

Article

Reliability Treatment of Silicon in Oilfield Wastewater by Electrocoagulation

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Abstract: Scaling caused by silicate in oilfield wastewater gathering system pipelines can cause serious pipeline blockage. Therefore, this study adopts facile, effective and environment friendly electrocoagulation method to remove the silicon in oilfield wastewater. After confirming the level of factors through single factor experiments, the optimal scheme for electrocoagulation was selected by orthogonal experiments and verification tests, the silicon content would be dramatically decreased from 81.51 mg/L to 21.88 mg/L when pH = 6, reaction time = 20 min, current density = 27 mA/cm² and wastewater temperature = 35 °C. In addition, the silicon removal rate would reach up to 85.90% when the pH of oilfield wastewater was kept as its original condition without changing other optimal factors; such an enhanced silicon removal effect could be attributed to the calcium ions chemical coagulation after the mechanism investigation.

Keywords: high-silicon wastewater treatment; electrocoagulation; calcium ion; orthogonal experiment



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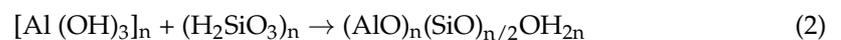
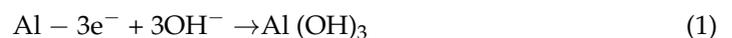
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1. Introduction

The recovery and reutilization of oilfield wastewater has attracted the attention of researchers, especially in oil-producing countries where water resources are scarce [1]. Among this research field, scaling caused by silicon in gathering system pipelines can cause severe pipeline blockage, which is an enduring research hotspot [2]. The silicon-containing wastewater mainly comes from the steam throughput or steam drive extraction technology, which is mostly used in the development of thick oil. A large amount of steam is injected into the formation while the silicon rocks under the formation will be depolymerized by hydrolysis to generate silicates, resulting in massive silicon-containing wastewater [3]. In sewage, silicon exists in three forms. (1) As dissolved silicon, it mainly exists in the form of metasilicic acid (H₂SiO₃) and a certain amount of orthosilicic acid (H₄SiO₄) and its ionized H₃SiO⁴⁻ and H₂SiO₄²⁻ plasma; (2) as colloidal silicon, the dissolved silicon is inter-polymerized in supersaturated solution to form polymerization products composed of polysilicic acid, such as di-polysilicic acid and tri-polysilicic acid, and forms colloidal silicon through condensation and dehydration. (3) As particulate silicon, it is mainly composed of sand and all kinds of suspended silicon-contained substances in water [4,5]. As for the formation mechanism of silicon scale, on the one hand, SiO₃²⁻ from dissolved silicon could self-polymerize and eventually generate hard silica crystals. On the other hand, calcium or magnesium ions in the wastewater would transform as hydroxide and precipitate with the colloidal silicon [6]. These two types of precipitations are most likely to form mixed silicon scale, which eventually leads to blockage in the oilfield recovery system. Among the current common methods for treating silicon-containing wastewater [7], chemical flocculation requires additional flocculants and also produces a large amount of sludge, which is not conducive to subsequent solid–liquid separation [8]. Reverse osmosis is another method of removing silicon from the wastewater, which has the advantage of

high filtration accuracy, but the disadvantage is that half or more of the water is wasted. At present, in order to make it so that the reverse osmosis method can be applied to remove the silicon in wastewater with a large scale, researchers have proposed many methods of membrane modification, such as surface chemical modification, layer self-assembly, and so on [9,10]. Ultrafiltration is effective for the removal of large diameter colloidal silicon and particulate silicon in sewage, but it cannot remove dissolved silicon in wastewater [11]. However, the above two kinds of membrane methods cannot be used for a long time due to the damage of silicon scale to the membrane life [12]. The ion exchange method is only effective for the removal of dissolved silicon in wastewater and has no effect on the colloidal silicon and particulate silicon. In addition, the ion exchange method has high requirements for water quality, so the wastewater must be pretreated before the ion exchange method treatment [13,14]. The flotation method is only effective for colloidal and particulate silicon and cannot remove the dissolved silicon, so it is necessary to use other methods to convert dissolved silicon into colloidal or particulate silicon. In order to remove silicon in wastewater more economically and effectively, a new process with higher silicon removal rate is urgently needed. In recent years, there has been an increasing number of studies on applications of electrocoagulation as an environmental-friendly technology to remove the silicon in wastewater [15,16]. However, not enough researches have focused on silicon in oilfield wastewater [17,18], which is very important in oilfield production and chemical engineering. Therefore, it is very necessary to systematically study the removal of silicon in oilfield wastewater through electrocoagulation and consider the requirements of environmental protection, economy and industrialization.

The principle of silicon removal by electrocoagulation is mainly under the action of an electric field, where an active anode (such as aluminum) to produce a large number of metal ions. After generation of hydrolysis at the anode, these ions would form hydroxides (Equation (1)). As for dissolved silicon, the aluminum hydroxide in the solution further aggregates itself to form as polymer, and eventually, it forms a stable hydroxy-aluminosilicate with the metasilicic acid (Equation (2)), preventing the metasilicic acid converting to colloidal silicon and particulate silicon and eventually forming silicon scale when conditions change. As for colloidal silicon and particulate silicon, the aluminum hydroxide could chemically coagulate with other ions, colloids and other pollutants in the wastewater. Finally, hydroxy-aluminosilicate and coagulation would transform as hydroxide floc, which could remove silicon from wastewater effectively [19–21]. The advantages of this method are no extra-addition of chemical reagents, negligible amount of sludge, controllable silicon removal rate, convenient operation and simple equipment [22–25].



In this paper, orthogonal experiments and verification tests were implemented to investigate the optimal scheme and mechanism for electrocoagulating the silicon, especially for SiO_3^{2-} and colloidal silicon, in Hongshan Oilfield wastewater. First, the single factor experiments were carried out to study the influence of the initial pH, current density, reaction time and wastewater temperature on the silicon removal rate, which could confirm the level of each factor in the subsequent orthogonal experiments. Then, the optimal scheme was verified through orthogonal experiments from rough range in this multi-factor and multi-level research. Meanwhile, the silicon removal effect was explored through the implementation of confirmatory tests under the optimal scheme. Last but not least, the optimal scheme was optimized and the silicon removal mechanism was analyzed towards practical high-silicon-content Hongshan oilfield wastewater. This paper aims to find the optimal reaction conditions of removal of silicon in oilfield wastewater through electrocoagulation and paves the way to apply this environmentally-friendly and economic method in the practical oilfield production and chemical engineering industries.

2. Materials and Methods

2.1. Material and Reagent

The investigated high silicon wastewater comes from the Hongshan Oilfield in Karamay, Xinjiang, China, and the silicon content in the wastewater (without special explanation, the following silicon content is calculated as silicon dioxide) is as high as 83.69 mg/L, and the specific concentrations of other ions are listed in Table 1. If the wastewater plans to be reused as boiler feed water, process and product water, then the silicon content must be controlled less than or equal to 30 mg/L after treatment.

Table 1. Ion contents of wastewater from the Hongshan Oilfield.

Chemicals	Value
pH	8.08
HCO_3^- (mg/L)	466.09
Cl^- (mg/L)	19,126.29
Ca^{2+} (mg/L)	453.04
Mg^{2+} (mg/L)	167.00
SiO_3^{2-} (mg/L)	628.20
$\text{K}^+ + \text{Na}^+$ (mg/L)	12,048.84
Mineralization degree(mg/L)	32,889.46
Density (g/cm^3)	1.02
Silicon (mg/L)	83.69

The chemical reagents used in the process of water quality analysis meet the requirements of each index testing method. Hydrochloric acid (0.1 mol/L) and sodium hydroxide (0.1 mol/L) were applied to adjust the pH of wastewater, and the reagents used were analytically pure and bought from China National Pharmaceutical Group Corporation.

2.2. Experimental Setup

As shown in Figure 1, the reactor part of the experimental setup is a beaker with volume of 250 mL, and the upper part is an electrocoagulation device, in which the anode is a polished aluminum sheet and the cathode is a platinum electrode with an effective area of 5 cm², and the cathode and anode are connected by MP1005D (Maihao Electronic Technology Co., LTD, Dongguan, China) type adjustable regulated constant current power, while the wastewater served as electrolyte. Throughout the experiment, the beaker is in a constant temperature (30–70 °C) water bath.

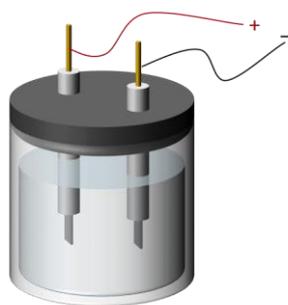


Figure 1. Schematic diagram of electrocoagulation setup.

2.3. Experimental Method

First, the silicon containing wastewater was prepared according to the silicon content of the wastewater from the Hongshan Oilfield. After insulation treatment and pH adjustment (pH was tested by pH-100, Tuohe Electromechanical Technology Co., Ltd., Shanghai, China), samples were taken for silicon determination. Second, the aluminum sheets were polished and soaked in hydrochloric acid to remove the oxide layer. After drying and

weighting, the aluminum sheets were applied as an anode while the platinum electrode served as a cathode. The current was set from 0.05 A to 0.2 A, and the reaction time was controlled from 10 min to 30 min. Finally, the aluminum sheet was dried and weighted after electrocoagulation while the treated wastewater was filtered, and samples were taken for the determination of silicate.

Subsequent experiments on simulated wastewater only need to use $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ and KHCO_3 to prepare simulated wastewater according to the content of ions in Hongshan Oilfield wastewater, and repeat the other steps mentioned above.

2.4. Analysis Method

According to GB/T 12149-2017 "Determination of silica in industrial circulating cooling water and boiler water", UV-6000 (Yuanxi Instrument Co., Ltd., Shanghai, China) UV-Visible spectrophotometer was used for the determine the silica concentration in this experiment through the silica-molybdenum blue photometric method. Detailed analysis steps could be seen in supporting information. Each sample was tested three times, and the average value was taken as the final result.

Before and after the electrocoagulation, the silicon content of the wastewater was measured, and the silicon removal rate was calculated with the following formula; silicon removal rate (%) = $(C_0 - C_t)/C_t$, C_0 represents the silicon content before electrocoagulation test (mg/L), and C_t represents the silicon content after the electrocoagulation test (mg/L).

3. Results and Discussions

3.1. Single Factor Experiment

The initial pH has a significant effect on the silicon removal effect. It can be seen in Figure 2a that the silicon removal rate was first increased and then decreased with the increase of pH; the silicon removal rate reached the top as 79.35% when pH was 6.11. A different pH and different concentration of hydroxide would result in the different morphology of aluminum hydroxide. When the solution is acidic, the main products in the solution are $\text{Al}(\text{OH})^{2+}$ and $\text{Al}(\text{OH})^{2+}$; while the solution is alkaline, the main hydrolytic products are $\text{Al}(\text{OH})^{4-}$. In other words, the existence form of alumina hydroxide flocs in the solution is closely related to the pH of the solution because the initial pH of the solution directly determines the existence form of alumina hydroxide in the initial reaction stage [16,26]. In addition, the solubility of silicon in water is greatly affected by pH. When pH is greater than 9.5, a small increase in pH can lead to a substantial increase in the solubility of silicon in water. When the solution pH is greater than 8.0 but less than 9.5, the solubility of silicon increases slightly with the increase of temperature. When the solution pH is greater than 6.0 but less than 8.0, the solubility of silicon is almost constant with the increase of temperature [26,27].

The silicon removal effect is also affected by current density. As depicted in Figure 2b, the silicon removal rate was boosted when the current density was increased from 10 mA/cm^2 to 20 mA/cm^2 , and the silicon removal rate was remained unchanged at around 93% when the current density was 40.4 mA/cm^2 , such phenomenon was accorded with related research. According to Faraday's law of electrolysis, the amount of aluminum dissolved by the anode is directly proportional to the current density. With the increase of current density, the number of aluminum ions in solution and the volume of aluminum hydroxide was augmented, which raised the silicon removal rate as a result. However, too high of a current density would lead to extra cost and little enhancement of the silicon removal effect [28,29].

The influence mechanism of the reaction time on the silicon removal effect is similar to that of the current density. As shown in Figure 2c, the silicon removal rate increased with the extension of reaction time. The silicon removal rate was increased rapidly in the first 15 min and gradually slowed down after 15 min. With the raising of reaction time, the aluminum ions in solution and the aluminum hydroxide floc volume would increase,

which leads to the enhancement of silicon removal. However, too long reaction time would result in growth of operation cost due to long voltaic time [30].

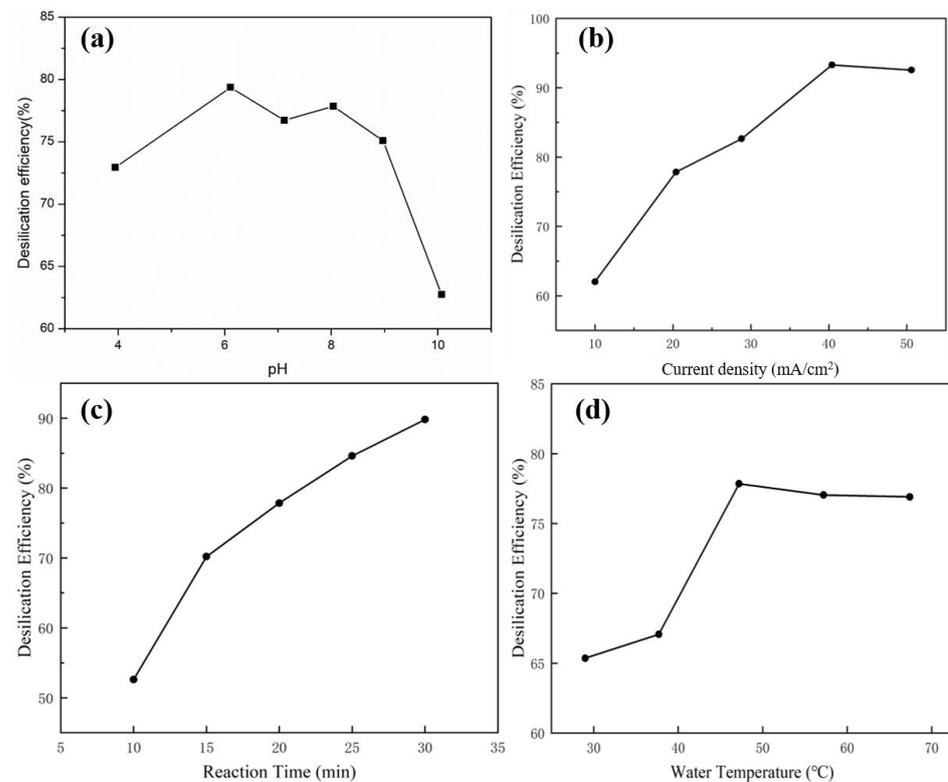


Figure 2. The impact of initial pH (a), current density (b), reaction time (c) and wastewater temperature (d) on silicon removal rate.

As for the impact of wastewater temperature on silicon removal effect, a high temperature would boost the Brownian motion and improve the collision probability of silicon ion and aluminum ion, which would accelerate the generation of flocculation. As the temperature rise further, too high of a temperature could decrease the structural stability of flocculants according to the DLVO (Derjaguin-Landau-Verwey-Overbeek) theory [31]. Wastewater temperature also affects the solubility of silicon; the solubility of silicon increases gradually with the increase of temperature. Sheikholeslami and Tan demonstrated that the solubility of silicon increases with increasing temperature in the range of 20 °C to 50 °C. However, there are two problems with too high temperature. On the one hand, inter-polymerization of the dissolved silicon would be accelerated, which is not conducive to silicon removal. On the other hand, the solubility of calcium carbonate decreases with increasing temperature, and calcium carbonate could provide crystal nuclei for the silicon scale and accelerate the formation of the silicon scale [27]. In this experiment, the silicon removal rate reached the highest value of 77% at 50 °C (Figure 2d).

3.2. Orthogonal Experiment

Through the above single factor experiments for initial pH, current density, reaction time and wastewater temperature, the optimal ranges of the four influencing factors were obtained on the premise of ensuring an appropriate silicon removal rate. Furthermore, orthogonal experiments were implemented to find the best experimental scheme from such a rough range in this multi-factor and multi-level study. In summary, the four factors of pH, current density, reaction time and wastewater temperature were selected as the main parameters of the orthogonal experiments with the silicon removal rate, silicon content after degradation and anode mass loss as evaluation indexes, and the specific levels of each factor are listed in Table 2.

Table 2. The level of each parameter.

pH	Current Density (mA/cm ²)	Reaction Time (min)	Wastewater Temperature (°C)
5	18	14	35
6	21	17	40
7	24	20	45
8	27	23	50

According to the design rules of the orthogonal experiment statement heading, the smallest table should be selected when the number of factors is less than or equal to the number of columns of the orthogonal table and the number of factor levels is consistent with the orthogonal table. In this experiment, the interaction between factors is not considered, and four factors and four levels of orthogonal experiments have been determined, and an empty column, as the error column, should be preferably needed in the orthogonal experiment table. Therefore, the five-factor, four-level orthogonal table $L_{16}(4^5)$ was finally chosen [32]. According to the experimental conditions provided by the orthogonal table, the electric flocculation silica removal experiments were implemented (Table S1).

Considering the evaluation indexes of the silicon removal rate (Table S2), silicon content after degradation (Table S3) and anode mass loss (Table S4), the theoretical optimal scheme of the orthogonal experiment was verified as pH = 6, reaction time = 20 min, current density = 27 mA/cm² and wastewater temperature = 35 °C (Table 3), which could economically make silicon-containing wastewater meet the national discharge standard under the mild reaction conditions and within a limited reaction time. Based on the above theoretical optimal scheme of the orthogonal experiment, a verified experiment was conducted and the results are shown in Table S5. The silica removal effect is better than any data in the orthogonal table, the silicon removal rate reached up to 73.16%, and the post-experimental silicon content was 21.88 mg/L (below the national regulation 30 mg/L), which proves that this scheme is indeed the optimal scheme for this orthogonal experiment.

Table 3. Optimal scheme of the orthogonal experiment.

Influencing Factor	Evaluation Indexes	Silica Removal Rate	Post-Experimental Silica Content	Anode Mass Loss	Theoretical Optimal Scheme
pH		6	6	8	6
Current Density (mA/cm ²)		27	27	18	27
Reaction time (min)		23	23	14	20
Temperature (°C)		40	35	50	35

3.3. Changes in Silicon Scale during Electrocoagulation

In order to further investigate the change of silicon scale particles during the process of silicon removal by electrocoagulation, under the above theoretical optimal scheme, only the reaction time was changed to 10 min, 20 min and 30 min, and the flocs after each experiment was taken and the size of silicon scale particles were observed under a 20 × microscope. Meanwhile, blank groups were set up, the silicon in the wastewater was replaced by sodium sulfate while other conditions remained unchanged. As can be seen from Figure 3a,c,e, the blank control group on the left does not generate silica precipitation, only the flocs formed by the aluminum hydroxide colloid. Comparing with the electrocoagulation groups on the right side, it is obvious that there are black dots in the aluminum hydroxide flocs in the electrocoagulation group, which are the silicon precipitates formed. Under the microscope, the diameter of silicon precipitation is about 15 μm at 10 min (Figure 3b), the diameter of silicon precipitation grows to about 18 μm at 20 min (Figure 3d), and the diameter of silicon precipitation grows further and becomes about 24 μm at 30 min (Figure 3f). The

above experimental results prove that silicon precipitation could be achieved through electrocoagulation, and the longer the deposition time, the better the precipitation effect.

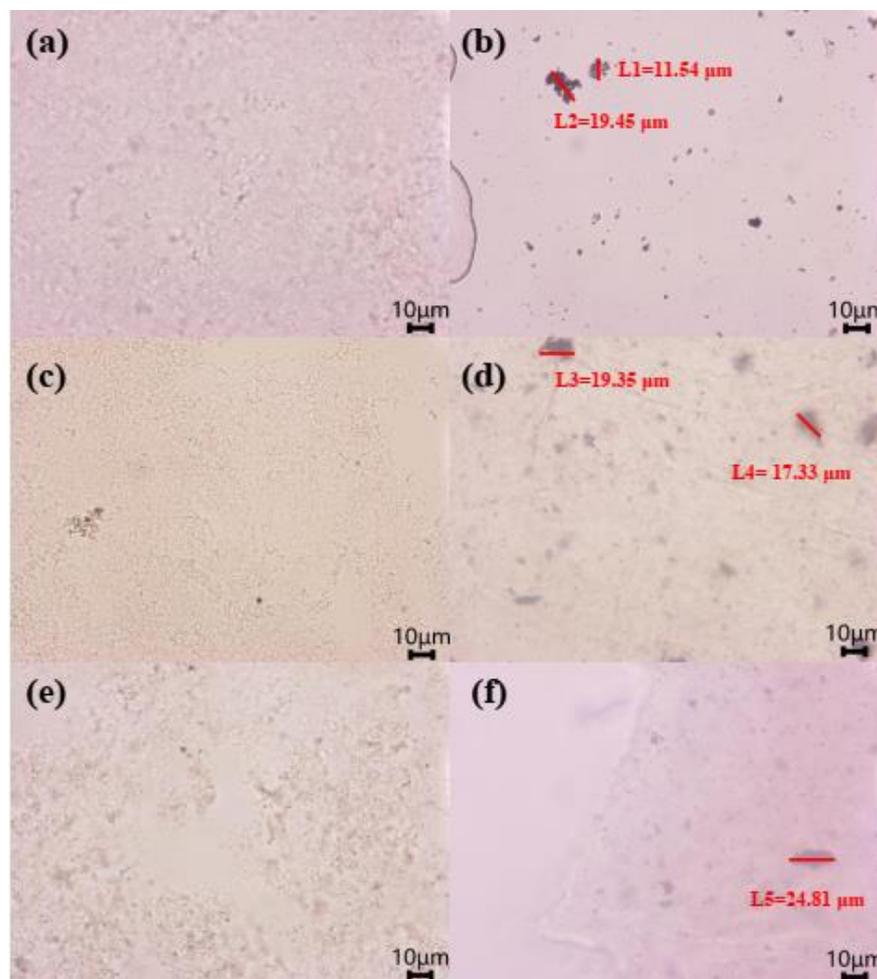


Figure 3. Silica scale particle size after 20 min (b), 30 min (d) and 40 min (f) electrocoagulation and corresponding blank control group (a,c,e).

3.4. Experiment on Wastewater from the Hongshan Oilfield

In practical application, for the Hongshan Oilfield wastewater with $\text{pH} = 8.08$, it requires a large amount of acidic solution to adjust the pH to around 6.0, which would greatly increase the cost of silica removal by electrocoagulation. Therefore, it is of vital necessity to investigate the silica removal effect of the optimal solution of electrocoagulation without adjusting the pH . Under the conditions of $\text{pH} = 8.0$, reaction time = 20 min, current density = 27.2 mA/cm^2 and wastewater temperature = $35 \text{ }^\circ\text{C}$, the silicon content decreased from 76.22 mg/L to 10.75 mg/L , with a silicon removal rate of 85.90% and the anode mass loss was 0.0209 g, which was comparable to other relevant researches [7,33]. Obviously, the silicon removal effect was better than that of the optimal scheme of orthogonal experiments. However, it is known that the silicon removal effect of electrocoagulation decreases if the solution is alkaline during the electrocoagulation process according to relevant researches. We thought the enhanced silicon removal effect might be owing to the fact that other ions in the wastewater exhibit a significant contribution to the silica removal effect under alkaline conditions despite the decreased silica removal effect of electrocoagulation.

The following experiments were conducted to determine whether the increased silicon removal effect at $\text{pH} = 8$ was related to calcium and magnesium ions or not. Three sets of experiments were designed based on the presence of 453.04 mg/L calcium ions and

167.00 mg/L magnesium ions in the wastewater from the Hongshan Oilfield, and the experiments were implemented on simulated wastewater with no addition of magnesium ions, no addition of calcium ions, and no addition of calcium or magnesium ions while other conditions for silica removal by electrocoagulation remain unchanged. As shown in Figure 4, the silicon removal rates of calcium or magnesium or neither are less than that of normal conditions, which proves that calcium and magnesium ions do contribute to the final silica removal effect at pH = 8. In addition, the promoting effect of calcium ions was more efficient than that of magnesium ions. In detail, calcium ions could reduce the post-experimental silicon content by about 9 mg/L compared with that without addition while magnesium ions could only reduce it by about 4 mg/L. After literatures review [8,34,35], calcium ions exist in the form of calcium hydroxide and calcium carbonate under alkaline conditions, which could flocculate the silicon in the wastewater together. Meanwhile, magnesium ion exists as magnesium hydroxide under alkaline condition, which has poor flocculation ability; this is the reason why promoting the silicon removal effect of magnesium ions was less efficient than that of calcium ions.

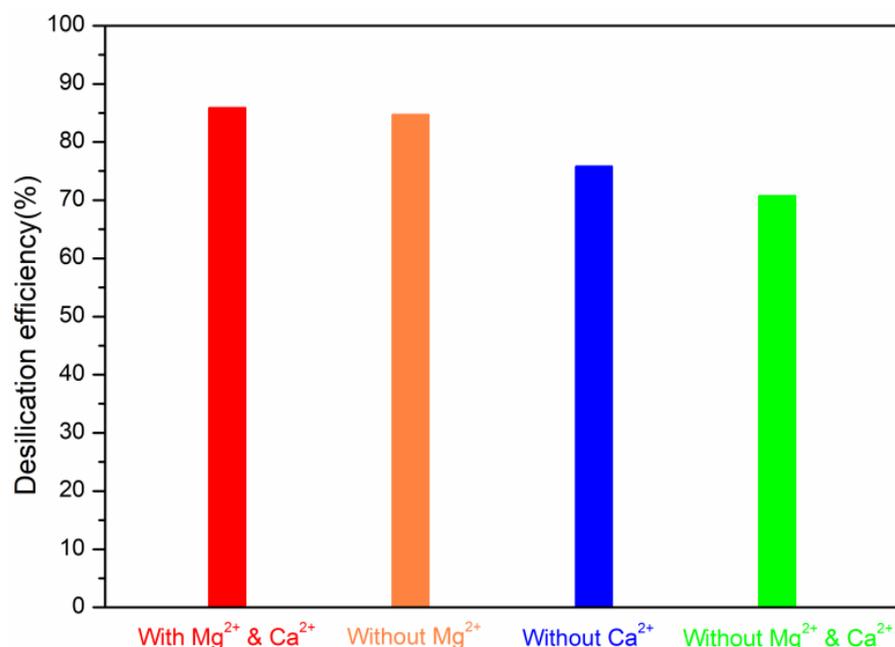


Figure 4. The impact of Ca²⁺ and Mg²⁺ on silicon removal rate.

3.5. Mechanism Investigation

In order to clearly explain the mechanism of silicon removal by electrocoagulation, the relevant mechanism is shown in the Figure 5. In this experiment, polished aluminum sheet was applied as the anode while the platinum sheet served as the cathode. As for the sacrificial anode, the aluminum sheet lost electrons to form Al³⁺, which then combine with hydroxide ions produced at the cathode to form aluminum hydroxide. Thus, the Al(OH)₃ could coagulate with silicon and colloids, as well as other impurities. In addition, calcium ions and magnesium ions in sewage, as well as calcium ions introduced from high calcium acetate containing wastewater, could also form flocculants of Ca(OH)₂, CaCO₃ and Mg(OH)₂ in the reaction tank and carry out chemical flocculation treatment for other impurities such as silicon in sewage [19–21]. Therefore, the flocculants formed by the above two flocculation treatments would eventually be transformed into precipitates, and the silicon in sewage can be completely removed by subsequent filtration and other treatments.

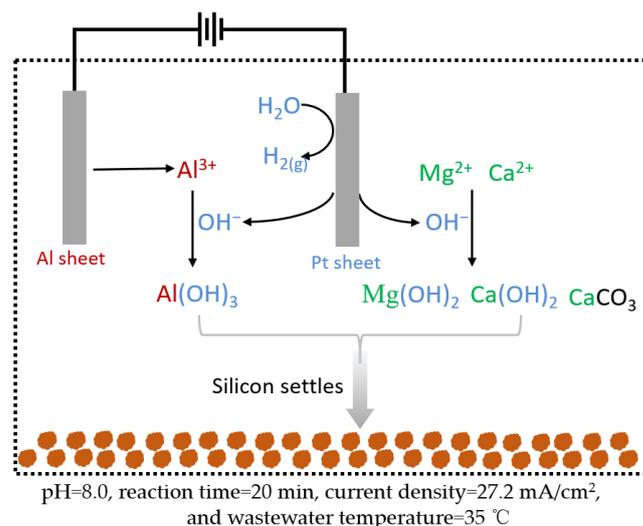


Figure 5. Silicon removal mechanism of electrocoagulation.

4. Conclusions

In conclusion, the innovation of this paper lies in the systematic experimental methods to obtain the best experimental scheme for the treatment of high-silicon oilfield wastewater by electrocoagulation, analyze the mechanism of silicon removal and then apply it to the practical wastewater after optimization the scheme. First, the optimal silicon removal scheme towards simulated wastewater was selected as pH = 6, reaction time 20 min, current density 27 mA/cm² and wastewater temperature 35 °C through the single factor and orthogonal experiments. Secondly, the silicon content of Hongshan Oilfield wastewater would decrease from 76.22 mg/L to 10.75 mg/L under the conditions of pH = 8.0, reaction time 20 min, current density 27.2 mA/cm² and wastewater temperature 35 °C, with a silicon removal rate of 85.90% and 0.0209 g mass loss of the pole plate. Last but not the least, the experimental results prove that calcium and magnesium ions do contribute to the final silica removal effect at pH = 8. We hope this study would be useful for the industrial application of electrocoagulation to remove silicon from oilfield wastewater.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/w15010206/s1>, Table S1. Specific data of orthogonal experiment; Table S2. Silicon removal rate analysis after electrocoagulation; Table S3. Silicon content data analysis after electrocoagulation; Table S4. Anode mass loss analysis after electrocoagulation; Table S5. Verified experiment for theoretical optimal scheme.

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