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Experimental Process of Coagulation-Floculation by Aluminum Sulfate of Surface Waters of the Djiri River (Brazzaville, Congo)

Bernard Ndoki[1*], Michel Elenga[1], Arnaud Tamba Sompila[2], Dominique Nkounkou[3], Maturin Nzikou[4]

[1]Laboratoire de Génie Des Procédés Industriels, INRSIIT, Brazzaville, Congo
[2]EPRAN-Congo, Pôle D'Excellence en Alimentation et Nutrition, Faculté des Sciences et Techniques, Université Marien Ngouabi, Brazzaville, Congo
[3]Institut de Recherche en Sciences de L'ingénieur, Innovation et Technologie, INRSIIT, Brazzaville, Congo
[4]Laboratoire de Génie des Procédés Industriels, Ecole Nationale Supérieure Polytechnique (ENSP), Université Marien Ngouabi, Brazzaville, Congo
Email: *barnerdoki@gmail.com

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Abstract

This article investigates the coagulation-flocculation of surface water from the Djiri River using aluminum sulfate via jar tests, considering pH and coagulant dosage as the primary operating variables. The study reports improved TOC removal rates of 67% and 92%, respectively, at a moderately acid pH (5.5 to 6.5) and with increasing coagulant dosages (1 to 4 mL), for initial TOC concentrations in the 6 to 21 mg/L range. The manuscript also interprets the removal mechanisms using correlation analysis and a Freundlich adsorption-type approach. Analysis of the Freundlich isotherm reveals very good R^2 correlation coefficients, ranging from 0.88 to 0.99, for various TOC concentrations and varying coagulant dosages. The experimental TOC removal process could be the result of a phenomenon favoring adsorption onto aluminum hydroxide flocs.

Keywords

Water Organique Mater, Coagulation-Floculation, Aluminum Sulfate, Mechanisms, Adsorption

1. Introduction

Surface waters are often subjected to urban, agricultural, and industrial pressures, which represent a major problem of exposure from a large number of potential sources of contamination [1]. They then become increasingly polluted, thus reducing their potability [2].

Indeed, water quality is altered by an overload of organic matter, by toxic substances from agricultural activities, and by industrial and domestic effluents discharged into the receiving environment without treatment [3] [4].

The Djiri River, located north of Brazzaville, is a vital resource. It supplies approximately 70% of the city's drinking water via the Congolese water plant.

Its catchment area is subject to severe erosion, due to the sandy nature of the soils and the urbanization of the area, particularly through the construction of roads along the slopes [5].

Since the soil is the first interface between water and minerals, during the journey of water towards the outlet of watersheds, various interactions occur between the water and the solid elements of the soil.

The result of this interaction is the production in solution of chemical elements resulting, on the one hand, from the alteration of rocks, and, on the other hand, from the decomposition of organic matter [6].

Therefore, surface waters must be treated before being consumed, as they pose a serious threat to human health.

Treatment methods such as microfiltration, oxidation, coagulation/flocculation, membrane separation, or combinations of these methods are used to meet the water quality standards required for consumption [7].

Coagulation/flocculation is one of the most widely used and effective physicochemical methods for surface water treatment.

Colloidal particles, soluble compounds, and very fine solid suspensions present in waters are removed by coagulation/flocculation through destabilization and the formation of flakes.

Optimization studies were conducted, making it possible to determine the multiple interactions between the variables. This approach was used to create the optimal combination of variables [7]-[9].

In this study, the treatment of surface water from the Djiri River using the coagulation/flocculation method was investigated.

The objective of this study is to determine the optimal treatment conditions that analyze the interactions of the variables selected for water treatment.

In this study, a jar test experimental design was applied to optimize the selected parameters.

By selecting pH and coagulant dose as independent variables, the effects on TOC removal, TSS, and turbidity were evaluated as response variables.

2. Materials and Methods

2.1. Materials

The physicochemical characterization of the surface waters of the Djiri River was carried out over a period of 10 days using ten (10) raw water samples collected directly from the river. The 10 samples were collected on consecutive days at the same location, time, and depth. The collected water samples were placed in 1.5 L polyethylene bottles that had been washed beforehand with 1 M nitric acid and then with distilled water. In the field, prior to filling, the bottles were rinsed three times with the water to be sampled. The water samples were then transported to the laboratory in a cooler at 4˚C, in accordance with the AFNOR NF EN 25667 (ISO 5667) standard [10]. Physicochemical analyses were performed in accordance with the same AFNOR standard and focused primarily on determining pH, suspended solids (SS), total organic carbon (TOC), turbidity (Turb.), and conductivity (EC). The following parameters were measured on-site: pH and electrical conductivity (EC) were measured using a Consort C933 multiparameter; sample turbidity and suspended solids (SS) were measured using a turbidimeter (HACH/2100Q); and total organic carbon (TOC) was measured using a DW-DI 1500 TOC analyzer [11].

2.2. Methods

- **Coagulant Solutions**

During our tests, we used powdered aluminum sulfate [$Al_2(SO_4)_3 \cdot 18H_2O$], prepared by dissolving it in distilled water, as the coagulating reagent. A 10 g/L stock solution was prepared periodically.

The pH of the raw water was adjusted using a limewater solution.

- **Coagulation-Flocculation**

The coagulation-flocculation method has a high capacity for removing TOC, turbidity, and TSS from raw water.

The coagulant [$Al_2(SO_4)_3 \cdot 18H_2O$] was used in this study. The coagulation-flocculation experiments were performed using a four-paddle-shaker jar-test device (Velp scientifica).

Jar test trials were implemented for the optimization of coagulant concentration, as well as the coagulation-flocculation pH. Before each series of tests, a solution of alumina sulfate [$Al_2(SO_4)_3 \cdot 18H_2O$] at 10 g/L was prepared.

Beakers of equal volume (1 L) were used to examine the different coagulant dosages in each trial. Each beaker contained 0.5 L of raw water.

TOC, turbidity, and TSS were selected as response variables, and the effectiveness of removing these parameters was evaluated as a function of parameters such as pH and coagulant dose [11].

Initially, at a temperature of 20˚C, coagulation was carried out under rapid agitation at 250 revolutions per minute for 3 minutes.

The coagulant is introduced during this step by injection with a pipette, and the pH is adjusted immediately.

Next, the slow agitation (flocculation) step is carried out at 30 revolutions per minute for 20 minutes.

After settling for 20 minutes, the supernatant was collected and then analyzed. The percentage of organic compound removal was evaluated by the yield, which is expressed as:

$$R\% = [(Co - Cf)/Co] \cdot 100 \quad (1)$$

Co and Cf represent the initial and final concentrations of the organic compound, respectively, expressed in mg/L.

Statistical analysis of the obtained data was performed using STATISTICA 7.1 software (Stat Soft, France, 2006) to determine basic statistics, calculate the correlation matrix, etc.

As we were interested in the general tendency of organic compound removal (R%), the Freundlich isotherm expressing the amount of compound adsorbed per unit of adsorbent (x/m) as a function of the residual concentration of the organic compound at equilibrium (Ce), was used, with the pH of the water or the coagulant as a fixed effect [12] [13].

As a reminder, Freundlich's law is expressed respectively as:

$$Qe = k \cdot Ce^{1/n} \quad (2)$$

$$\log(Qe) = 1/n \cdot \log Ce + \log k \quad (3)$$

Qe = quantity of compound adsorbed per unit of adsorbent;

Ce = organic compound in a state of equilibrium.

3. Results and Discussion

3.1. Overview

This section presents the results obtained during the treatment of surface water from the Djiri River using the coagulation-flocculation method. The values of TOC, TSS, and turbidity, which constitute pollution, were determined through the characterization of the surface water.

Optimization experiments were carried out with the coagulant $Al_2(SO_4)_3$ for pollution removal. By isolating pH and coagulant dose as independent variables, their effects on TOC, TSS, and turbidity removal were evaluated as response variables.

3.2. Evaluation of Parameters (Raw Water: RW)

3.2.1. Descriptive Statistics of Parameters (Raw Water: RW)

The results obtained from the analysis of the raw water parameters are presented in **Table 1**.

Table 1. Descriptive statistics of parameters (raw water: RW).

Parameters	T˚C	pH	Turb	TOC	TSS
1	23.7	6.9	13.1	10.57	21.97
2	24.02	6.75	7.42	5.19	12.55
3	24.6	6.7	20.09	14.06	33.44
4	23.5	6.78	15.2	10.64	25.27
5	23.8	6.67	30.2	21.09	49.89

Continued

6	25.04	6.54	7.09	4.9	11.94
7	24.35	6.65	8.71	6.09	14.73
8	23.8	6.7	16.9	11.16	28.21
9	24.4	6.61	17.03	11.92	28.23
10	24.1	6.53	15.9	10.56	26.57
Mean	24.131	6.683	15.164	10.618	25.28
Minimum	23.5	6.53	7.09	4.9	11.94
Maximum	25.04	6.9	30.2	21.09	49.89
Variance	0.2203211	0.0124456	47.606516	22.907418	128.16182
S.D	0.4693838	0.1115597	6.8997475	4.7861694	11.320858

Examination of the Table 1 shows:

- A very narrow temperature range, varying from 23.5˚C to 25.05˚C, with a mean of 24.13˚C ± 0.46˚C. These temperatures are typical of the atmospheric temperatures observed in Brazzaville.
- The pH ranges from 6.5 to 6.9, with a mean of 6.68 ± 0.11. These pH values indicate that all samples are slightly acidic. This water acidity could be attributed to the geological nature of the soil and the mineralization of soil organic matter driven by oxygen [14].
- Turbidity ranges from 7.09 to 20.09 NTU, with a mean of 15.16 NTU ± 6.89 NTU. None of the recorded values complies with WHO regulations (<5 NTU). This indicates pollution by organic or mineral matter [15].
- Total organic carbon (TOC) in the raw water ranges from 4.9 to 21.09 mg/L, with a mean of 10.61 mg/L ± 4.78 mg/L. These values exceed the WHO guideline value (<5 mg/L). TOC is a characteristic of natural organic matter.
- Suspended solids (SS) in the raw water range from 11.94 to 49.89 mg/L, with an average of 25.28 mg/L ± 11.32 mg/L. All SS values in the raw water exceed the WHO guideline value. The high SS values could be attributed to the presence of organic matter, including mineral particles [15] [16].

3.2.2. Correlation between Parameters

Calculating the correlation coefficients between the parameters also allows us to highlight the linear relationships between several variables that take place in the water under study. Table 2 and Table 3, respectively, present the correlation matrix of raw water and decanted water after the jar test.

For raw water, there is a very strong correlation between these three parameters: turbidity, TOC, and TSS ($r = 0.99$, $p < 0.05$). This observation shows strongly significant linear relationships between these three parameters. Water quality is closely linked to the presence and concentration of suspended solids (SS) it contains. A high concentration of suspended solids (debris, particles) often leads to an increase in water turbidity and TOC, because organic matter is mostly adsorbed onto these solid particles. Suspended matter reduces light penetration and can affect photosynthesis in aquatic plants, thereby disrupting ecosystems [17].

Organic matter (TOC) comes mainly from biological processes related to the activity of photosynthetic organisms, such as excretion, but also from bacterial and zooplankton excretion, viral lysis, or the degradation of phytoplankton biomass by grazing [18].

A weak association between pH and the parameters turbidity ($r = 0.01$, $p < 0.05$), TOC ($r = 0.09$, $p < 0.05$), and TSS ($r = 0.01$, $p < 0.05$) was observed, respectively.

The overall analysis of data seems to show an antagonistic relationship between pH and total organic carbon content in waters. This observation shows that organic matter in surface waters comes from only two main sources: a natural origin through the decomposition of organisms and an anthropogenic origin linked to human activities [17] [18].

A strong association was observed between turbidity and TOC ($r = 0.99$, $p < 0.05$), between turbidity and TSS ($r = 0.86$, $p < 0.05$), and between TOC and TSS ($r = 0.86$, $p < 0.05$). This shows that these three parameters are highly dependent.

Table 2. Parameter correlation matrix (raw water).

	T˚C	pH	Turb	TOC	TSS
T˚C	1				
pH	−0.65	1			
Turb	−0.33	0.01	1		
TOC	−0.35	0.09	0.99	1	
TSS	−0.33	0.01	0.99	0.99	1

Significant correlations ($p < 0.05$) are in bold.

Table 3. Parameter correlation matrix (settled water).

	pH	Turb	TOC	TSS	Doses Al^{3+}
pH	1				
Turb	−0.01	1			
TOC	−0.01	0.99	1		
TSS	0.14	0.86	0.86	1	
Doses Al^{3+}	−0.21	0.67	0.67	0.47	1

Significant correlations ($p < 0.05$) are in bold.

4. Coagulation-Flocculation with Aluminum Sulfate

Influence of pH and Coagulant Dosage on the Removal of Organic Compounds

The effects of binary interactions of independent parameters on the removal of organic matter were analyzed in **Figures 1(a)-(j)**.

4.1. Influence of pH

The study of this parameter is important because it conditions, on the one hand, the dissociation of the functional groups existing in the structure of organic compounds according to their pKa and, on the other hand, the speciation of the coagulant (soluble or insoluble, charged or neutral forms) [19] [20].

Coagulation and flocculation of raw water with aluminum sulfate were performed over a pH range of 4.5 to 6.9. **Figure 1** summarizes the results obtained and shows the evolution of the removal of organic matter (TOC) from the raw water tested as a function of the coagulant dose and pH.

We can observe that the optimum for the elimination of these compounds corresponds to acidic pH levels, between 5.5 and 6.75.

The results obtained confirm the bibliographic data concerning the elimination of organic compounds for pH levels generally between 5 and 7, depending on the nature of the coagulant [21] [22].

It is interesting to note that, depending on the pH, organic matter can be neutral or dissociated.

Since the removal efficiency of these compounds decreases for pH values above 7, it is therefore obvious that one of their forms would be much less removed by flocculation with aluminum sulfate.

Since pH also conditions the chemical forms of aluminum, it is clear that the mechanisms involved in the elimination of these compounds can be very diverse [23] [24].

The optimal pH value was determined by monitoring changes in the TOC of the treated raw water and the pH as a function of coagulant dosage (**Figure 1**) at 20˚C, using aluminum sulfate (0.5 to 5.5 mL of aluminum taken from the stock solution) across a pH range of 5.5 to 6.75.

At a moderately acidic pH (approximately 5.5 - 6.5), the continuous supply of aluminum, from the coagulant, with a soluble structure, exhibits strong complexation by organic ligands and promotes the dominance of organo-Al complexes [25] [26].

4.2. Influence of the Coagulant Dose

Several studies have shown that the removal efficiency of organic compounds by aluminum salts is improved by optimizing pH, temperature, and coagulant dose [27].

In order to evaluate the effectiveness of the coagulant in the treatment of raw water taken from the Djiri River, for the different TOC values and according to the raw water sample of 4.9 - 21.09 mg/L, respectively, we monitored the evolution of TOC as a function of the different doses added and at 20˚C.

Figures 1(a)-(j) show that the removal efficiencies of organic compounds vary depending on the raw water sample taken. Furthermore, the effect of the coagulant dose introduced also varies from one raw water sample to another.

These figures show that the TOC gradually decreases depending on the raw water sample from 4.9 - 21.09 mg/L to the residual value of 0.68 and 2.46 mg/L, respectively, using aluminum sulfate for doses between (0.5 - 5.5 mL) of elemental aluminum, then increases again.

This reduction to a minimum value through the use of aluminum corresponds to an optimal dose of TOC.

Beyond this value, TOC increases again when the pH of the solution becomes acidic, due to the high solubility of coagulation metals in acidic media [28].

The removal of organic matter by the coagulant could be explained by a complexation or ligand exchange between monomers, dimers, or cationic metal polymers and organic matter [29].

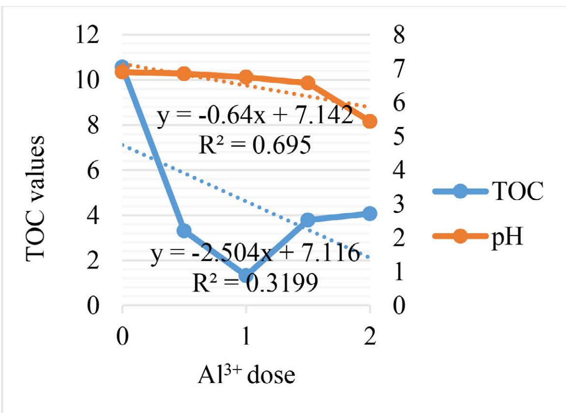
Since residual TOC is essentially in soluble form, it is difficult to eliminate by coagulation-flocculation; it persists even with increasing coagulant doses.

To determine the optimal amount of coagulant, we determined the efficiency of the change in TOC of the treated water on the TOC of the initial raw water (ΔTOC/TOCi).

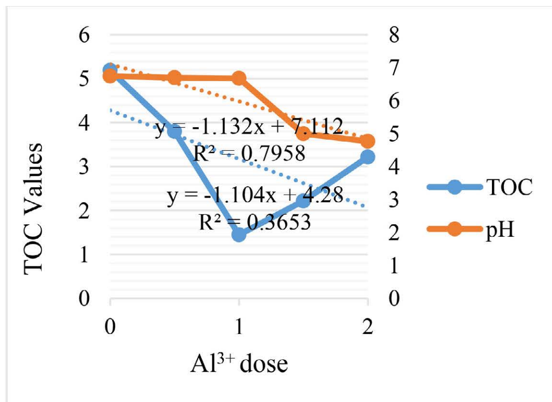
The **Figures 1(a)-(j)** summarize the results of these tests.

The optimal dose of coagulant is chosen according to the quality of the raw water being treated.

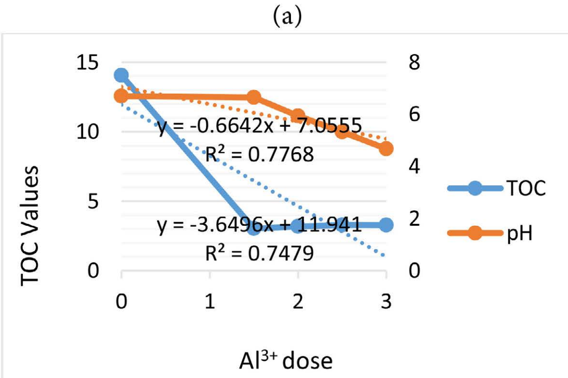
It is generally obtained when the ratio (ΔTOC/TOCi) becomes more important, and therefore, a high yield.



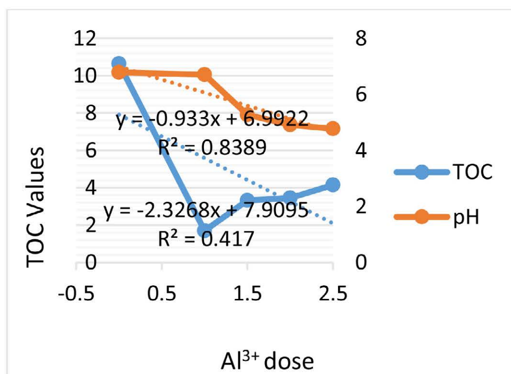
(a)



(b)



(c)



(d)

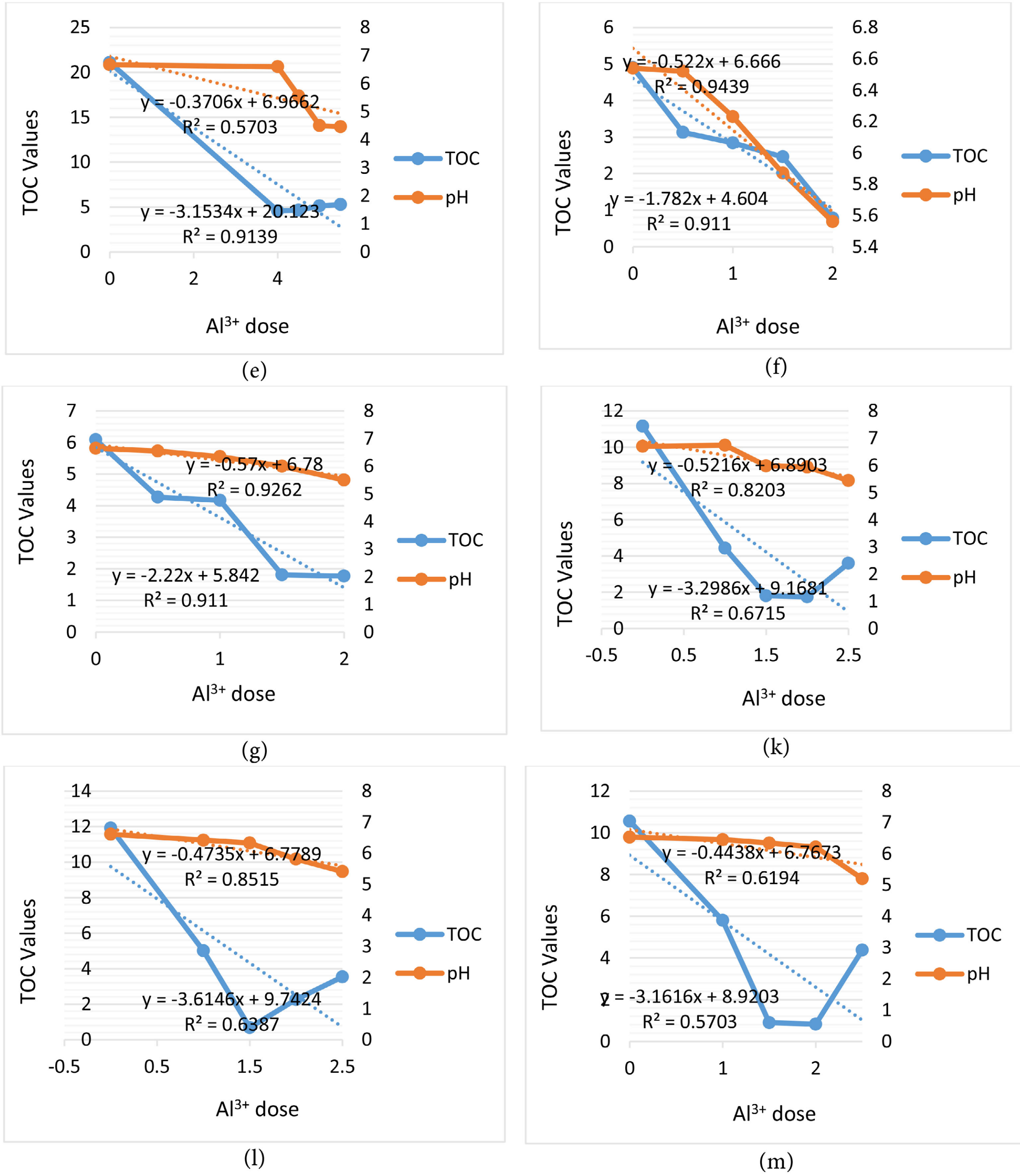


Figure 1. Influence of pH and coagulant dose on the elimination of organic compounds.

5. Influence of the Initial Concentration of Organic Matter (TOC)

To assess the effect of variation in the initial concentration of organic compounds (TOC), we treated the raw water with increasing doses of aluminum sulfate coagulant, ranging from 0.5 to 5.5 mL of elemental aluminum.

Different elimination thresholds were observed, which could suggest the possible involvement of an adsorption process [30] [31].

To clarify the role of adsorption reactions in the mechanisms of elimination of organic compounds, we used our experimental results according to Freundlich's adsorption laws.

Figures 2(a)-(j) present the Freundlich isotherms for each raw water sample taken. They show that these isotherms are linear.

Therefore, for organic compounds in raw water, we can consider adsorption onto aluminum hydroxide flocs involving Van Der Waals forces or hydrogen bonds.

This could be justified by the plot of the Freundlich isotherms expressing the quantity of compound adsorbed per unit of adsorbent (x/m) as a function of the residual concentration of the organic compound at equilibrium (Ce).

Most of the raw water samples studied, before lime correction, were found to be in the pH range of 5.00 - 5.5, corresponding to an aluminum-buffered domain where the water pH is mainly regulated by aluminum hydrolysis reactions.

The mechanisms involved in the coagulation of organic matter could be more complex and very diverse, because pH conditions the chemical forms of aluminum.

At the optimal pH for their elimination (pH = 5.5 and 6.5), it is possible that aluminum could be found in several forms: free aluminum ion (Al^{3+}), the most reactive to form Al-MO complexes, the hydroxylated monomeric forms [$Al(OH)^{2+}$, $Al(OH)_2^+$] also bind significantly to organic matter (MO).

Finally, it should be noted that pH could itself be influenced by the temperature of the environment, which will have an impact on the solubility of the forms of aluminum and the dissociation of organic substances [13] [32].

Obtaining straight lines with good correlation coefficients for different doses of coagulant and different concentrations of TOC shows that the Freundlich law is well followed and that the adsorption mechanism is predominant for our experimental conditions.

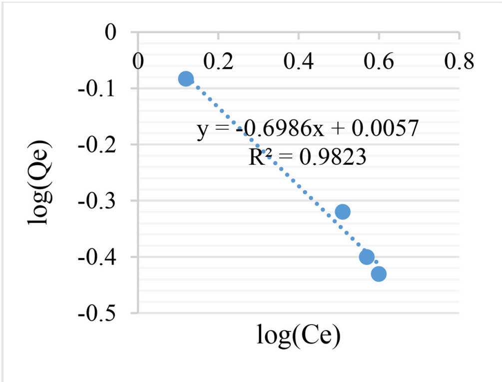
The fact that the TOC/aluminum reaction is not stoichiometric supports the hypothesis that the adsorption of these compounds is purely physical.

Furthermore, for each TOC concentration, the slopes of the lines are almost identical, indicating the same affinity of the compound for aluminum regardless of the coagulant dose.

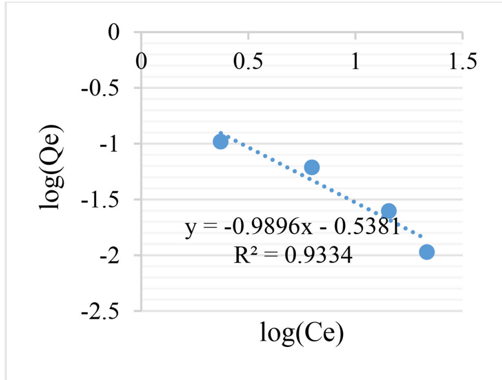
Given the simultaneous presence of several hydrolyzed aluminum species (soluble or insoluble) and the form of organic matter at these different pH levels, the reactions involved are probably numerous.

But the contribution of each type of interaction is often difficult to estimate.

However, it should be remembered that at acidic pH, the cationic species of aluminum are predominant, and one of the possible reactions is the formation of insoluble complexes between organic matter and soluble aluminum (Al-MO) [33].


log(Qe)
y = -0.6986x + 0.0057
R² = 0.9823
log(Ce)

(a)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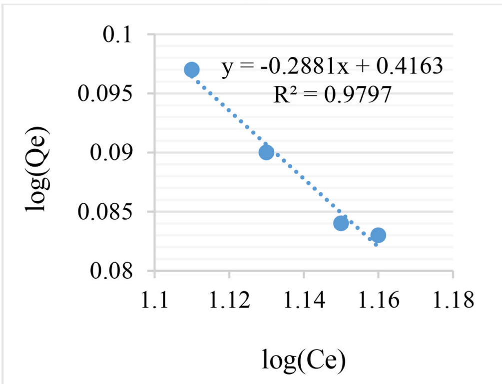
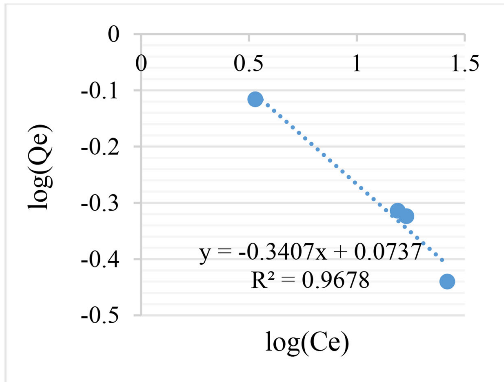
y = -0.9896x - 0.5381
R² = 0.9334
log(Ce)

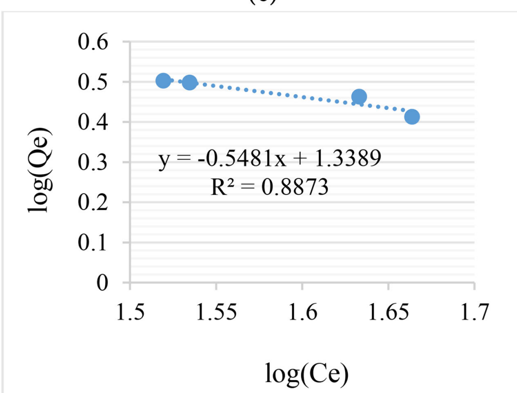
(b)


log(Qe)
y = -0.2881x + 0.4163
R² = 0.9797
log(Ce)

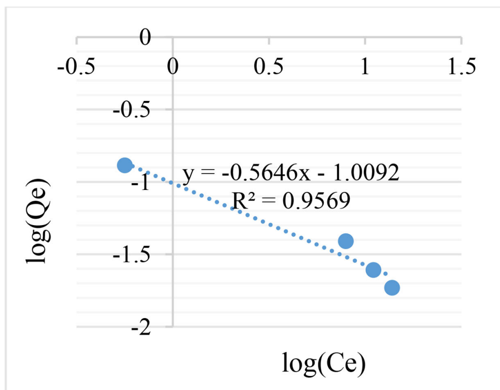
(c)


log(Qe)
y = -0.3407x + 0.0737
R² = 0.9678
log(Ce)

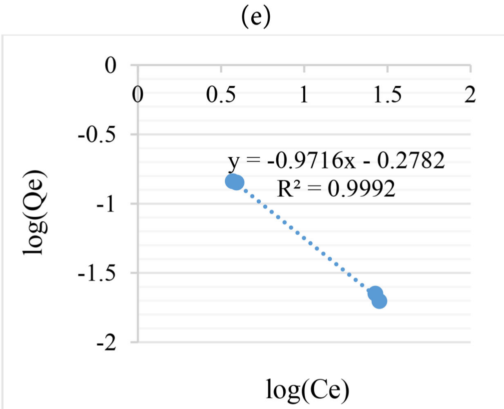
(d)


log(Qe)
y = -0.5481x + 1.3389
R² = 0.8873
log(Ce)

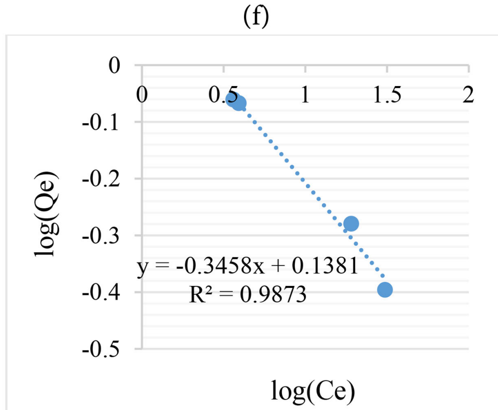
(e)


log(Qe)
y = -0.5646x - 1.0092
R² = 0.9569
log(Ce)

(f)


log(Qe)
y = -0.9716x - 0.2782
R² = 0.9992
log(Ce)

(g)


log(Qe)
y = -0.3458x + 0.1381
R² = 0.9873
log(Ce)

(h)

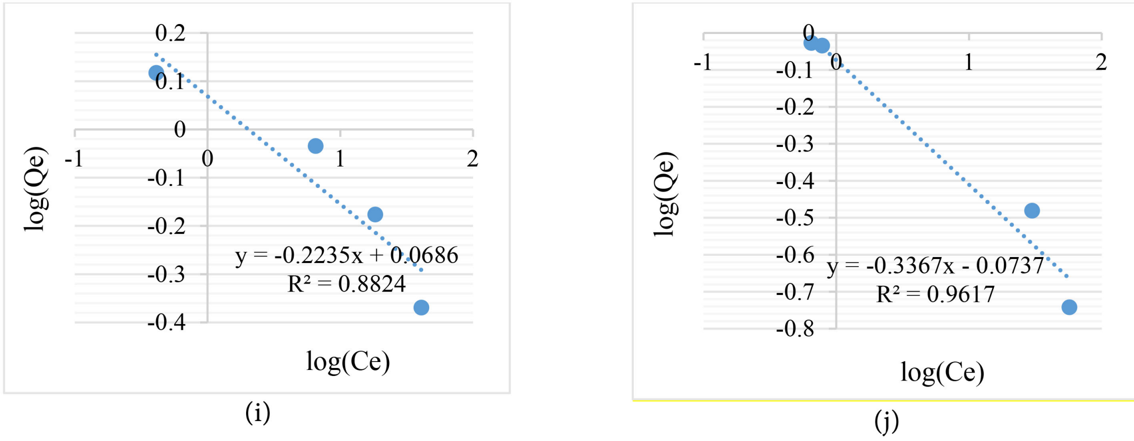


Figure 2. Experimental results of TOC removal according to the Freundlich isotherms.

6. Conclusions

In this study, experimental coagulation-flocculation tests on the surface waters of the Djiri River highlighted appreciable but variable potential for the removal of organic matter by aluminum sulfate.

The effect of coagulation-flocculation on organic matter (TOC) was examined through Jar-test trials in order to evaluate removal yields using different reaction parameters such as coagulant dosage, pH, and initial TOC concentration of the aqueous medium.

The results show that coagulation-flocculation by aluminum sulfate leads to reductions of organic matter of 67% to 92%, respectively.

These reductions are observed respectively for coagulant doses in the range of 1 to 1.5 mL for respective initial TOC levels of 6 to 12 mg/L (Figures 1(a)-(j)), whereas for measured initial organic matter (TOC) values in the range of 14 to 21 mg/L, coagulant doses of 3 to 4 mL would be required (Figure 1(c) and Figure 1(e)).

This observation also shows that the elimination of organic compounds is a function of the initial concentration of these compounds and the pH of the environment.

The influence of pH revealed better elimination at pH between 5.5 and 6.5, with correlation coefficients (R^2) between 0.74 and 0.91.

The exploitation of the Freundlich isotherm through Figure 2 shows very good correlation coefficients for different TOC concentrations and varying doses of coagulant.

This figure shows that Freundlich's law is well followed and that our experimental TOC removal process could be the result of an adsorption phenomenon on the aluminum hydroxide flocs.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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