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Research Article

Effect of Flow Rate on Hexavalent Chromium Reduction in Electroplating Wastewater Using TiO_2 Immobilized Resin

Pengaruh Debit Aliran terhadap Penyisihan Cr^{6+} pada Limbah Elektroplating Menggunakan Resin Immobilized TiO_2

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Abstract

This study aims to analyze the effect of flow rate on the reduction of hexavalent chromium (Cr^{6+}) in electroplating wastewater treated with a TiO_2-immobilized resin in a continuous fixed-bed reactor. The electroplating wastewater, characterized by Cr^{6+}, was treated with RIPT-TiO_2 functioning as a catalyst to enhance the contaminant degradation process. The flow rate was then examined to observe the effect on the reduction of Cr^{6+} concentration during the reaction. The results showed that increasing the flow rate within the range of 40–70 mL/min affected the reduction of Cr^{6+}, and ultimately influenced the photocatalytic efficiency. The flow rate affected the contact time between the wastewater and the catalyst. The best removal efficiency, reaching 50.83% at 180 minutes, was obtained at a lower flow rate. This study provides important contributions to the development of sustainable and efficient electroplating wastewater treatment technology using a continuous reactor system with a TiO_2-immobilized resin.

Keywords: Cr^{6+} reduction; flow rate; electroplating; photocatalyst; TiO_2

Abstrak

Penelitian ini bertujuan menganalisis pengaruh debit aliran terhadap penurunan Cr^{6+} limbah elektroplating yang diolah menggunakan resin terimmobilisasi TiO_2 dalam reaktor kontinu fix bed. Limbah elektroplating yang memiliki karakteristik Cr^{6+} diolah dengan RIPT-TiO_2 yang berfungsi sebagai katalis untuk meningkatkan proses degradasi kontaminan. Debit aliran diuji untuk melihat efeknya terhadap penurunan kadar Cr^{6+} selama reaksi berlangsung. Hasil penelitian menunjukkan bahwa peningkatan debit aliran (dalam rentang 40-70 ml/menit) mempengaruhi penurunan kadar Cr^{6+} yang dapat mempengaruhi efisiensi fotokatalis. Debit aliran mempengaruhi waktu kontak antara limbah dengan katalis. Pada debit aliran 40 ml/menit diperoleh hasil penyisihan terbaik, yaitu mencapai 50,83% pada menit ke-180 menit. Studi ini memberikan kontribusi penting dalam pengembangan teknologi pengolahan limbah elektroplating secara berkelanjutan dan efisien menggunakan sistem reaktor kontinu berbasis resin immobilized TiO_2.

***Kata kunci**: debit aliran; elektroplating; fotokatalis; reduksi Cr^{6+}; TiO_2*

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1. Introduction

Wastewater from the electroplating industry is considered a hazardous waste due to the various heavy metal content, including chromium (Cr), nickel (Ni), copper (Cu), and zinc (Zn), as well as unstable pH [1]. Several studies have shown that the Cr^{6+} levels of untreated electroplating wastewater can range from 2 mg/L to 5 mg/L, depending on the type of process and chemicals applied [2]. Electroplating wastewater also produces varying pH conditions, ranging from very acidic conditions (e.g., pH 3.2) to highly alkaline (pH 12.7 to 13.8), depending on the type of plating process used [3]. A study conducted on electroplating wastewater at PT. DI, reported that the wastewater temperature ranged from 25°C to 26°C, while other studies have shown a temperature increase of up to 2°C during processing, capable of affecting reaction efficiency [4]. Disposal of wastewater without proper treatment has the potential to pollute the environment, endanger human health, and damage aquatic ecosystems. Therefore, developing and implementing efficient, effective, and sustainable processing technology is necessary.

Various wastewater treatment methods have been developed, ranging from chemical precipitation [5] to ion exchange [6] and adsorption [7]. However, these methods often have drawbacks, such as producing toxic sludge or high operational costs [8]. Recently, photocatalysts have been widely used as a technology that can remove pollutants in an environmentally friendly manner. In photocatalytic reactions, catalyst materials function to accelerate the rate of chemical reactions with the help of energy from light [9]. In photocatalysis, light energy is used to activate the catalyst, which then interacts with the substrate to produce the desired product. The photocatalytic process can degrade organic pollutants and reduce heavy metal ions to less toxic forms under exposure to ultraviolet (UV) light [10].

Despite the benefits, the application of photocatalytic technology on an industrial scale still faces challenges. One of these is the separation of the catalyst from the solution after the treatment process. An effective solution to this problem is using resin media as a photocatalyst support. By binding the photocatalyst to the resin, the separation process is facilitated, and reactor efficiency can be increased. Furthermore, continuous reactor systems offer advantages in scalability and operational efficiency compared to batch systems, enabling the continuous treatment of large volumes of wastewater [11].

Titanium dioxide (TiO_2) is a semiconductor widely known as a photocatalyst with the ability to degrade organic pollutants and reduce heavy metal ions [12]. However, TiO_2 in powder form is often difficult to separate from wastewater after processing, resulting in low efficiency. To overcome this problem, TiO_2 can be immobilized or attached to a supporting medium, such as resin.

Resin Immobilized Photocatalyst Technology (RIPT) is a technology that integrates photocatalysts with ion exchange resin [13]. In this system, photocatalysts are bound in the resin, enabling more efficient and effective wastewater treatment. The technology provides benefits in terms of ease of separation and wastewater treatment, as well as increasing the ability to remove pollutants [14]. In a previous study by Ayuningtyas et al. [11], a Resin

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Immobilized Photocatalyst was used to remove BOD, resulting in a final BOD concentration of 69.41 mg/L from an initial 707.97 mg/L. Another study by Zahrah et al. [15] used a similar method to remove Cr^{6+}. However, the study was conducted in a batch system and obtained the best removal percentage of 96%.

One of the important parameters that affects the performance of a continuous reactor is the flow rate. This parameter determines the wastewater residence time in the reactor, which directly affects the interaction between the wastewater, the photocatalyst, and UV radiation. In addition, the flow rate also affects the Cr^{6+} content of the wastewater during the process [16]. The reduction in Cr^{6+} content is crucial because it potentially changes the kinetics of the photocatalyst reaction and the stability of the resin media. Presently, studies specifically examining the effect of flow rate on the Cr^{6+} content of electroplating wastewater in a continuous photocatalyst reactor using resin media remain limited.

Based on the description above, this study aims to explore in depth the effects of flow rate variations on the removal of Cr^{6+} from electroplating wastewater in a continuous photocatalyst reactor using RIPT-TiO_2 media. The results are expected to provide a better understanding of the optimization of reactor operational parameters for increasing the effectiveness of electroplating wastewater treatment and contribute to the development of more efficient and sustainable technologies.

2. Research Method

2.1 Tools and materials

This study required several tools in a continuous reactor system, including essential components such as a DH-1000 model pump with a capacity of 350 liters/hour, a 15-watt UV-C lamp, a UV-Vis spectrophotometer, a pH meter, and 200-mesh filter paper.

The materials used include electroplating wastewater obtained from an electroplating industry in Padeg Village, Cerme District, Gresik Regency, Dowex cation resin, a rutile-type TiO_2 catalyst with a purity of 98%, and distilled water.

2.2 Preparation

The preparation phase started by ensuring the optimal quality of the initial wastewater, which had been characterized and prepared. Initial wastewater characteristics were measured to serve as the study baseline data, including the Cr^{6+} content and pH of the electroplating wastewater samples. Subsequently, the wastewater was diluted with distilled water using a 1:1 ratio. This dilution procedure is a crucial pretreatment aimed at preventing clogging in the reactor and ensuring the feed solution maintains a consistency that allows for smooth, even flow throughout the system.

Once the initial wastewater was prepared, the immobilized RIPT-TiO_2 catalyst was prepared, weighing 50 g. The components of the continuous flow reactor system were then assembled. Pumps were installed to control fluid flow, while UV-C lamps served as the energy source to activate the photocatalysis process. To secure the catalyst within the reactor during testing, 200-mesh filter paper was installed to prevent the immobilized resin granules from being carried away with the

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wastewater stream. Once all preparation and assembly were complete, variations in operational parameters such as flow rate were then tested under UV-C irradiation.

2.3 Data collection on Cr^{6+} concentration, pH, and temperature

The continuous reactor was designed with gravity flow to ensure stable wastewater flow. The diluted electroplating wastewater was placed in the reactor initial tank. The wastewater flowed into the reactor box containing RIPT-TiO_2 and a UV-C lamp. Before entering the reactor box, the flow rate was regulated using a valve. In this study, the flow rate used were 40, 50, and 70 mL/min. After passing through the reactor box, the processed wastewater flowed into the final tank. During this process, samples were taken at intervals of 10, 30, 60, 120, and 180 minutes.

Cr^{6+} levels were measured as the primary parameter in the effluent samples, followed by pH and temperature (supporting parameters). Specifically, Cr^{6+} levels were measured using a UV-Vis spectrophotometer at a wavelength of 540 nm, while pH and temperature were measured using a pH meter equipped with a sensor. The pH and temperature data for analysis were obtained throughout the Cr^{6+} reduction process to observe trends in changes. This analysis is crucial for understanding the effect of flow rate on process effectiveness. Data comparison was conducted to observe specifically how flow rate affects changes in Cr^{6+} levels in wastewater, as well as the effect on the quality of the wastewater produced.

3. Results and Discussion

3.1 Characteristics of Electroplating Wastewater

To determine the effect of flow rate on the Cr^{6+} content in a continuous photocatalyst reactor, a test was conducted on the characteristics of electroplating wastewater located in Padeg Village, Cerme District, Gresik Regency. Based on the quality standards of the Minister of Environment (MLHK) Regulation No. 5 of 2014, the Cr^{6+} content should meet the quality standard of 0.1 mg/L to be discharged into water bodies. Table 1 shows the results of the electroplating wastewater characteristics test.

Table 1 shows a Cr^{6+} content of 1090.8 mg/L with a pH of 3.4 (acidic). These results show that the Cr^{6+} content in electroplating wastewater does not meet the quality standard limit of 0.1 mg/L stipulated in the Minister of Environment Regulation No. 5 of 2014. Therefore, it should be treated before being discharged into water bodies.

Table 1. Electroplating wastewater characteristics test results

Parameter	Unit	Test Results	Quality standards	Source
Cr^{6+}	mg/L	1090.8	0.1	Minister of Environment Regulation No. 5 of 2014
pH	-	3.4	6-9	Minister of Environment Regulation No. 5 of 2014
Temperature	°C	25.7	-	-

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The high Cr^{6+} content and low pH (acidic) in electroplating wastewater are characteristics directly derived from the chemical raw materials used in the plating process. Electroplating is generally carried out using the compound Chromium (VI) Oxide (CrO_3), which forms chromic acid (H_2CrO_4) when dissolved in water. These properties of CrO_3 and chromic acid cause two main issues, namely high concentration of Cr^{6+} (highly soluble and toxic in water), and the very low pH due to the release of H^+ ions. This acidic condition is often worsened by the use of other strong acids (H_2SO_4 and HCl) in the process of measuring Cr^{6+} through spectrophotometry [17].

3.2 Effect of Flow Rate on Cr^{6+} Removal in Continuous Photocatalyst Reactor

Based on the data in Figure 1, the three discharge variations experienced an increase in the percentage of Cr^{6+} removal in the electroplating wastewater as the sampling time increased. The highest removal efficiency was observed at 180 minutes with a discharge rate of 40 mL/min, achieving a removal efficiency of 50.83%, a final Cr^{6+} concentration of 536.4 mg/L, and a pH of 0.6. These results are in line with a previous study by Nurjanah [18], where the best Cr^{6+} removal was obtained at the longest time, namely 120 minutes, with a removal efficiency reaching 95%. However, the results obtained differ from previous studies because the reference used a batch system where the contact time of the pollutant with RIPT/UV light was longer.

The longer the exposure of Cr^{6+} to the catalyst and UV light source, the greater the possibility of the photocatalytic reaction [19]. However, the Cr^{6+} and pH levels in the electroplating wastewater processed using RIPT-TiO_2 still do not meet the quality standards set out in Minister of Environment Regulation No. 5 of 2014, namely a Cr^{6+} content of 0.1 mg/L and a pH value of 6-7. This shows that for industrial applications, pretreatment and the addition of a neutralization unit are necessary after the photocatalytic process.

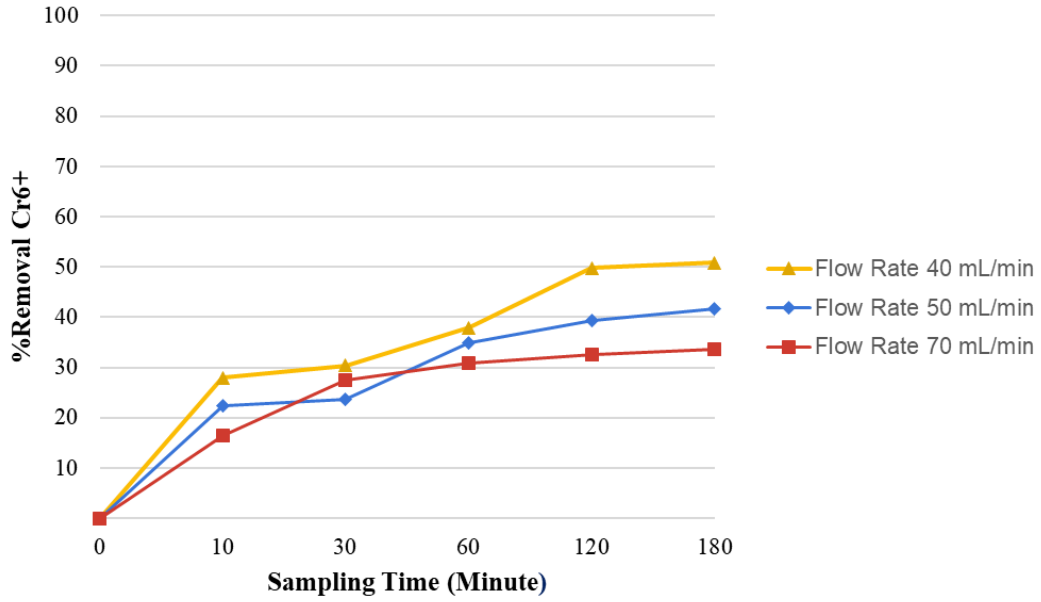


Figure 1. Graph of the effect of flow rate on Cr^{6+} removal

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In the photocatalytic reaction, electron-hole pairs on the surface of the photocatalyst react with radicals in water to reduce Cr^{6+} to the less toxic Cr^{3+} or oxidize other organic compounds [20]. As shown in Figure 1, the flow rate of 40 mL/min using RIPT-TiO_2 produced the highest removal percentage of 50.83%, followed by 50 mL/min and 70 mL/min. This is in accordance with Hifdillah analysis [21], stating that the smaller the flow rate, the longer the contact time between the wastewater and the resin, thereby maximizing the ion exchange process. Low flow rate provides a long contact time for the UV lamp to irradiate RIPT-TiO_2 and produce many hydroxyl radicals as well as superoxide anions that will reduce the Cr^{6+} level [22]. High flow rate, on the other hand, provides a short contact time. Consequently, only a few radicals are produced, and the removal results are less than optimal.

At low flow rate, the solution residence time in the adsorbent column is longer, even though the mass transfer rate of Cr^{6+} molecules to the catalyst surface is slower. This longer residence time provides more opportunities for the molecules to diffuse and interact with the adsorbent surface [23]. Meanwhile, at high flow rate, although turbulence can increase the initial mass transfer, the very short residence time does not provide enough time for the Cr^{6+} molecules to experience complete reduction before leaving the reactor [24].

RIPT-TiO_2 uses the photocatalytic properties of TiO_2 activated by UV radiation in the wavelength range of 275 nm to 405 nm, which provides the energy needed to initiate the catalytic process [25]. In this study, a UV-C lamp was used because it has the lowest wavelength between UV-A and UV-B, which is around 200-280 nm. The shorter the wavelength of UV-C light, the greater the ability to degrade pollutants [26]. When exposed to UV light, electrons in TiO_2 are released and move to the conduction band, leaving empty "holes" in the valence band. Reactive electron holes (h^+) produce hydroxyl radicals (·OH), while excited electrons (e^-) reduce Cr^{6+} ions to Cr^{3+} [27].

When the number of TiO_2 particles successfully immobilized on the resin is insufficient, the number of active photocatalytic sites to interact with UV light and reduce Cr^{6+} will be very limited. Consequently, the photocatalytic reaction is limited, and the expected removal of Cr^{6+} from RIPT-TiO_2 is not achieved optimally.

3.3 pH of Wastewater During the Photocatalytic Process of Cr^{6+} Removal

Based on the data in Figure 2, the best Cr^{6+} removal percentage was found at a flow rate of 40 mL/minute of 50.83%, with a pH value of 0.6. This is in accordance with Slamet [28], who reported that photogenerated holes on the surface of TiO_2 react with water (H_2O) to form hydroxyl radicals (·OH) while releasing H^+ ions. In the presence of other organic contaminants in the electroplating waste, oxidation may occur to produce mineral or carboxylic acids, which can lower the pH.

This drastic decrease in pH is caused by an oxidation reaction triggered by the holes (H^+) in TiO_2. When the waste contains reduced Cr species (Cr^{3+}), high-energy UV potentially causes the reverse oxidation of Cr^{3+} to Cr^{6+} on the catalyst surface, as shown in the reaction equation.

$$Cr^{3+} + H^+ + H_2O \rightarrow CrO_4^{2-} + H^+$$

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Under acidic conditions, CrO_4^{2-} is in equilibrium with $HCrO_4^-$, which is a species of chromic acid (H_2CrO_4), a strong acid. The formation of this acid will continue to release H^+ ions into the solution [29].

During the photocatalytic process using RIPT-TiO_2, the pH of the waste was significantly reduced to below 1. This phenomenon can be explained through the photocatalytic reaction mechanism. TiO_2 as a photocatalyst is activated by UV light, which causes electrons to jump from the valence band to the conduction band, leading to the formation of positive holes (h^+) [30]. These holes then react with water molecules to form hydroxyl radicals (·OH), which are strong oxidants in decomposing organic compounds in the waste. Through this oxidation process, organic compounds are degraded into end products such as carbon dioxide (CO_2) and water (H_2O)[31].

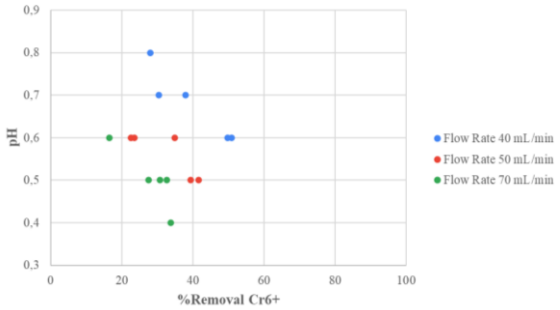


Figure 2. pH during the photocatalytic process of Cr^{6+} Removal

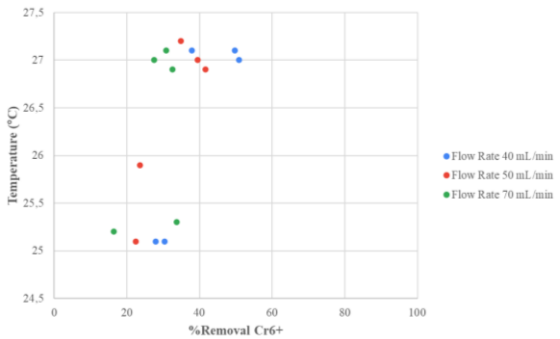


Figure 3. Temperature during the photocatalytic process of Cr^{6+} removal

The CO_2 formed dissolves in water and reacts to form carbonic acid (H_2CO_3), which causes the pH of the wastewater to drop to a very acidic level. In addition to carbonic acid, bicarbonate (HCO_3^-) and carbonate (CO_3^{2-}) ions are formed during this process and contribute to the pH decrease [32].

When the degradation of organic compounds occurs intensively, and the formation of acid products exceeds the initial buffer capacity of the waste, the pH can decrease significantly to below 1. Therefore, the sharp decrease in pH after the photocatalytic process with RIPT-TiO_2 is due to the formation of acidic products from the degradation of organic compounds in the waste [33].

3.4 Wastewater Temperature During the Photocatalytic Process of Cr^{6+} Removal

Based on the data in Figure 3, the best Cr^{6+} removal percentage was found at a flow rate of 40 mL/minute, namely 50.83% at a temperature of 27°C. Temperature plays a role in increasing the energy of photons produced in the photocatalytic process [34].

At high temperatures, photocatalytic activity typically increases due to more efficient electron transfer and the formation of more hydroxyl radicals. The greater the radiation energy absorbed by a material, the higher the temperature. This occurs because the energy causes the particles that make up the material to vibrate and move faster [26]. At high flow rate, the waste entering the reactor at a rapid rate acts as an effective cooling medium. The continuous flow of waste absorbs and carries away the heat generated by the UV lamp. Conversely, at low flow rate, the waste residence time in

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the reactor is longer. Given that the volume of fluid flowing is low, the cooling capacity is drastically reduced [35].

The results show that although the flow rate does not directly generate heat, it plays a crucial role in controlling and dissipating the heat generated by the light source. Therefore, flow rate indirectly affects the reactor efficiency and operating temperature [35].

4. Conclusion

In conclusion, a flow rate of 40 mL/minute provided the highest Cr^{6+} removal percentage, reaching 50.83%, with a final Cr^{6+} content of 536.4 mg/L and a pH of 0.6. However, both parameters still fall short of the quality standards stipulated in the Minister of Environment Regulation No. 5 of 2014, which require a Cr^{6+} content of 0.1 mg/L and a pH of 6-7. This implies that for industrial applications, pretreatment and the addition of a neutralization unit after the photocatalytic process are necessary.

Industrial applications using a fixed-bed reactor and RIPT-TiO_2 media are highly efficient. The fixed-bed design naturally achieved the required hydraulic residence time. The greatest advantage is eliminating the need to separate the catalyst after the photocatalytic reaction, significantly reducing operational and maintenance costs. This system is ideal as a final polishing unit because it can reduce Cr^{6+} while simultaneously lowering COD without producing excessive chemical sludge.

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