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A Case Study for Removing Highly Concentrated Chlorides from Industrial Wastewater

Elham Abdel Kader, Guzine El diwani, R. El-Araby* and Salwa I. Hawash

Chemical Engineering and Pilot Plant Department, National Research Centre, Cairo, Egypt El-Bohouth Street, Dokki; P.O. Box 12622 Phone: +201111227080, Fax: +20-2-3370931

Abstract

Removal of chloride ions is necessary to improve water quality before being reused for irrigation of non-edible trees. The objective of this study is to remove chloride ions from acidic industrial wastewater using ultra high lime aluminate (UHLA) process. Treated wastewater was generated from National Egyptian Renewable Chinese Laboratory for solar energy plate's manufacture in Sohag, Egypt. A set of experiments have been conducted to predict the optimum operating conditions for optimization the chlorides removal. In experiments, alum (aluminum sulfate) and/or CaO (lime) was added to adjust wastewater pH. The reagent dosages, lime and alum doses, reaction times, pH levels, and starting chloride concentrations in the removal of chloride ions have been examined. According to experimental findings, complex precipitation of calcium aluminate chloride hydroxide $Ca_4\ Al_2Cl_{12}\ (OH)_{12}$ was responsible for controlling the removal of chloride ions, and highest removal has been achieved at doses of lime to alum that ends to an acceptable limit. In this investigation, about 90 % was the highest removal of chloride ions occurred at a dosage of 2.9 molar lime/alum at a high pH (10.5). Thus, it was found that chloride elimination is influenced by both the alkaline pH and the content of aluminum.

Key words: Industrial Wastewater, alum, calcium chloroaluminate, chlorides removal.

1-introduction

Chloride ions can be found in natural water sources such as rivers, lakes, and oceans, as well as in industrial wastewater generated by various industries including chemical, pulp and paper, and petroleum **(R. Kumar et al., 2019)**. Chloride removal is an important process in wastewater treatment and water desalination because high concentrations of chloride ions in drinking water have health risks, including increased blood pressure and cardiovascular disease likewise excess of chlorine in drinking water alters its flavor and produces tri halo-methane, in the presence of organic matter which is carcinogenic **(Hsu et al. 2001)**. whereas high concentrations of chloride ions in wastewater can lead to the salinization of soil and water, which can have a negative impact on plant growth, aquatic life, and wildlife as well as can yield corrosion of pipes and other infrastructure in wastewater treatment plants, which can lead to leaks and breakdowns in the system (**Scott Kiser et al. 2018).** Additionally, effluent with a high chlorine level can damage buildings **(M. J. et al. 2018)**. A treatment technique is required to lower the amount of chlorides and to reduce the total dissolved solids in water **(G., Kim et al., 2021).**

The World Health Organization's guidelines for residual chlorine levels set a maximum of 0.03-0.4 mg/L **(WHO: Geneva, Switzerland. Drinking-Water-Quality 2021)**.
The solutions to the aforementioned problems include reverse osmosis, ion exchange filtration, and distillation. The choice of chloride removal method depends on several factors, including the specific characteristics of the wastewater, the required water quality, and the economic constraints of the treatment process **(K. M. et al., 2017).** The addition of magnesium ions to the water at a dose of less than 20% of the initial chloride's concentration by weight at pH 10 may be successful (**Xia et al. 2011)**. One of the most

*Corresponding author e-mail: rehamelaraby@hotmail.com.; (R. El Araby).
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straightforward and affordable techniques for removing chlorides from wastewater is de-chlorination by precipitation. In high salt wastewater, the role of cations in cuprous chloride precipitate removal was investigated **(Giyeon Yun 2001).** It was discovered that some cations, such as ferric and magnesium, oxidize sulphite, has negative effects on the removal of chloride ions. However, ferrous and manganese have little to no impact on the removal of chlorine. While hexavalent chromium increases the rate at which chlorides are removed by turning chloride ion into chlorine gas **(Wang, Y. et al., 2019; Liu, W., 2019).** Ion exchange is a potential process for purifying reclaimed water of chlorides. The velocity of the oxygen gas, temperature, and reaction time, among other processing variables, were investigated.

The use of calcium aluminates-layered double hydroxides (CaAl-LDHs) in chemisorption is a promising method for removing chlorides from water. CaAl-LDHs are a type of layered double hydroxide that has been shown to have a high adsorption capacity for chloride ions.

In chemisorption, the chloride ions are adsorbed onto the surface of the CaAl-LDHs through a chemical reaction, rather than a physical one. The CaAl-LDHs have a positively charged surface that attracts the negatively charged chloride ions, and the chloride ions are then held onto the surface by chemical bonds **(S. Chakraborty et al., 2018).**

While using the ultra-high lime with aluminum procedure was examined for chloride removal from landfill leachate, conventional methods for doing so are not economically feasible **(Kim G. 2021).** Precipitation as calcium chloroaluminate using lime and aluminum is one method to reduce chlorides in wastewater **(Kim, G. et al., 2021).** Due to an increase in the ratio of chloride to hydroxide ions in the solution, which favored the precipitation of calcium chloroaluminate solid over calcium hydroxyl-aluminate solids, temperature, pH, and chloride concentration are among the most crucial factors that affect the removal of chlorides from wastewater. It was discovered that while end chloride concentrations significantly rose with rising water temperature greater than 40°C, chlorides removal efficiency increased with increasing initial chlorides concentration at the same pH (**Abdel-Wahab et al., 2006).**

In recent years, the ultra-high lime with aluminum procedure has emerged as a promising alternative method for removing chlorides from wastewater, particularly at high concentrations. This method involves adding a high dose of lime and aluminum to the wastewater, which results in the formation of aluminum hydroxide flocs that can be easily removed from the water along with the chloride ions. This process can be effective even at high chloride concentrations and can result in a high degree of chloride removal **(Zhang, L. et al.,2021)**. While using the ultra-high lime with aluminum procedure was examined for chloride removal from landfill leachate, conventional methods for doing so are not economically feasible **(Gao, K., et al., 2021)**. The ultra-high lime with aluminum process is attractive economically because of the less expensive chemicals in addition to the easy operation **(Chen, G. et al., 2017).** Temperature, pH, and chloride concentration are among the most crucial factors that affect the removal of chlorides from wastewater. It was discovered that while end chloride concentrations significantly rose with rising water temperature greater than 40°C, chlorides removal efficiency increased with increasing initial chlorides concentration at the same pH **(Abdel-Wahab et al., 2006) .**

When chlorides are precipitated as calcium chloroaluminate in the presence of calcium and aluminum at a high pH, the removal of chlorides is increased by adding more aluminum up until 20 mg/L, beyond which the removal of chlorides becomes modest. With the increase of pH, obviously more chloride was removed. At pH of 10, the removal reached 90% **(Chen, G. et al., 2017; Goulart, L. et al., 2021)**.

Precipitation as calcium chloroaluminate using lime and aluminum is one method to reduce chlorides in wastewater **(Xuewen Wang**, **et al., 2021).**

Ultra-high-lime with aluminum was used to explore the impact of pH on the removal of chlorides, and the results showed that the best pH for the most effective removal of chlorides was discovered to be 12 ± 0.2 **(Weizao et al., 2019).**

The feasibility of precipitating calcium hydroxide and sodium aluminates to remove chlorides from solutions with high chloride concentrations was examined**.**

Highest percent of chlorides removal (84%) was reached at molar ratio of Ca:Al:Cl 10:4:1. Additionally raising the reaction temperature, starting pH, and starting chlorine concentration also increases the effectiveness of removing chloride **(Ye et al. 2021)** .

When it comes to the efficiency of chloride removal methods in case of very high concentrations of chloride ions in wastewater, the ultra-high lime with aluminum procedure may be more effective than other methods such as reverse osmosis and ion exchange. Very high concentrations of chloride ions in wastewater can be challenging to treat, as they can cause scaling and fouling of treatment equipment and membranes. Reverse osmosis and ion exchange may struggle to effectively remove chloride ions at very high concentrations, and may require more frequent membrane cleaning or resin regeneration, which can add to operating costs and reduce efficiency **(S. Chakraborty et al., 2018).** The economic appeal of the ultra-high lime with the aluminum process lies in its use of cost-effective chemicals and straightforward operation **(Chen et al. 2017).**

The major goal of this case study is to identify the crucial variables and ideal situations that enhance the capacity to remove large concentrations of chlorides from industrial wastewater by using ultra-high lime with the aluminum procedure.

2. Material and Methods

2.1. Materials

Industrial raw waste water is discharged from the different operation units located in the National Egyptian Renewable Chinese Laboratory then collected in a large container before treated. Aluminum sulfate (alum) 98% from Fluka AG, CH company, calcium oxide (lime) from research Laboratories PVT. LTD.

2.2. Methods

The pH of a specific volume of industrial wastewater was tested, and then the wastewater was mixed thoroughly with a specific amount of [Al_2 (SO_4).16H_2O]. After a designated period of stirring, (CaO) was gradually added to regulate the pH level, and stirring continued to facilitate complex formation. The addition of alum and lime was carried out under flash-mixing conditions. The solid and liquid components were separated by means of filtration, and the chloride content of the filtrate was analyzed and compared to the Egyptian Code No. (501/2015) **(ECP 501 / 2015).** Chloride, sulfate, carbonate, and arsenic ions were measured in wastewater using ion chromatography (IC) following the protocol described by **(Li, Y. et al. 2018)** with modifications. Wastewater samples were collected from the influent and effluent of a wastewater treatment plant and filtered through a 0.45 µm membrane filter. IC analysis was performed using a Dionex ICS-2100 ion chromatography system equipped with a conductivity detector. XRF technique was used to detect pollutants removal after the filtration step. A schematic of the experimental procedure is presented in Figure (1).

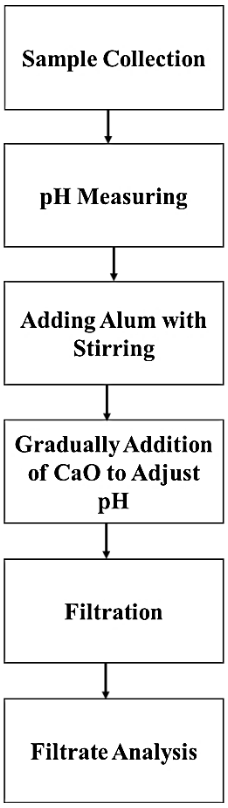

Fig. (1): Experimental Flowchart

2.2.1 Chloride Removal Studies

Experiments were conducted using batch- mode technique with pH variation (7-12) **(Dikdima, J. et al, 2022)** as measured by a pH meter (HANNA apparatus model-211). The initial chlorides concentration was 3015 ppm. Different alum doses were applied to the same sample for different reaction time ranged from 0.25 hour to 4.5 hours.

2.2.2 Overall Reaction Rate of Chloride Precipitation:

The chloride removal by UHLA process, dry lime ($Ca(OH)_2$) and dry sodium aluminate ($NaAlO_2$) were used as chemical reagents to precipitate chloride as calcium chloroaluminate solid ($Ca_4Al_2Cl_2(OH)_{12}$) according to the following reaction

$$4Ca^{2+} + 2Al^{3+} + 2Cl^{-} + 12OH^{-} \longrightarrow Ca_4 Al_2 Cl_2 (OH)_{12} \downarrow \qquad \textbf{(1)}$$

According to the **reaction (1) (El Diwani, G. et al., 2022),** the depletion of Cl^- ions in the solution has been followed.

$Cl^- \rightarrow$ product

$$r_{Cl^-} = -d\,C_{Cl^-}/dt = kt \qquad \textbf{Eq. (1)}$$

According to Reaction (1), the depletion of Cl^- ions in the solution has been studied.
It can be concluded that the reaction is a unimolecular type, first- order reaction;

by separating and integrating the above equation, we obtained

$$-\ln C_{Cl^-}/C_{Clo} = kt \qquad \textbf{Eq. (2)}$$

The reaction kinetics and rate constant for chloride removal have been calculated under the assumption that the reaction is homogenous, irreversible, and precipitates all produced solids **(Hu, X. et al., 2021).**

The formula for calculating the chloride ion concentration in a water sample using silver nitrate titration is in Equation (3):

$$Cl^- \text{ concentration (mg/L)} = (V_2 - V_1) \times M \times (35.45 / V_{sample}) \qquad \textbf{Eq. (3)}$$

where:

V_1 = volume (mL) of silver nitrate solution required for the blank titration
V_2 = volume (mL) of silver nitrate solution required for the sample titration
M = molarity of the silver nitrate solution (mol/L)
35.45 = molar mass of chloride ion (g/mol)
V_{sample} = volume (L) of the water sample used in the titration

This formula calculates the chloride ion concentration in milligrams per liter (mg/L) or parts per million (ppm) in the water sample **(S. R. Khan, 2016).**

2.3. Analysis and Characterization

A comprehensive chemical analysis was done for the wastewater After collecting it in a large container in preparation for its treatment and before mixing it with municipal wastewater. The chloride ion concentration in the wastewater was determined using Mohr's precipitation titration, the remaining chlorides in the liquid fraction were identified **(APHA 2005),** where silver nitrate was used to titrate the chloride ions in aqueous solution **(S. R. Khan, 2016)**. The difference between the initial and residual chlorides served as the basis for calculating the chlorides removal efficiency.

The Analysis and Consultation Unit - Domain of Evaluation and Remediation of Hazards Wastes at the National Research Centre in Egypt carried out a comprehensive chemical analysis of both raw and treated industrial wastewaters.
The solid fraction precipitate was dried and analyzed by X-ray fluorescence (XRF) using Oxford X-met 7500 mining analyzer, which was conducted at the Analysis and Consultation Unit - Preparation and Chemical Analysis by XRF Lab at the National Research Centre in Egypt.

3- Results and Discussion

3.1. Raw Wastewater Specifications

From the complete chemical analysis of raw wastewater shown in **Table (1)** it is clear that, the pH, chlorides, bicarbonates, and dissolved solids were the principal contaminants. The table showed that the concentration of chloride ions in the wastewater was 3015 mg / L which was considered highly saline.

Table (1): Complete Chemical Analysis of The Industrial Wastewater Considered.

ELEMENT TO BE ANALYZED	RESULTS	UNITS	ACCEPTED LIMITS According to Egyptian Code No. (501 / 2015) Maximum Limit *
1] PHYSICAL PARAMETERS:			
pH	1	------	8.5
Color	Colorless	mg/l Pt Co	Colorless
2] PHYSICOCHEMICAL PARAMETERS:			
Chloride [Cl^-]	3015	mg/l	400
Sulphate [SO_4^{--}]	74.47	mg/l	500
Bicarbonate HCO_3^-]	1125	mg/l	400
Sodium Absorption Rate[SAR]	4.3	mg/l	9
Sodium [Na^+]	170.85	mg/l	230
Potassium [K^+]	220	mg/l	-------
Magnesium [Mg^{++}]	30.629	mg/l	100
Calcium [Ca^{++}]	69.89	mg/l	230
Fluoride [F^-]	528.17	mg/l	15
Phosphate [PO_4^{+++}]	-----	mg/l	30
Lithium [Li]	-----	mg/l	2.5
Phenol	------	mg/l	0.002
Total dissolved solids TDS]	12000	mg/l	3000
Total suspended solids [TSS]	1.2	mg/l	< 30
Biochemical Oxygen Demand [BOD]	12	mg/l	< 400

***(APHA: "Standard Methods for the Examination of Water and Wastewater."20th Edition, American Water Works Association Water Environment Federation, Washington, D.C., U.S.A (2005).**

3.2. Effect of Alum Dose

A series of experiments using different concentrations of UHLA ranged from 50 to 400 mg/L procedure have been conducted. Chlorides removal from industrial effluent which was extremely acidic increased with increasing alum dose till 350 g/L. As demonstrated in Figure (1), no more significant improvement was seen after that, hence the dose of alum 350 mg/L was advised as the optimal dose for removing chlorides in this experiment.

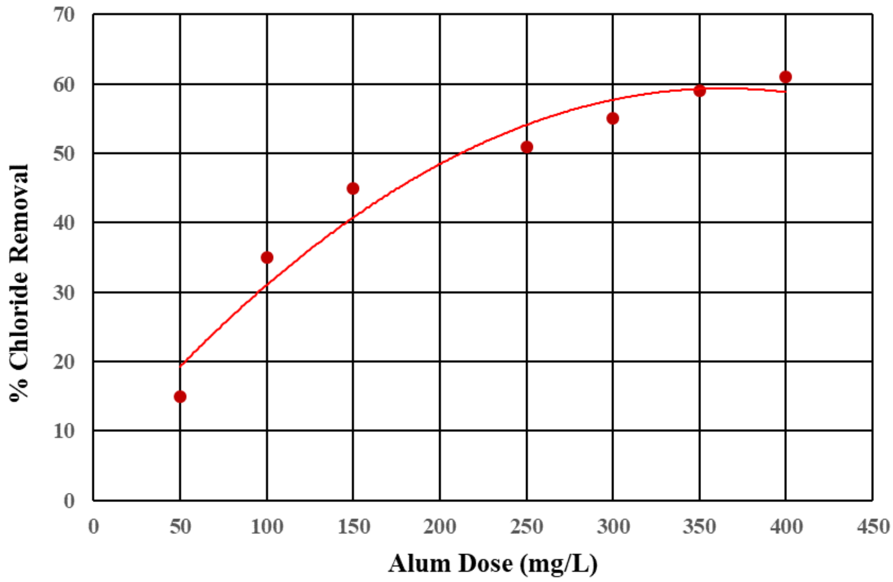

Fig. (1): Effect of Alum Dose on % Chloride Removal.
(Reaction Time = 2 h. &pH =9)

3.3. Effect of Lime / Alum dose

Good chloride removal was obtained at reasonable ranges of lime dose / alum dose. Chlorides removal was controlled by calcium chloroaluminate complex precipitation $Ca_4Cl_2Al_2(OH)_{12}$. At theoretical stoichiometry, (ratio of calcium to aluminum should be equal 2.0), this complex is the only important precipitated solid. From experimental results this ratio is typically more than theoretical stoichiometric and maximum chlorides removal was found at 2.9 as shown in Figure (2).
Deviation from stoichiometry at equilibrium could be due to the formation of another solid phase, calcium hydroxy aluminate Ca_4Al_2 $(OH)_{12}]$ in which Cl^- ion in $Ca_4Cl_2Al_2(OH)_{12}]$ was replaced by OH- ions to form Ca_4Al_2 $(OH)_{12}]$ which agrees with previous researches (**Chen et al. 2017).**

General formula of aluminate phase Ca_2Al $(OH)_6]x^-$ indicates the composition of the positively charged layer unit **(Ahmed Ibrahim et al., 2003)**. When ratio of alum to lime dose is ≥ 0.5 indicates that the data of equilibrium experiments do not fall around theoretical stoichiometry.
Results of this study revealed that maximum chlorides removal (about 84%, 82% &81%) were reached at lime to alumina molar ratios 2.9, 2.09 &1.4 as shown in Figure (2).

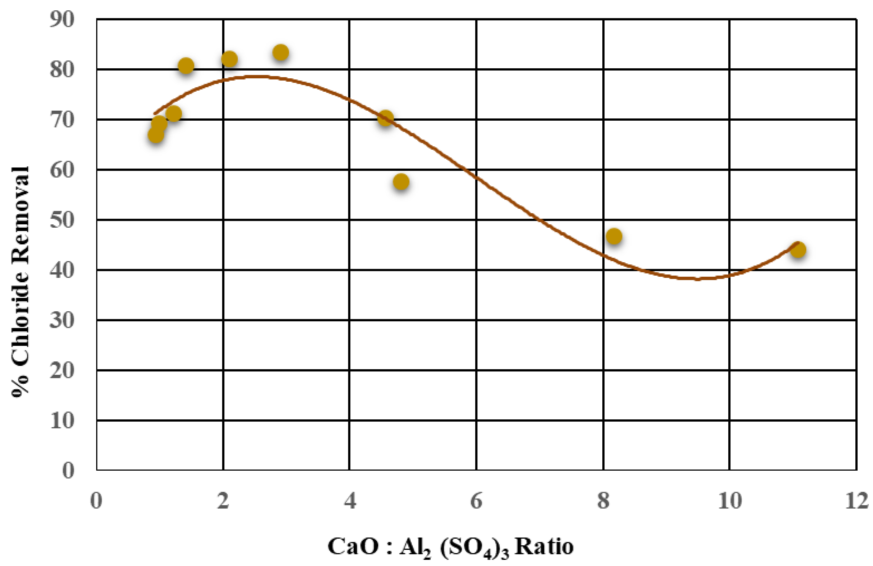

Fig. (2): Effect of Lime Dose to Alum Dose Ratio on % Chloride Removal.

3.4. Effect of pH

Proper pH was adjusted with lime (CaO) addition; both alum and lime were added under constant stirring rate (300rpm) at which precipitation of complex calcium chloro- aluminate takes place immediately at high alkaline pH.

Chlorides removal was controlled by precipitation of calcium chloro-aluminate Ca_4 Al_2 Cl_2 (OH) **(Xianjia Peng et al. 2018).** Satisfactory obviously chlorides removal results were obtained at certain alkaline pH (10.5) which was adjusted by lime (CaO) addition as it is clear in Figure (3). As shown from Figure (4), chlorides removal increased linearly with increasing pH. Also, it is clear that chlorides removal using (UHLA) process was impacted with pH value due to the solubility of calcium chloro-aluminate $Ca_4Al_2Cl_2$ $(OH)_{12}$] because this solubility is a function of pH value. Chloride, aluminum, calcium and hydroxide concentrations played the key roles in removal of chlorides as $Ca_4Al_2Cl_2(OH)_{12}$**(Ahmed Ibrahim et al., 2003)**. Therefore, based on the obtained results, a pH of 10.5 was recommended as the optimal condition for chloride removal.

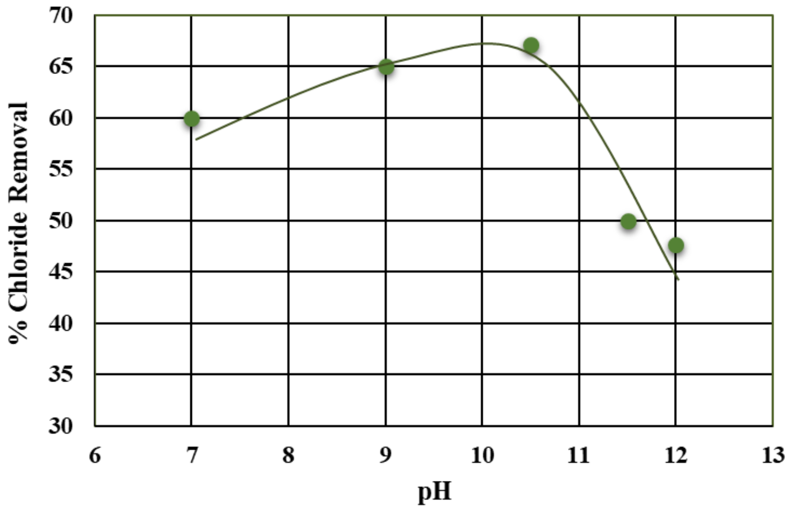

Fig. (3): Effect of pH on % Chloride Removal
(Alum Dose= 350 mg/L &Reaction Tim = 2h.)

3.5. Effect of Reaction Time

A set of experiments were done using (UHLA) process under different stirring time periods from 15 minutes to more than 4 hours. Experimental results plotted in Figure (4) revealed that, by increasing reaction time chloride percent removal increased till reaching 4 hours then no sensible advance in this removal was observed. At a reaction time of 4 hours, removal efficiency about 84% was achieved.

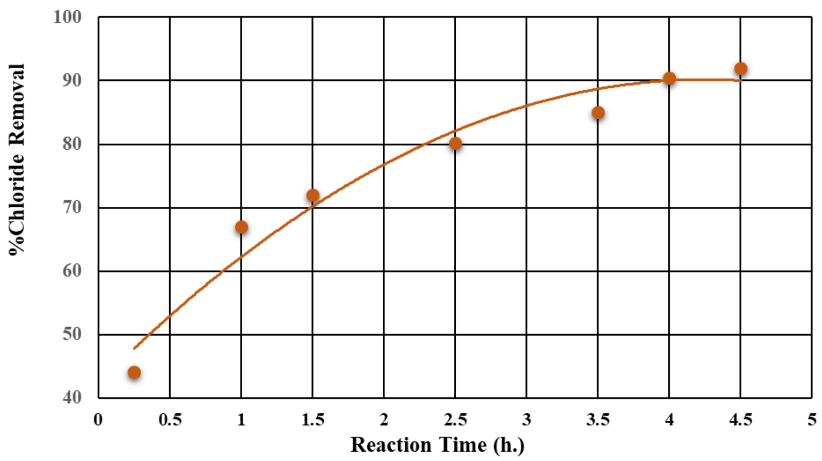

Fig. (4): Effect of Reaction Time on % Chloride Removal
(Alum Dose= 350 mg/L & pH = 10.5.)

3.6. Overall Reaction Rate of Chloride Ion Precipitation using the Ultra-High Lime with Aluminum Process (UHLA)

In this research, the ultra-high lime with Aluminum process was evaluated for chloride removal from the industrial wastewater generated from solar panels manufacture.

Experimental results showed that the UHLA process can remove chloride due to the formation of a calcium chloroaluminate solid complex phase (Friedel's salt) (**Ahmed Ibrahim et al., 2003)** which is a reasonable mechanism that is able to describe the chloride removal process and based on the assumptions that Ca_4 Al_2 Cl_2 $(OH)_{12}$ is the proper formula for calcium chloroaluminate solid precipitate as in **Reaction (1) (Suthatta Dontriros et al., 2020).**

By plotting kinetic data in **Figure (5),** a kinetic rate constant (k) has been obtained where it represented the slope of the straight line which equals 0.0065 min^{-1}.

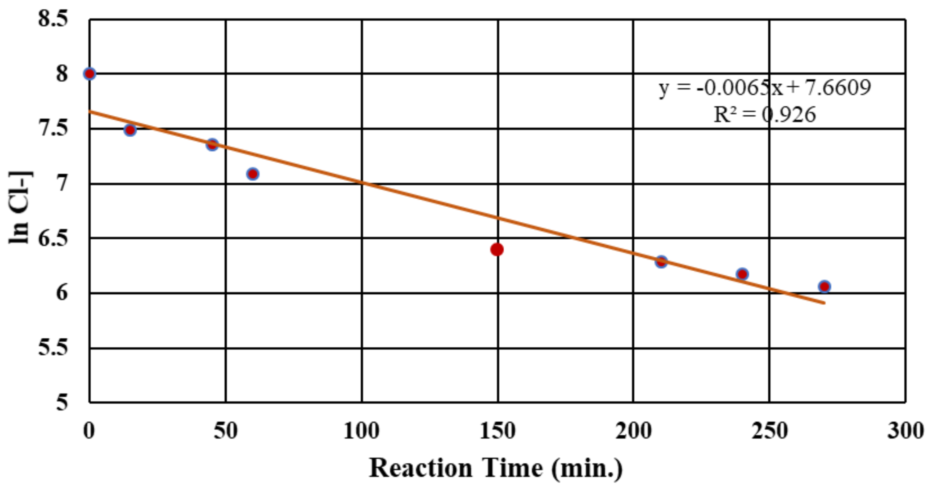

Fig. (5): Kinetic Data Plot.

According to the experimental data, Chloride is precipitated at optimum conditions of 350 mg/L Alum dose, 30 ± 5°C, alkaline pH (10.5) and 300 rpm stirring rate and 4 hours reaction time.

3.7. Post Analysis of The Treated Wastewater

The XRF technique indicated that the precipitate was mostly composed of Cl (41.18%), indicating a reduction in chloride contaminants in the treated wastewater as shown in **Table (2).**

Table (2): The Chemical Composition of The Precipitate After Treatment

Components	(wt.%)
SiO_2	2.126
Al_2O_3	18.96
Fe_2O_3	0.56
TiO_2	0.04
MgO	1.55
MnO	0.006
CaO	31.10
Na_2O	0.61
K_2O	3.09
P_2O_5	0.14
SO_3	0.08
F	0.272
Cl	**41.18**
NiO	0.004
ZnO	0.0
SrO	0.278
As_2O_3	0.003
Total	**99.999**

The results of the optimal operational conditions for wastewater treatment were applied. Table (3) shows the results of the chemical specifications of the treated water.

Table (3): The Chemical Analysis of Wastewater After Treatment

ELEMENT TO BE ANALYZED	Before	After	ACCEPTED LIMITS *
Chloride Cl^- (mg/l)	3015	250	400
Bicarbonate HCO_3 (mg/l)	1125	35	400
Potassium K^+ (mg/l)	220	26	-------
Calcium Ca^{++} (mg/l)	70	24	230
Fluoride F^- (mg/l)	528	0.2	15
Total dissolved solids TDS (mg/l)	12000	2285	3000

*According to Egyptian Code No. (501 / 2015)

3.8. Environmental Impact Evaluation

Decreasing chloride levels in wastewater from 3000 mg/L to 250 mg/L can have several positive environmental impacts, including:

1 .Reducing the salinity of receiving water bodies: High chloride concentrations in wastewater can increase the salinity of receiving water bodies, which can negatively impact aquatic plant and animal life. By reducing chloride levels, the salinity of the receiving water bodies can be maintained at a more natural level **(Ongley et al. ,2014).**

2 .Reducing the risk of corrosion: High chloride concentrations in wastewater can increase the risk of corrosion in pipes and other infrastructure, which can lead to leaks and other problems. By reducing chloride levels, the risk of corrosion can be reduced, which can extend the lifespan of the infrastructure **(Trujillo et al., 2015).**

3 .Reducing the risk of soil contamination: High chloride concentrations in wastewater can also lead to soil contamination if the wastewater is used for irrigation or other purposes. By reducing chloride levels, the risk of soil contamination can be reduced, which can improve soil quality and protect local ecosystems **(Gharabaghi et al., 2017)**.

4 .Improving water quality for downstream users: High chloride concentrations in wastewater can make the water unsuitable for certain downstream uses, such as industrial processes or irrigation. By reducing chloride levels, the water can be made suitable for these uses, which can have economic and environmental benefits **(Al-Balushi et al., 2018).**

CONCLUSIONS

From experimental results it is concluded that:

1- The results of the equilibrium and kinetics analysis showed that the UHLA method has a substantial potential benefit for removing chlorides and other contaminants from industrial wastewater that was extremely acidic.
2- It should be highlighted that calcium chloroaluminate precipitation as $[Ca4Al_2Cl_2(OH)_{12}]$ was the main factor controlling the removal of chlorides from wastewater utilizing UHLA technology in this investigation.
3- It was observed that the removal of chlorides increased with increasing alum addition up to the optimal dose of approximately 350 gm/L, after which the removal of chlorides became modest.
4- The alkalinity or pH of the treated solution is a major role in the process of removing chlorides, and in our situation, the ideal pH was around 10.5.
5- Regarding reaction or stirring time, experimental findings showed that four hours was the right amount of time that produced positive outcomes.
6- It is concluded that the rate of chloride depletion in the solution showed that the reaction is a unimolecular type, first-order reaction.

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Availability of data and materials

Not applicable

Author contribution

Conceptualization & methodology, Elham Abdel Kader & S.I. Hawash; **investigation,** Guzine El diwani &R. El-Araby; **supervision,** Guzine El diwani; **validation,** Guzine El diwani, Elham Abdel Kader, S.I. Hawash & R. El-Araby; **interpreted the data and contribute to writing the paper (original draft),** Elham Abdel Kader, S.I. Hawash & R. El-Araby; **writing (review and editing),** Guzine El diwani, Elham Abdel Kader, S.I. Hawash & R. El-Araby.

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Grid Connected PV system".

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Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Competing interests

The authors declare no competing interests.

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