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Removal of butyl acetate in an industrial effluent through a physicochemical treatment.

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Butyl acetate is an organic compound widely used in industry, focusing on industrial paint waste effluents. Exposure to this pollutant at an average of 925 ppm can cause dizziness, drowsiness, respiratory tract irritation, and central nervous system suppression. That is why it seeks to minimize the risk associated with this contaminant through treatment alternatives. The characterization of the sample yielded the detection of the following contaminants: methyl ester, 1-butanol, 1-4 dioxane, butyl ester, 1-chlorobutane. The objective of this work was the design and development of combined treatments for the removal of butyl acetate in industrial waste effluents. For this, a physicochemical treatment coupled to an advanced oxidation process (PAO) was implemented. The results showed that the coupled treatments are efficient for the removal of butyl acetate, methyl ester, 1-chlorobutane and 1-butanol, while for 1-4 dioxane it was reduced by approximately 83%. The study was complemented with the evaluation of the following parameters, resulting in a decrease in electrical conductivity of 99.5%, total suspended solids of 74%, color of 93%, turbidity of 55% and COD of 73%.

Keywords: industrial effluents, Physicochemical treatment, Advanced oxidation process (PAO).

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INTRODUCTION

Butyl acetate is an ester with a fruity odor, it can be produced at industrial and laboratory level by Fischer esterification, in the presence of the butanol isomer and acetic acid using a catalyst (sulfuric acid), it is a colorless liquid highly flammable and hazardous to health, affecting the central nervous system (NIOSH, 2019). It is used for the manufacture of synthetic wood, lacquers, varnishes, perfumes, printing inks, solvents and solvents in polyurethane resins and in paints (paint strippers) for special applications (Kobe, 1958; Kirk-Othmer, 1998; Castellani, 2014), among others. Due to the increase in industrial activity, it has caused a negative impact on access in quantity and quality of the water resource, this due to the indiscriminate use, pollution and final disposal of effluents without prior treatment (Mastroianni et al., 2017). The contamination of surface and groundwater has been generated by high concentrations of various chemical species. These come from diffuse (pesticide use, manure production in agriculture) and point sources of pollution (industrial landfills, as well as in industrial mining practices) (Mastroianni et al., 2017). Effluents from industrial processes contain toxic products, harmful organic compounds and metals, varying both in composition and in type and concentration, depending on the processes that generate them (Hernández and González, 1993). Natural organic compounds in the environment tend to degrade slowly and naturally into less harmful components; however, the volatile organic compounds (VOCs) found in effluents from industrial processes are not considered biodegradable (CONAMA, 1998).

Several studies have reported that when OWP/adsorption, OWP/biological process and OWP/coagulation-flocculation to name a few are combined, they show favorable and low-cost results (Sala et al., 2014; Ghasem. 2005). It is also known as hybrid technologies, which have been applied in the treatment of highly contaminated wastewater from the olive, textile and paper industries, with heavy metals, leachates from landfills, pesticides, among others, without leaving aside the aeronautical industry, which produces waste with a high load of hazardous organic compounds (Koprivanac et al., 2006).

In the present work we found that under a physicochemical treatment and an advanced oxidation process it was possible to completely remove the butyl acetate present in the industrial waste effluent, so our methodology could be relevant in treatment systems contaminated with this organic compound.

Experimental

The sample was provided by a waste collection company, which was taken by the same company to the Universidad Autónoma de Querétaro, Airport Campus (laboratory building), which was stored in a totem with a capacity of 1000 L, kept in the shade at room temperature.

Jar testing

A series of jar tests were carried out modifying pH, concentration of metal precipitator, coagulant, and flocculant, which were performed using a stirrer (Phipps & Bird, U.S.A.), for testing 6-seater jars with six 1 L beakers, the stirring time used was 10 min at 150 rpm and 15 min of rest, following the methodology of Arboleda, (2000), Table 1 shows the solutions used in the tests.

Table 1. Concentrated solutions defined

Chemical reagent	Concentration (mg/L)
CYQBAMET50 Metal Precipitator	1 (w/v)
CYQBAMET50	1 (w/v)
CYQBAFLOCK 90/10 Coagulant	1 (w/v)
Flocculant CYQBAFLOCK 70/24	1 (w/v)
Calcium oxide CALMOSACORP	0.1 N

The selected test is described below:

The test was performed by adjusting the pH from 3 to 2 with 1.8 mL of 98 % 2HSO4, 2 mL of 1 % metal precipitant was added, the pH was again adjusted from 2 to 7 with 45 mL of 20 % calcium oxide, then 2.5 mL of 10 % coagulant 90/10 was added, and finished with the addition of 1.3 mL of 1 % flocculant 70/24, as shown in Figure 1.

Figure 1. The sample is observed after the physicochemical treat-

Once the jar test was completed, it was reproduced at a volume of 80 L, under the same conditions, as shown in Figure 2.

The efficiency of the physicochemical process was evaluated by the total solids test, following NMX-AA-034-SCFI-2001.

Figure 2. The test was reproduced at a volume of 80 L.

Filtration system

An acrylic column was used, which was previously packed with activated carbon, sand and 1-1.5 cm silica gravel, using 15 cm of height in each layer.

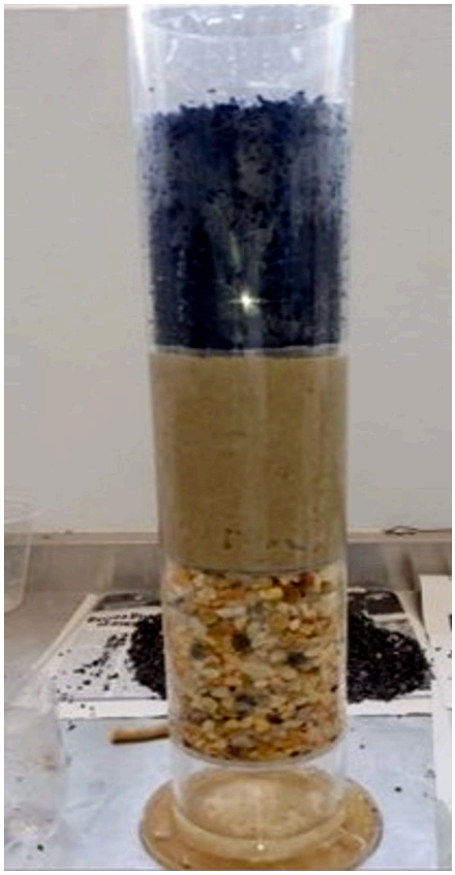

Figure 3. Filtration system packed with activated carbon, sand and silica gravel.

RESULTS AND DISCUSSION

Characterization of the sample

The quantification of butyl acetate in the initial and treated samples was carried out by gas chromatography coupled to mass spectrophotometry at the Center for Academic Studies on Environmental Contamination (CEACA), obtaining chromatograms of the initial sample as shown in Figure 4.

It was also sent to an intertek+ABC Analitic certified laboratory, complementing the analysis and identification of heavy metals, volatile and semi-volatile organic compounds. The Table 2. Shows that chromium had the highest value of 1.9002 mg/L compared to barium with a concentration of 0.0630 mg/L, followed by mercury with 0.0210 mg/L and cadmium with a value of 0.0036 mg/L, which are within the maximum permissible limits of the standard.

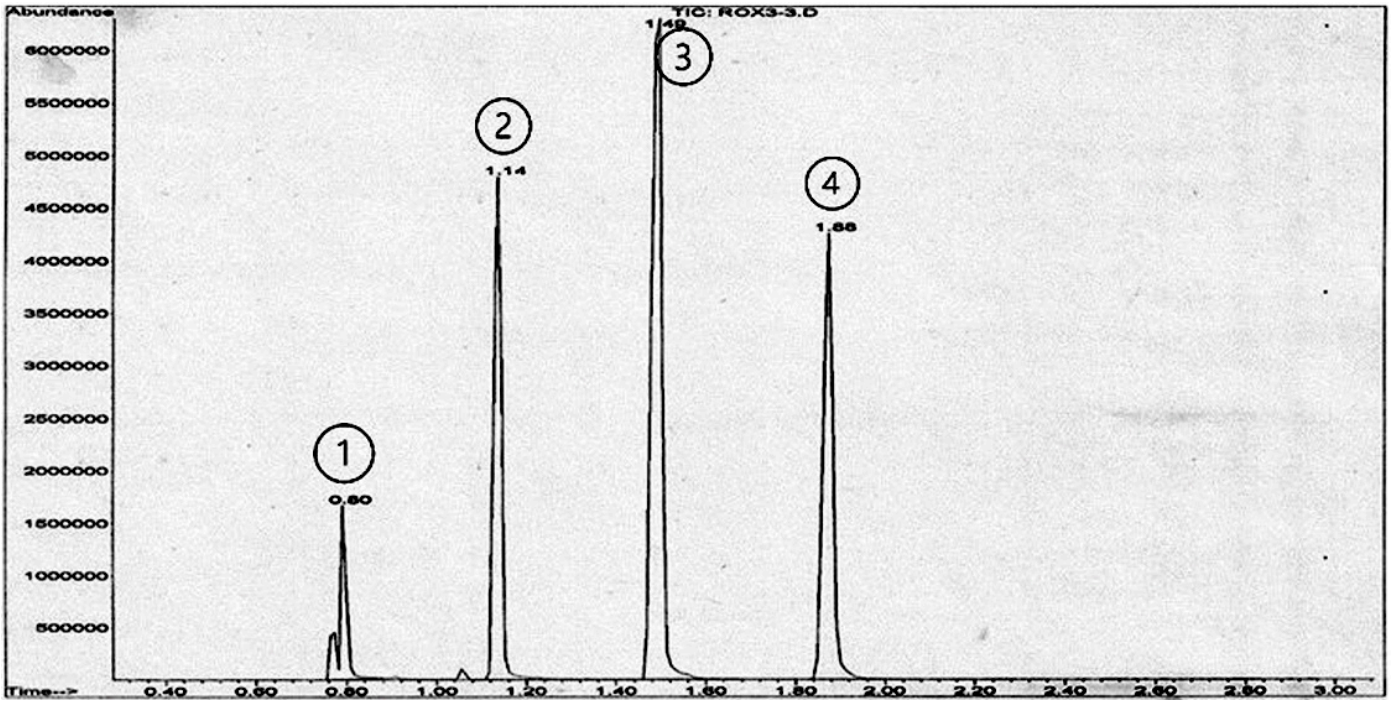

Figure 4. Chromatogram of the initial test sample. Shows the chromatogram of the compounds detected from the initial sample, resulting in 1) 1-chloro butane with 103,485 abundances, 2) 1-butanol with 5,813,946 abundances, 3) 1-4 dioxane with 10,365,888 abundance and 4) butyl ester with 6,506,138 abundances.

The sample was initially characterized according to NOM-052-SEMARNAT-2005. The CRIT characterization yielded the following results: it presented the characteristics of corrosivity, reactivity, flammability and toxicity to the environment.

Table 2. Detection of metals in the initial sample.

Parameter	Method	Concentration (mg/L PECT)	LMP (mg/L PECT)
Barium	US EPA 6010C-2007	0.0630	100
Cadmium	US EPA 6010C-2007	0.0036	1.0
Chrome	US EPA 6010C-2007	1.9002	5.0
Mercury	US EPA 7470A-1994	0.0210	0.2

MPL Maximum permissible limit; PECT Leachate from which the toxic constituents of the waste and their concentration are determined.

Detection of volatile organic compounds

According to the chromatogram of the untreated test sample, the following compounds were present: butyl ester, 1-4 dioxane, 1-butanol, nitrogen, 2-butene and water, while the physicochemically treated sample showed the identification of seven organic compounds: (1) bromochloromethane, (2) chloroform, (3) 1-2 dichloroethane, (4) 1-4 difluorobenzene, (5) toluene, (6) chlorobenzene and (7) 4-bromofluorobenzene, as shown in Figure 5.

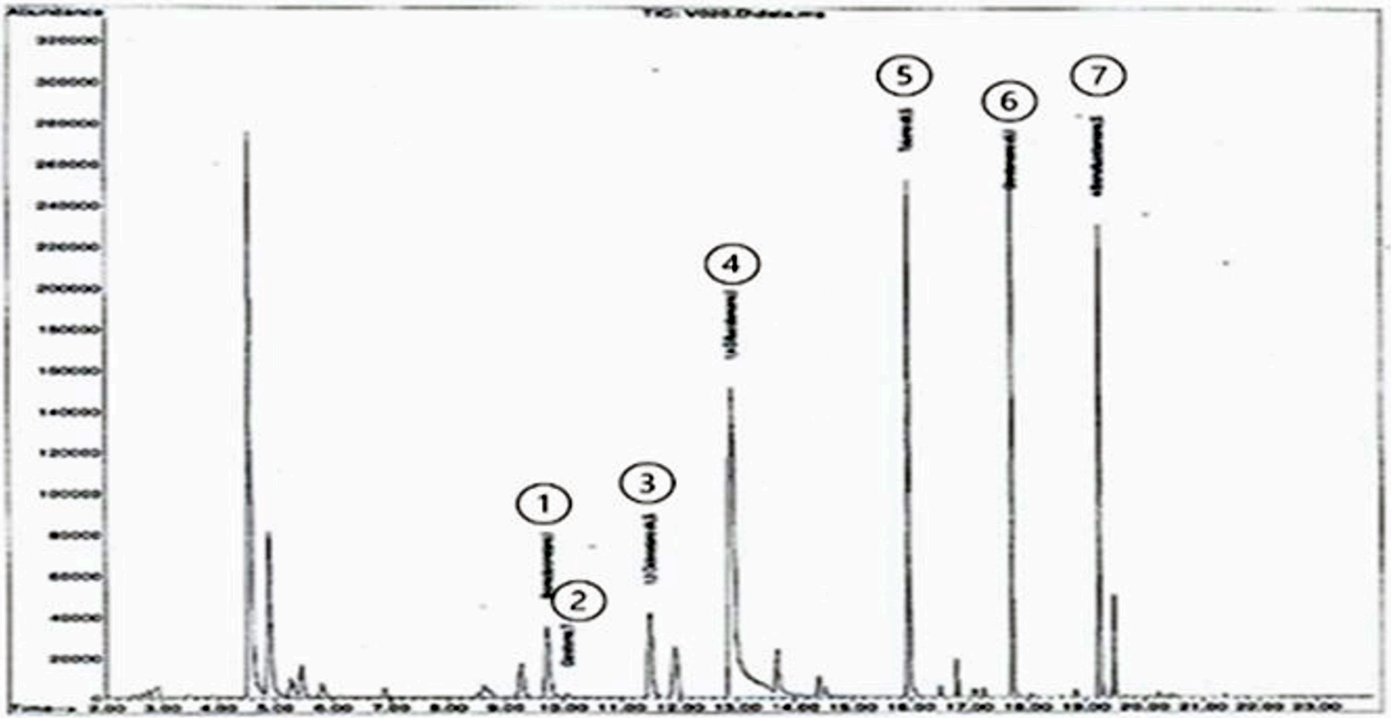

Figure 5. Chromatogram of majority volatile organic compounds present after physicochemical treatment: (1) bromochloromethane, (2) chloroform, (3) 1-2 dichloroethane, (4) 1-4 difluorobenzene, (5) toluene, (6) chlorobenzene and (7) 4-bromofluorobenzene, on the “x” axis is the relative abundance and on the “y” axis is the retention time.

Detection of semi-volatile organic compounds

The identification of the majority compounds in the sample in a retention time of 3 to 14 min, gave a total of twelve detected compounds indicated with number, being the following: 2-fluorophenol (1), phenol (2), 1,4-dichlorobenzene (3), nitrobenzene (4), naphthalene (5), 2-fluorobiphenyl (6), acenaphthene (7), 2,4,6 tribromophenol (8), phenanthrene (9), terphenyl (10), chrysene (11) and perylene, (12) which are shown in Figure 6.

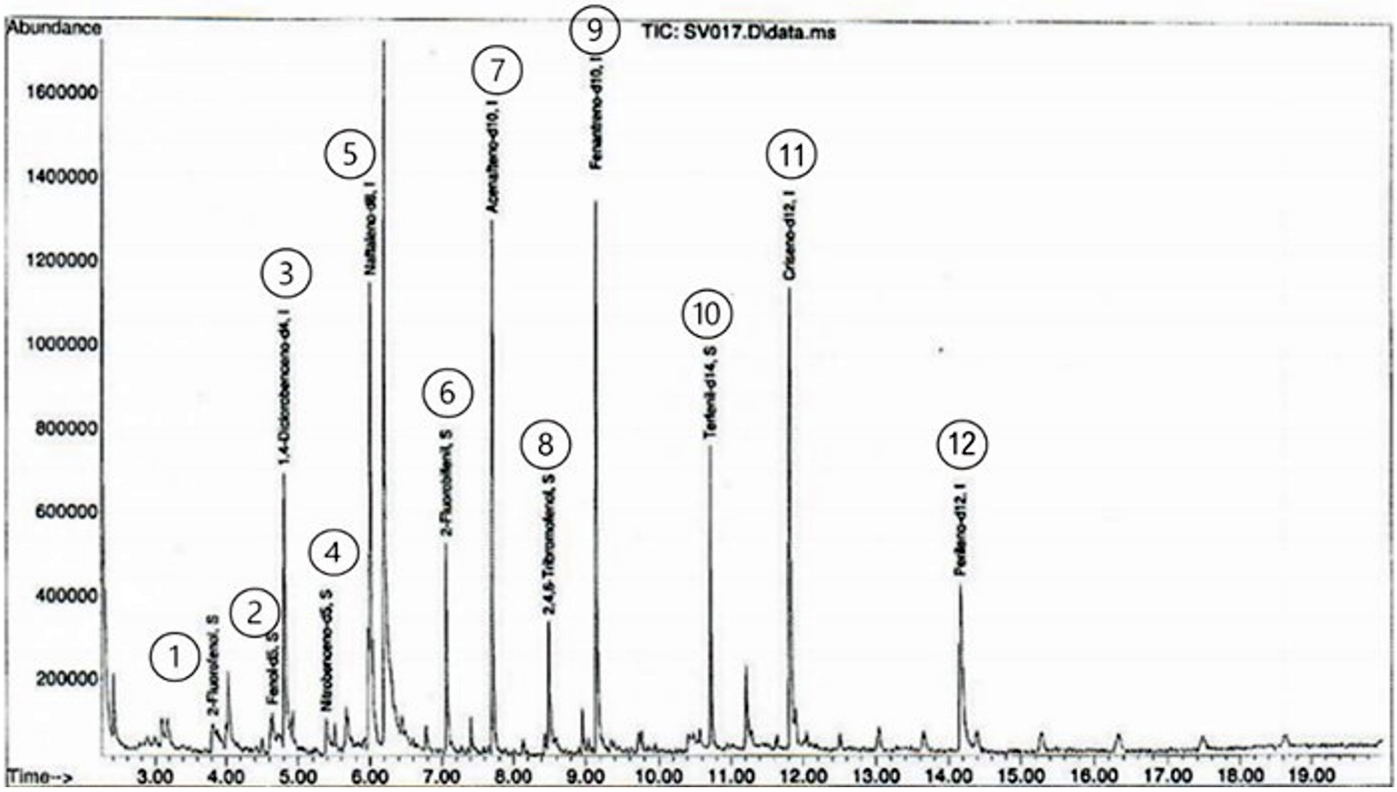

Figure 6. Chromatogram of semi-volatile organic compounds of the treated sample from the physicochemical process, where it can be observed that the base peak is found in the phenanthrene compound (9) with the highest abundance, followed by acenaphthene (7).

Physicochemical treatment

Once the jar test was completed, the precipitation of total solids was obtained in an average amount of 0.88 g, confirming with a solids test, as shown in Table 3.

Table 3 Results of the solids test.

Test number	Average initial weight (g)	Final average weight (g)	Average solidweight (g)
1	47.53±0.2440	46.71±0.2230	0.82±0.11
2	49.75±0.2590	48.87±0.2520	0.88±0.16
3	48.87±0.2510	48.20±0.2442	0.67±0.18
4	49. 62±0.2540	49.00±0.2572	0.62±0.17
5	48.63±0.2500	47.84±0.2450	0.79±0.15

Table 4 Determination of physicochemical parameters of the treated sample obtained after physicochemical treatment.

Parameter	Unit	Untreated initial result	Solids testing	Final result treated
Electrical conductivity	µs	1,392,500	--	7,298
Total suspended solids	Mg/l	139,000	0.8826	36,000
Color	Upt-co	1,507,000	--	100,000
Turbidity	Ntu	7,560	--	3,400
COD	Mg/l	35,720	--	9,590
pH	N/a	3.2	2	7

Once the physicochemical treatment was completed, the sample was passed through a filtration system to remove entrained solids and remove color. The sample was analyzed for volatile compounds by gas chromatography coupled to mass spectrometry, where (1) 1- butanol with an abundance of 110,000 and (2) 1-4 dioxane with an abundance of 1,070,811 were identified as the majority compounds, as shown in Figure7.

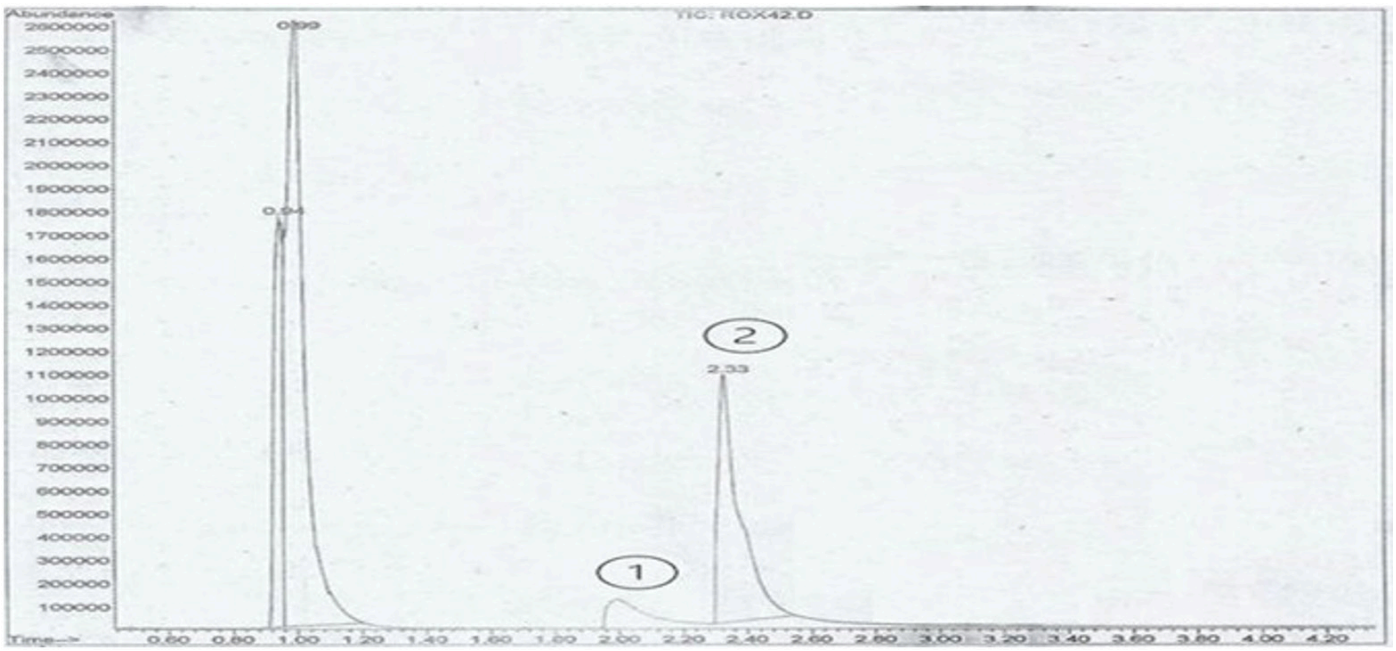

Figure 7. Chromatogram of the treated sample after physicochemical treatment.

After treatment, the sample was again characterized according to NOM-052-SEMARNAT-2005. The CRIT characterization yielded the following results: No corrosivity, reactivity, flammability and toxicity to the environment.

CONCLUSIONS

The initial sample presented CRIT characteristics and at the end of the treatment it no longer presented CRIT characteristics.

The physicochemical treatment proved to be effective for the removal of the compounds 1-chloro-butane, 1-butanol.

On the other hand, the 1-4 dioxane compound presented an abundance of 10,365,888 initial and after treatment presented a reduction in abundance of 110,000 and the butyl ester compound presented 6,506,138 initial abundance, and 1,070,811 final abundances.

Physicochemical treatment is a treatment option for this type of effluent and removal of some compounds.

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