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ORIGINAL RESEARCH PAPER

# REMOVAL OF PHENOL AND SURFACTANT FROM LANDFILL LEACHATE BY COAGULATION-FLOCCULATION PROCESS

**H. BAKRAOUY[1], S. SOUABI[1*], K. DIGUA[1], A. PALA[2]**

[1] *Hassan II University, Faculty of Sciences and Techniques, Laboratory of Water and Environmental Engineering, BP 146, Mohammedia, Maroc*
[2]*Dokuz Eylül Universisty, Faculty of Engineering, Environmental Engineering Department (ÇEVMER), IZMIR, Turkey*

*Corresponding author: salah.souabi@gmail.com

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**Abstract:** Following the action of rainfall and natural fermentation, the stored waste produces a liquid fraction called leachate. This leachate is rich in organic matter (biodegradable but also refractory) and trace elements. There are many techniques of treating the leachate, in particular, biological, physicochemical, membrane processes. The choice of a technique instead of another depends on several parameters including: the age of the leachate, composition...

In this work we applied a coagulation-flocculation process to treat intermediate landfill leachate of Rabat city with a combined ferric chloride coagulant and a polymer flocculant. We were inspired by full factorial design, including twenty five experiments, to determine optimal dosages of coagulant and flocculant. We operate at *p*H 8.4, the best removal efficiencies obtained were 88 % for Turbidity, 98 % for Phenol and 82 % for surfactant. The optimum dosages values determined by this study were 13.2 $g \cdot L^{-1}$ of coagulant, 62 $mL \cdot L^{-1}$ of flocculant.

**Keywords:** *coagulant, flocculant, coagulation-flocculation, landfill leachate, phenol, surfactant*

## INTRODUCTION

Nowadays, changing lifestyles and the growth of the industry generates more and more municipal and industrial waste.
There are many methods for treating waste, such as incineration, composting, landfilling... Yet, experiments and comparative studies have shown that the most suitable technique, the easier implementing and controlling, and the cheapest is landfilling. Moreover, landfilling minimizes the impact on the environment which is produced by other techniques and allows the waste to decompose under controlled conditions until it is transformed to stabilized waste.
However, the combined effect of percolation of rainwater and natural fermentation of landfilled waste causes formation of a liquid and a biogas very polluted. Biogas can be used to produce energy [1], while the highly contaminated leachate must be treated as not to alter surface water and groundwater.
Leachates contain a large amount of organic matter (biodegradable and non-biodegradable) made especially of humic acids, ammonia nitrogen, organic and inorganic salts, trace elements. It is therefore required to eliminate or reduce the organic matter (represented by COD, BOD...) before discharging the leachates into surface waters. Toxicity analyzes were conducted by Clement and *al*. [2] and Sisinno and *al*. [3], and confirmed the potential dangers of leachate and the need to treat it before discharge aiming to not harm the environment.
Conventional treatments of leachate are classified into three categories: (1) combined treatment with domestic sewage, (2) biodegradation and (3) the physicochemical processes [4].
One of physicochemical processes widely used is coagulation-flocculation. This method can be used as a pretreatment or as a final purification step.
Many researches realized by Kang and *al.* [5], Silva and *al.* [6], demonstrated the efficiency of coagulation flocculation in the treatment of stabilized and old landfill leachates.
Zamora and *al.* [7] and Tatsi and *al.* [8], used this process as pretreatment but also as final polishing step aiming to remove nonbiodegradable organic matter.
Other researchers examined the use of different coagulants aiming to study the assessment of ph effect and optimize the process conditions by determining the most efficient coagulant, in terms of high removal efficiencies of organic matter [8, 9].
In the same context, Apostol and *al.* [10] aimed to determine the optimum conditions to reach high removal efficiency of colloidal particles using Response surface methodology (RSM).
The primary objective of the present research investigation was to examine the efficacy of coagulation- flocculation process using ferric chloride as coagulant and a cationic polymer as flocculant, for the treatment of leachate sample collected from landfill site of Rabat, capital of Morocco. The main emphasis was given to removal of major pollutant as COD, $BOD_5$, color, turbidity, surfactant and phenolic compound.
We will describe in the following, the results obtained for the last parameters: Turbidity, Phenol and Surfactant.

## MATERIALS AND METHODS

### Landfill leachate

The landfill leachate samples used in this study were collected from OUM-AZZA landfill, located in Rabat, Morocco. This site covers a surface of about 110 ha, which has been in operation since 2007, and receives 700 000 T of solid wastes annually (2013).
The climate of Rabat is quite humid due to the proximity of the sea and dam of Sidi Mohamed Ben Abdellah. The average rainfall is 485 mm/year, the number of rainy days per year ranges between 60 and 75.
The average monthly rainfall distribution shows the existence of two distinct rainfall seasons:
- A wet season from October to April (90 % of annual rainfall);
- A dry season from May to September with 10 % of annual rainfall.

The minimum temperatures are reached in winter, and are of the order of 5 °C. The maxima reach 40 °C in summer.

### Analysis of physico-chemical parameters

The chemical oxygen demand (COD), biological oxygen demand ($BOD_5$), surfactants, phenol and other physicochemical parameters of landfill leachate characterization measurement were performed according to standardized methods [11]. The procedures of the analysis realized are listed hereafter.

***COD Analysis***
The chemical oxygen demand (COD) was determined using Open Reflux Method (5220-B). Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion (2 hours), the remaining unreduced $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed and the oxidizable matter is calculated in terms of oxygen equivalent. The excess dichromate $K_2Cr_2O_7$ is determined by Mohr's salt.

***$BOD_5$ Analysis***
The method consists of filling the samples in airtight bottles of specified size and incubating them at specified temperature (20 $^{o}$C) for 5 days in a BOD meter with mercury.

***Turbidity***
The turbidity was determined by a turbidity meter (Model 2100N HACH).

***Determination of Color, conductivity and pH***
Color was determined using a UV/Visible spectrophotometer (Model 9200 UV/VIS). The *p*H was determined by a *p*H meter model 6209. The electrical conductivity was measured by a conductivity meter intelligent *p*H YK-2001PH.

***Determination of phenol***

Samples phenolic compounds, treated with Folin Ciocalteu, develop a blue color whose absorption is measured at 725 nm after standing for an hour in the dark.

***Determination of surfactant***

The determination of surfactant is based on the formation of a soluble complex in toluene between the surfactant, which is an anionic compound, and methyl violet, which is a cationic compound. The reading is carried out spectrometric wavelength of 615 nm.

**Coagulation-flocculation experiments**

Ferric chloride ($FeCl_3 \cdot 6H2O$) is used as coagulant to destabilize the colloidal and suspended solids. The cationic polymer Flocculent was also examined.
Coagulation experiments were achieved in a conventional jar-test apparatus, equipped with 6 beakers of 1L volume in ambient temperature. The experimental process was performed in two stages: the initial rapid mixing for 10 min at 150 rpm, followed by a slow mixing for 20 min at 30 rpm. After 24 h of settling period, the supernatant was withdrawn from the beakers for analysis.
To assess the efficacy of the coagulant and flocculent chosen for leachate treatment, the following parameters were determined: turbidity, phenol and surfactant removals.
The experiments were carried out at *p*H 8.4 as this was the natural *p*H value determined in the original landfill leachate. Coagulant dosages (Ferric Chloride 40 wt %) varied in the range of 12.8 – 14.4 $g \cdot L^{-1}$ at 0.4 $g \cdot L^{-1}$ intervals, while flocculant dosages (Hymoloc DR3000 30 %) ranged from 50 to 66 $mL \cdot L^{-1}$ at 4 $mL \cdot L^{-1}$ l intervals.
Characteristics of coagulant and flocculant used are shown in tables 1 and 2.

***Table 1.*** *Characteristics of coagulant*

| Parameter | Value |
|---|---|
| $FeCl_3$ [%] | 40 |
| Conductivity [$ms \cdot cm^{-1}$] | 20.2 |
| *p*H | 1.40 |

***Table 2.*** *Characteristics of flocculant*

| Parameter | Value |
|---|---|
| Molecular Weight | High |
| Density | 35 [%] |
| Viscosity | <600 [cp] |
| *p*H | 3.0 - 4.1 |
| Cationicity | 1.2 [$g \cdot cm^{-3}$] |
| Appearance | White Liq. |

Twenty-five experiments were carried out under the *p*H value chosen, with different combinations of coagulant and flocculant dosages

**Calculations**

The removal of the studied parameter from leachate was calculated based on the following formula:

$$Removal\ Percentage = \frac{C_0 - C_F}{C_0} * 100 \quad (1)$$

where $C_0$ and $C_F$ are respectively the initial and final concentrations of the studied parameter.

## RESULTS AND DISCUSSION

### Statistical design

A factorial experimental design was used to determine the influence of the reactants dosages on the efficiency of the coagulation-flocculation process. A $5^2$ full factorial experimental design (Two factors each at five levels) was used in this study. The factors chosen were: Coagulant and flocculant dosages. Twenty-five experiments were carried out under a *p*H value equal to 8.4. Turbidity, Phenol and Surfactant removals as responses were selected to evaluate landfill leachate treatment efficiency. Our interest was to determine the optimal dosages of reactants leading to high removal efficiencies and reduced costs.

The factor levels were determined after preliminary tests. Coagulant dosages varied in the range of 32 - 36 $mL \cdot L^{-1}$ at 1 $mL \cdot L^{-1}$ intervals (Coded factors -2, -1, 0, 1, 2), while flocculant dosages ranged from 50 to 66 $mL \cdot L^{-1}$ at 4 $mL \cdot L^{-1}$ l intervals (Coded factors -2, -1, 0, 1, 2). The values corresponding to each level are shown in Table 3.

***Table 3.*** *Factors values corresponding to each level*

| | Levels | | | | |
|---|---|---|---|---|---|
| Factors | -2 | -1 | 0 | 1 | 2 |
| Coagulant Dosage [$g \cdot L^{-1}$] | 12.8 | 13.2 | 13.6 | 14 | 14.4 |
| Floculant Dosage [$mL \cdot L^{-1}$] | 50 | 54 | 58 | 62 | 66 |

### Leachate characteristics

The landfill leachate used in this study has a dark color, which can be explained by the high turbidity (about 15 $g \cdot L^{-1}$) and the high amount of suspended matter (about 28.3 $g \cdot L^{-1}$). The *p*H reached 8.4, thus, the leachate is classified as intermediate. The Concentrations of Surfactant and phenol are a little high.

The characterization of the leachate is shown in Table 4.

The leachate studied was the subject of several analysis and different physicochemical parameters were identified as: *p*H, conductivity, turbidity, TKN, COD, $BOD_5$, $O_2$, $NH^{4+}$ and the metal trace elements.

***Table 4.*** *Landfill leachate characterization*

| Variable | Value |
|---|---|
| Color | Dark |
| *p*H | 8.4 |
| Conductivity [mS·cm$^{-1}$] | 22.8 |
| Turbidity [NTU] | 1619 |
| Colorization | 12,46 |
| COD [mg·L$^{-1}$] | 14 976 |
| $BOD_5$ [mg·L$^{-1}$] | 8724 |
| Phenol [mg·L$^{-1}$] | 241.8 |
| Suspended matter [g·L$^{-1}$] | 28.3 |
| NTK [mg·L$^{-1}$] | 4312 |
| $NH^{4+}$ [mg·L$^{-1}$] | 3738 |
| $P_t$ [mg·L$^{-1}$] | 49.73 |
| $O_2$ [mg·L$^{-1}$] | 0.5 |
| Cl [mg·L$^{-1}$] | 2650 |
| $Cr^{3+}$ [mg·L$^{-1}$] | 1.8 |
| Pb [mg·L$^{-1}$] | 12 |
| Cd [mg·L$^{-1}$] | 0.05 |
| Fer [mg·L$^{-1}$] | 27 |
| Hg [mg·L$^{-1}$] | 0.003 |
| Zn [mg·L$^{-1}$] | 6 |
| Cu [mg·L$^{-1}$] | 11 |

The results are given in Table 4. We see that the leachate contains a high inorganic and organic pollution load.

OUM AZZA’s leachate is characterized by a high organic load in terms of COD and $BOD_5$ which reach 15 g·L$^{-1}$ and 9 g·L$^{-1}$ respectively.

The study of $BOD_5$/COD ratio is a good indicator of the biodegradability of an effluent. The higher this ratio is, the more the effluent indicates important biodegradability. This is the case of our leachate since the ratio of $BOD_5$/COD is about 0.58.

High concentration of ammonium-nitrogen was measured (3738 mg·L$^{-1}$) and TKN (4312 mg·L$^{-1}$), while the principal heavy metal concentrations reached 1.8 m g·L$^{-1}$ for Cr, 0.05 mg·L$^{-1}$ for Cd and 12 mg·L$^{-1}$ for Pb.

The main factors affecting the characteristics of the leachate: The age of the landfill, the type of waste deposited and other hydrogeological factors.

## Coagulation-flocculation results

The Jar Test was done with different coagulant and flocculant dosages. In order to obtain an effective range of studied factors, some preliminary studies with wide coagulant dose and flocculant dose ranges of 3.2 - 26.4 g·L$^{-1}$ and 8 - 92 mL·L$^{-1}$ respectively, were carried out prior to the experiments. As a result, the ranges of coagulant dose chosen in this study were 12.8 - 14.4 g·L$^{-1}$ for coagulant dose. The flocculant dose worked well in the range of 50 - 66 mL·L$^{-1}$. The amount of residual

pollutant in effluent is measured at the end of each experiment. Optimum coagulant and flocculant dosages were determined by observing maximum removal of pollutant.
Table 5 shows the experimental matrix for the factorial design and the results of Turbidity, Phenol and Surfactant removals.

***Table 5.*** *Turbidity, Phenol and Surfactant removals efficiencies for all experiments*

| Sample | Vcoagulant [g·L$^{-1}$] | Vfloculant [mL·L$^{-1}$] | Turbidity Removal [%] | Phenol Removal [%] | Surfactant Removal [%] |
|---|---|---|---|---|---|
| 1 | 12.8 | 50 | 91.1 | 75.1 | 71 |
| 2 | 12.8 | 54 | 90.7 | 89.7 | 68 |
| 3 | 12.8 | 58 | 97.2 | 89.7 | 94 |
| 4 | 12.8 | 62 | 94.0 | 90.4 | 88 |
| 5 | 12.8 | 66 | 91.0 | 91.0 | 76 |
| 6 | 13.2 | 50 | 92.2 | 65.6 | 84 |
| 7 | 13.2 | 54 | 90.5 | 90.9 | 88 |
| 8 | 13.2 | 58 | 88.3 | 50.7 | 82 |
| 9 | 13.2 | 62 | 87.7 | 97.5 | 82 |
| 10 | 13.2 | 66 | 84.6 | 69.9 | 92 |
| 11 | 13.6 | 50 | 82.7 | 30.7 | 90 |
| 12 | 13.6 | 54 | 80.2 | 38.0 | 87 |
| 13 | 13.6 | 58 | 70.2 | 89.5 | 89 |
| 14 | 13.6 | 62 | 72.1 | 91.0 | 93 |
| 15 | 13.6 | 66 | 68.9 | 87.5 | 78 |
| 16 | 14 | 50 | 73.7 | 88.5 | 86 |
| 17 | 14 | 54 | 70.8 | 90.6 | 68 |
| 18 | 14 | 58 | 71.5 | 91.5 | 82 |
| 19 | 14 | 62 | 61.7 | 91.3 | 95 |
| 20 | 14 | 66 | 58.6 | 87.2 | 87 |
| 21 | 14.4 | 50 | 60.3 | 90.9 | 94 |
| 22 | 14.4 | 54 | 46.8 | 87.5 | 90 |
| 23 | 14.4 | 58 | 50.8 | 87.7 | 81 |
| 24 | 14.4 | 62 | 38.2 | 86.5 | 87 |
| 25 | 14.4 | 66 | 47.5 | 91.4 | 74 |

***Turbidity removal***

Many studies investigate the removal of turbidity using the coagulation-flocculation process, because it's one of the most important parameters used to improve the treatment efficiency.
The first parameter we investigate was turbidity removal. 97.2 % was the maximal removal efficiency obtained by adding 12.8 g·L$^{-1}$ of ferric chloride and 58 mL·L$^{-1}$ of flocculant.

We observed that tubidity increases by adding high dosages of coagulant, this phenomenon could be attributed to the restabilization of colloidal particulates when the coagulants were used at dosages in excess of the optimum value.
Figure 1 shows the variation of turbidity removal in function of coagulant and flocculant dosages.

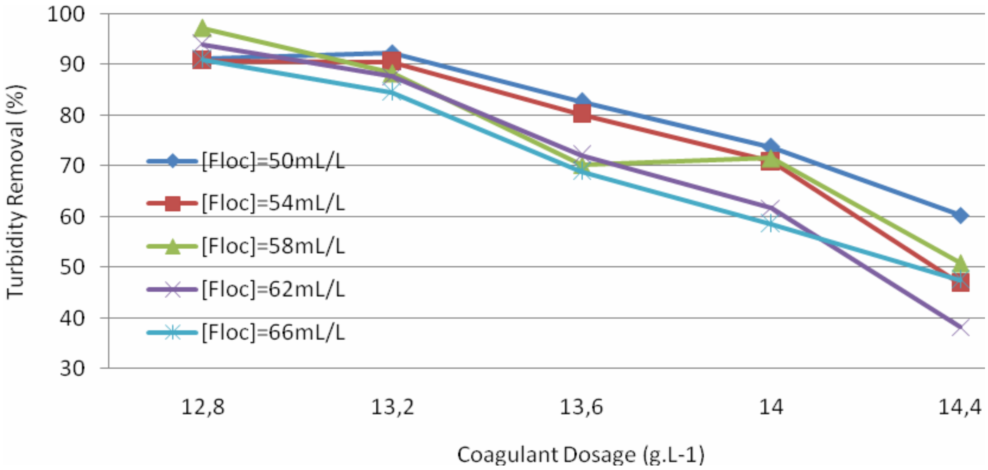


***Figure 1.*** *Turbidity removal pattern at different Coagulant dosages with different flocculant addition*

The application of RSM to optimize CF process for landfill leachate pretreatment using iron-based coagulants was investigated by Liu and *al.* [12]. The results demonstrated that RSM was an effective method for the optimization of experimental parameters in the treatment of landfill leachate. The parameters studied were: COD, color, turbidity and Humic Acid removals. Under the optimum conditions, the appreciable turbidity removal efficiencies were 98.85 % for $FeCl_3{\cdot}6H_2O$, 94.13 % for $Fe_2(SO_4)_3{\cdot}7H_2O$ and 89.79 % for Polyferric Sulfate were respectively achieved.
Maranon and *al.* [13] conducted many experiments of application of coagulation flocculation process in pretreatment of young landfill leachate. They tested three coagulants: ferric chloride, aluminium sulphate and aluminium polychloride (PAX), along with different types of flocculants. The best results were found using PAX, obtaining about 98 % of turbidity removal.
In Ghafari and *al.*'s research [14], the efficiency Poly Aluminum Chloride (PAC) in the treatment of stabilized leachate was studied. The removal efficiencies for COD, turbidity, color and total suspended solid (TSS) obtained using PAC were compared with those obtained using alum as a conventional coagulant. Central composite design (CCD) and response surface methodology (RSM) were applied to optimize the operating variables: *p*H and coagulant dosage. They demonstrated turbidity removal efficiencies of 94.0 % for PAC, and 88.4 % for alum.

***Phenol removal***

The maximal removal efficiency obtained reached 97.5 %. This is a high amount compared to the results obtained by Motling and *al.* [15], whose study, based on the combination of coagulation-flocculation process and adsorption, leaded to removal of 92 % of initial phenol concentration.

From Table 5 we see that for a given dose of coagulant, we always obtained removal efficiencies approaching 90 %, adding 58 - 60 mL·L$^{-1}$ of flocculant.
Figure 2 shows the effect of different coagulant and flocculant dosages on the removal of Phenol. It is clear that both coagulant and flocculant dosages have a significant effect on the removal efficiency.

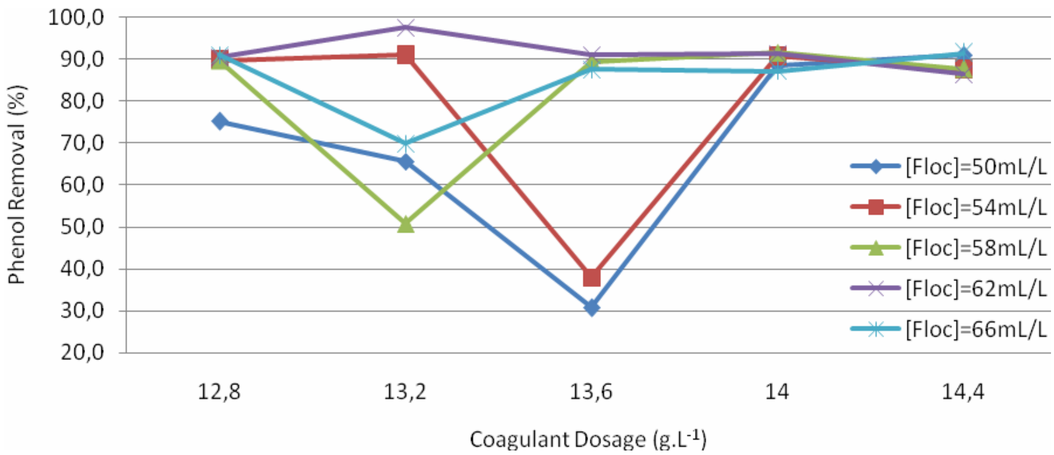


***Figure 2.*** *Phenol removal pattern at different Coagulant dosages with different flocculant addition*

The plots show that, as the amount in coagulant dosages increases, the phenol removal increases in a great extent, however it was also observed that after reaching to the optimum doses, the phenol removal started declining. The above trend perhaps attributed to the fact that in excess of optimum dosages of coagulant and flocculant, colloidal organics further restabilised over and above the optimum dosage.
Referring to Figure 1, the maximum removal efficiency reached 97.5 %, by adding 13.2 g·L$^{-1}$ of ferric chloride coagulant and 62 mL·L$^{-1}$ of cationic flocculant.
Landfill leachate contains a large number of dangerous compounds, such as aromatics, halogenated compounds, heavy metals, phenols, pesticides, and ammonium, which are considered dangerous even in small amounts.
Phenolic compounds are present in the environment, they are considered toxic. These compounds detected in the leachate include cresols, phenol, and substituted as well as chlorinated phenols.
Benfenati and *al.* [16] demonstrated that cresols and phenol (which are short-chain phenols) contained in leachates of urban and industrial landfills, originate from various types of wastes. Phenol and its substitutes are commonly produced by the transformation of several pesticides [17].

***Surfactant removal***

Existence of surfactants in the leachate was attributed to the disposal of laundry detergents or bath soaps in the waste stream [18].
Foo and *al.* [19] revealed that stabilized leachate contain high amount of polar and nonpolar organic constituents, expressed in term of COD.

To eliminate surfactants, Borghi and *al.* [20] proposed an adsorption process using iron oxide powders which are non toxic and at low cost.
The surfactant removals from the landfill leachate are shown on Figure 3.

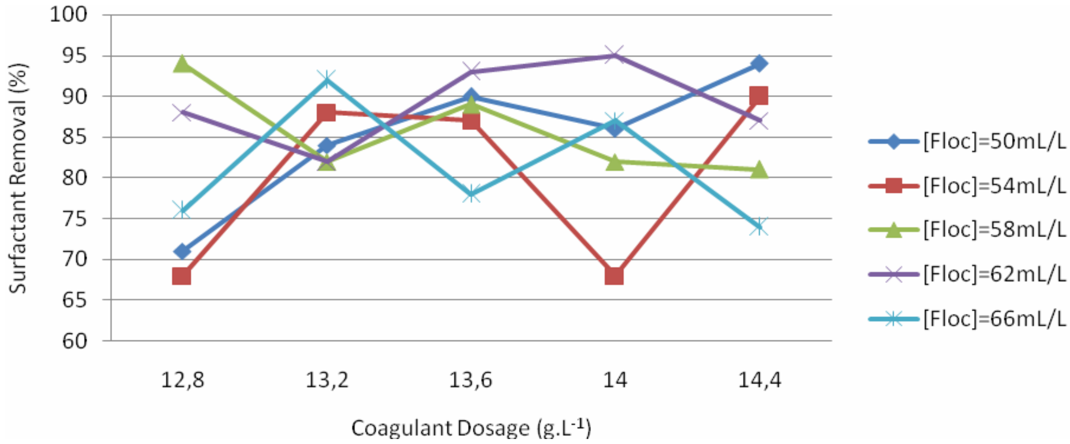


***Figure 3.*** *Surfactant removal pattern at different Coagulant dosages with different flocculant addition*

From Table 5, we concluded that the maximum removal reached for surfactant was 95 %, by adding 14 g·L$^{-1}$ of coagulant and 62 mL·L$^{-1}$ of flocculant.
In fact, we see that the adding of 62 mL·L$^{-1}$ of flocculant to whatever dosage flocculant gives good results.
The effect of coagulant and flocculant dosages on surfactant removal is shown in Figure 1. We obtained a mean removal of approximately 84 % of the initial surfactant concentration, which is an indicator of the efficiency of the coagulation-flocculation process to reduce such a pollutant.
In literature there is no agreement on the degree of ecological danger of synthetic surfactants. But many publications revealed the negative effects of surfactants on organisms [21, 22].
Surfactants present in the market today are considered non or low toxic. The toxicity of surfactant is related to the hydrophobicity of the molecule and the length of alkyl chain. Cavalli and *al.* [23] demonstrated that when the number of ethylene oxide units increases, the hydrophobicity decreases and reduces the toxicity. Ostrumov and *al.* [21] demonstrated that surfactants are different from the conventional pollutants because they stimulate enzymatic activities of aquatic organisms biodegradation is usually used to treat effluents containing surfactants, The process can be aerobic or anaerobic depending on the microorganisms present in water and the kind of surfactant [22 - 24].
Ostrumov and *al.* [21] and Ying and *al.* [22] demonstrated that non-ionic and cationic surfactants decompose slower compared to anionic surfactant.

## Process optimization

In this study, the primary purpose of the optimization is to confirm the optimal values of independent variables for landfill leachate treatment with coagulation-flocculation process from the models obtained by experimental data. Otherwise, removal efficiency

of Turbidity, Phenol and Surfactant are individual parameters, and their optimization was achieved under different optimal conditions. Thus, a compromise among the conditions for the three responses is desirable.
Referring to Table 5 and the plots on Figures 1, 2 and 3 we observed that at 13.2 g·L$^{-1}$ the responses reached high values for all the flocculant dosages chosen.
Note that in addition to these responses, we have also measured the removal of color.
Therefore, to search the optimal point, we trace the curves of the responses removals at 13.2 g·L$^{-1}$ at different flocculant dosages, which are shown on Figure 4.

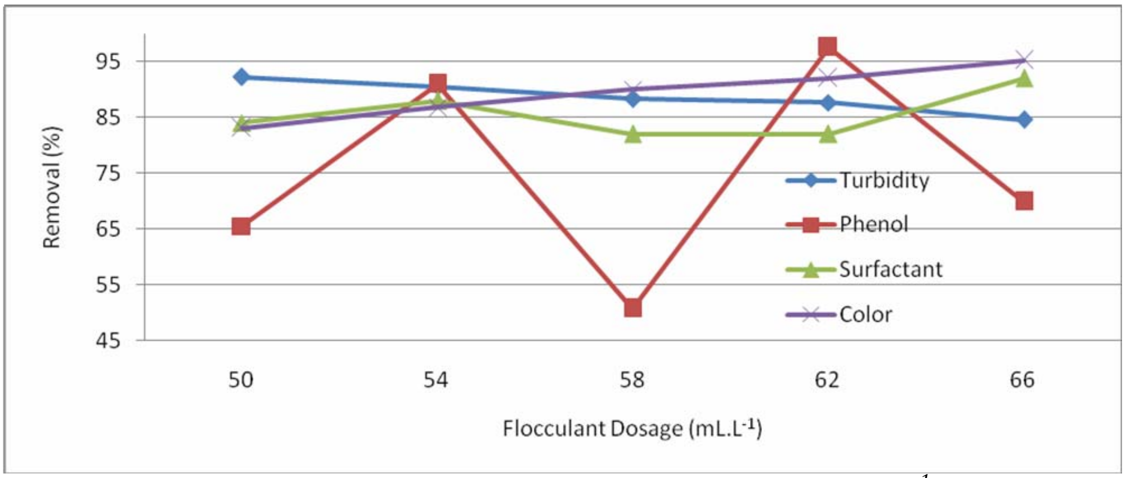


***Figure 4.*** *Turbidity, Phenol, Surfactant and Color removal at 13.2 g·L$^{-1}$ of coagulant with different flocculant addition*

As a result, at *p*H equal to 8.4, the optimal removals for turbidity, phenol and surfactant reached 88 %, 98 % and 82 % respectively. This was obtained by adding 13.2 g·L$^{-1}$ of Coagulant and 62 mL·L$^{-1}$ of flocculant.

## CONCLUSIONS

The application of experimental design (ED) to optimize Coagulation-Flocculation process for landfill leachate treatment using iron-based coagulant was investigated.
The results demonstrated that ED was an effective method for the optimization of experimental parameters in the treatment of landfill leachate. 25 experiments were carried out by adding different dosages of coagulant (Ferric Chloride) and flocculant (Polymeric), and three responses were analyzed: Turbidity, Phenol and Surfactant.
It was found that the coagulant is the most significant factor. As a compromise among simultaneous removals of Turbidity, Phenol and Surfactant from landfill leachate, the optimum conditions obtained were 13.2 g·L$^{-1}$ of Ferric Chloride and 62 mL·L$^{-1}$ of flocculant at initial *p*H 8.4. Under these conditions, the removals for Turbidity, Phenol and Surfactant reached 88 %, 98 % and 82 % respectively.
Therefore, Ferric chloride is recommended for landfill leachate treatment in a large scale, providing a simpler, more controllable and less reagent-consuming technique to the Coagulation-Flocculation process.

Additionally, the results also offered a certain reference value for the practical application of Coagulation-Flocculation process using iron-based coagulants in intermediate leachate pretreatment for recalcitrant organic substances removal.

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