentration of waste water (mg/L)

C_t = waste water concentration at time (mg/L)

Furthermore, this study also determined the adsorption capability of the adsorbent in capturing colloidal particles. The adsorption capacity may be calculated by using equation (2)

$$qt = \frac{(C_0 - C_t) \; x \; V}{W} \tag{2}$$

qt = adsorption capacity at time (mg/gram)

C_0 = Initial concentration of waste (mg/L)

C_t = concentration of waste at time (mg/L)

W = mass of adsorbent (gram)

Results and Discussion

Determination of the Best Coagulant

The objective of this study is to identify the optimal coagulant type that may effectively and efficiently lower the levels of certain metals in wastewater samples. This will help reduce the workload of the subsequent adsorption process. The coagulation process involved the use of different types of coagulants, specifically alum ($Al_2(SO_4)_3$), $FeSO_4$, and a mixture of alum and $FeSO_4$ in equal proportions (1:1). The coagulation process is conducted with a jar test apparatus, with starting parameters set at a pH of 9.6 and waste to coagulant ratios of 1:2 and 2:3. The stirring procedure is separated into two stages: quick stirring at 100 rpm and gradual stirring at 60 rpm. Rapid agitation is used to ensure the uniform dispersion of the coagulant throughout the wastewater, facilitating the fast development of flocs. Meanwhile, gentle agitation is used to promote the collision of colloidal particles, leading to the formation of bigger flocs (Chow et al., 2021b; Mathai et al., 2022b; Qasem et al., 2021b).In addition, the level of heavy metal content in treated wastewater will be assessed by visible spectrophotometry to identify the most effective coagulant.

Figure 1 illustrates a substantial reduction in Cr(VI) levels in wastewater, starting from an initial value of 100 ppm. During the coagulation of chromium with ferrous sulfate ($FeSO_4$), the use of $FeSO_4$ as a coagulant results in the formation of a larger precipitate (Pangeran et al., 2023). This greater size of the precipitate aids in the sedimentation process. The coagulation process with this particular coagulant leads to the formation of bigger flocs. This is attributed to the high charge density of Fe^{3+} ions, which results in the formation of stronger inter-particle bridges. The presence of Iron Sulfate ions in liquid waste allows for their absorption onto the surface of particles, resulting in the formation of coagulant complexes that have the ability to attract other particles. When Alum is used in the coagulation process, the resultant sediment is smaller in size compared to the prior type of coagulant. The combination of alum and $FeSO_4$ as a coagulant results in smaller particles, but it generates a significantly larger amount of sediment compared to the two prior coagulants. Alum coagulant is the most effective form of coagulant for binding Cr ions. Alum is well recognized as a prevalent and efficient coagulant for addressing diverse pollutants in wastewater. The findings of Oko et al., (2022) align with this result, indicating that the coagulant $Al(OH)_3$, when used at a dosage of 30 gr / L, is the most efficient in lowering the overall chromium content in batik wastewater. The efficacy of Al(OH)3 in decreasing heavy metal chromium is attributed to the properties of $Al(OH)_3$ coagulant, a polymer coagulant with the ability to adsorb organic materials and heavy metals in synthetic waste (Oko et al., 2022; Zoroufchi Benis et al., 2020). Types of organic salts such as iron and aluminum are most extensively employed as coagulation agents in most water treatment plants throughout the world (Asharuddin et al., 2023) this is because alum is the cheapest and simplest aluminum-based flocculant accessible (Muisa et al., 2020).

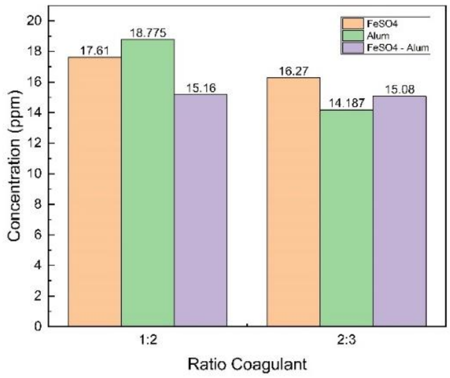

Figure 1 Determination of the best coagulant type between alum, $FeSO_4$ and Combination of $FeSO_4$ - Alum in the coagulation process.

Coagulants in the form of iron salts and aluminum salts will hydrolyze in water forming positively charged hydrocomplexes. These hydrocomplexes will form hydrometal polymers and generate $Fe(OH)_2$ and $Al(OH)_3$ precipitates, accompanied by a rise in the concentration of H^+ ions in solution. This positive charge will neutralize the negative charge on the Cr anion so that it can precipitate along with $Fe(OH)_2$ and $Al(OH)_3$. When compared between aluminum-based coagulants and iron-based coagulants, it can be noted that aluminum-based coagulants have greater performance than iron-based coagulants. This may be demonstrated from the capacity of aluminum-based coagulants to remove polluted parameters more efficiently than iron-based coagulants. This difference is due to the easier oxidation characteristics of aluminum metal ions compared to iron. Aluminum ions (Al^{3+}) are more easily released to absorb organic compounds and heavy metals, whereas iron ions (Fe^{3+}) have a reduced tendency to release (Purwaningsih et al., 2020).

Integration of Coagulation and Adsorption

The best coagulation outcomes for Cr(IV) metal ions employing alum coagulant, then further processed by adsorption technique using fly ash adsorbent without going through preceding activation step. This seeks to assess the influence of coagulation on the adsorption process by comparing the results of Cr(VI) reduction when utilizing the coagulation - adsorption process integration technique and the adsorption method.

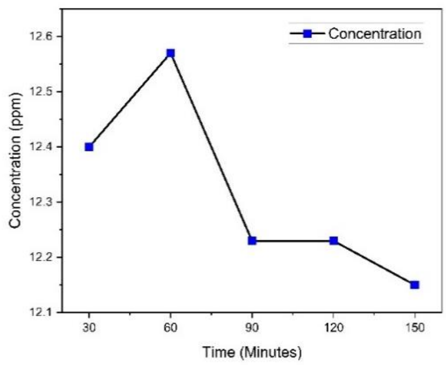

Figure 2 adsorption of Cr(VI) using fly ash adsorbent

In Figure 2, it can be observed that there was a drop in the initial Cr level following the coagulation process, specifically 14.187 ppm to 12.40 ppm during the initial 30 minutes, however there was a rise in Cr concentration levels after 60 minutes. This is due to the adsorption process that has not been optimized at the 60th minute, moreover external elements and solution conditions can also impact the release of chromium that has been adsorbed back into the solution. This discovery has been confirmed by research conducted by (Oko et al., 2022) which reveals that with time the amount of adsorbed chemicals increases, but subsequently decreases related to the adsorbent reaching saturation level. The lengthy contact period of the waste with the adsorbent in this investigation resulted in a significant decrease in Cr(VI) metal ions, since an adsorbent takes time to attain equilibrium in absorbing pollutant loads (Audiana et al., 2016). The most optimum period in lowering Cr(VI) concentration using fly ash adsorbent without activation is 150 minutes. The decrease in Cr(VI) concentration in the coagulation - adsorption integration system process did not have a significant effect because of the fly ash adsorbent used not getting through a previous activation process, which meant that metal sorption failed to perform optimally because to the absence of an increase in the porosity of the sorption material and allowed active sites for adsorption to form only a few.

Efficiency of fly ash adsorbent

The calculation of the percentage efficiency value using fly ash adsorbent without previous activation procedure is done by comparing the concentration of Cr(VI) metal in the wastewater sample after and before the integration process of coagulation and adsorption. The goal of the % efficiency value is to evaluate an adsorption procedure in absorbing Cr(VI) metal in wastewater samples efficiently. In addition, determining the efficiency of the adoption process can be interpreted as measuring the success of a process or system in achieving certain goals.

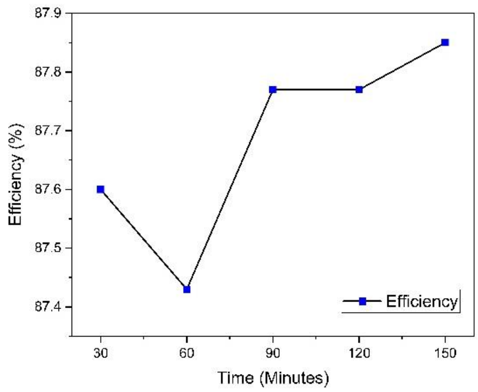

Figure 3 Efficiency of fly ash adsorbent

Based on the data obtained in Figure 3, the maximum efficiency value was reached at 150 minutes. If viewed in **Figure 3**, there is a decline in the efficiency value at time 60 minutes due to the desorption process or the release of Cr(VI) metal that has been absorbed. So that there is a rise in the concentration of Cr (VI) in the water sample and a drop in the efficiency value. Furthermore, the efficiency value grows again in the next minute, although the increase in value is not considerable. The greatest efficiency value was reached at minute 150, which is up to 87.9% of Cr (VI) metal that has been absorbed from wastewater samples utilizing the coagulation-adsorption integration technique.

Adsorption Capacity

The adsorption capacity of fly ash for Cr(VI) in polluted water was investigated with the concentration present in artificial wastewater samples as an example. All experimental parameters were kept the same to avoid the influence of experimental changes on the adsorption process (Dan et al., 2023; Wang et al., 2024). Cr(VI) solution was prepared by diluting the solids to a concentration of 100 mg/L.

Table 1 The Adsorption capacity of fly ash

Time (Minutes)	Adsorption Capacity (mg/gram)
30	0.0876
60	0.08743
90	0.08777
120	0.08777
150	0.08785

Table 1 displays the adsorption capacity value per adsorption time. There is a modest rise in the adsorption capacity value as the adsorption time increases. The greatest adsorption capacity measurement at 150 minutes was 0.087 mg/gram. This demonstrates that in the adsorption process utilizing fly ash adsorbent and the starting concentration of 100 mg/L waste and 5 grams of adsorbent mass, fly ash is only able to adsorb the maximum amount of Cr (VI) less than 1 mg/gram. This is due to numerous variables, one of which is that the adsorbent utilized does not carry out the activation process initially. So that it does not enhance the active groups on the adsorbent, and the specific surface area and pore size of the adsorbent are not big enough with varied pore sizes.

Conclusion

According to the conducted study, it can be inferred that alum is the most effective coagulant for reducing the concentration of the heavy metal Cr(VI). It was observed that alum decreased the levels of Cr(VI) in wastewater samples up to 14.187 ppm from an initial concentration of 100 ppm, with a comparative ratio of 2:3. The implementation of the coagulation-adsorption integration system utilizing fly ash adsorbent without prior activation procedure yielded insignificant results, with a concentration of just 12.14 ppm. The maximum efficiency figure achieved was 87.85% and the adsorption capacity value was 0.087 mg/gram.

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