Adsorption of Ni (II) from Water by Natural Bentonite: Studies on Equilibrium Isotherm, Kinetics

Sabrina Berkani and Faroudja Mohellebi

Abstract−The aim of this work is to investigate the adsorption potential of commercial natural bentonite in the removal of Ni (II) ions from aqueous solution. The characterization studies were performed using X-ray Diffraction, Fourier Transform Infrared Spectrometer, thermal gravimetric analysis, Differential Thermal Analysis and Fluorescence spectroscopy. Capacity and specific surface areas are also given. The results show that our sample is a sodic montmorillonite who contains some impurities like quartz and calcite. We also studied the effect of thermic and acid activation on the adsorption of Ni (II) on bentonite. Batch adsorption studies were carried out with various initial Ni (II) concentrations, adsorbent dosages, pH, and contact time. The kinetic studies showed that the nickel adsorption process followed a pseudo-second order. The adsorption data were fitted to the Freundlich and Langmuir isotherm models. This behaviour is described by a monolayer Langmuir type isotherm. The adsorption process is found to be a second order kinetics.

***Keywords*−Bentonite, Adsorption, Heavy Metals, Thermic Acid Activation, Freundlich and Langmuir Isotherm.**

NOMENCLATURE

AAS	atomic absorption spectrophotometer
BET	Brunauer-Emmett-Teller
CEC	Cation exchange capacity
FTIR	Fourier transform infrared spectroscopy
TGA	Thermogravimetric analysis
XRD	X-Ray diffraction
XRF	X-ray fluorescence

I. INTRODUCTION

Nowadays, the problem of heavy metals became increasingly alarming. A common characteristic of the industrial effluents is related to the fact that they almost always contain toxic metals. The environmental protection forces to limit the contents of these metals to the allowed maximum.

Adsorption is one of the most important physicochemical processes that occur at the solid-liquid and solid-gas interfaces. Adsorption has become a preferred method for removal, recovery and recycling of toxic heavy metals from wastewater [1]. Different conventional and non-conventional type of adsorbents have been tried for removal of various metal ions [2], activated carbon [3], tree fern [4], coconut coir pith [5], sewage sludge [6], silica [7], polymetallic sea nodule [8], modified zeolite [9], etc.

The clay minerals in soil play the role of a natural scavenger by removing and accumulating contaminants in water passing through the soil. The mechanism of these processes is usually through ion exchange and adsorption. The high specific surface area, chemical and mechanical stability, layered structure, high cation exchange capacity (CEC), and tendency to hold water in the interlayer sites have made the clays excellent adsorbent materials [9].

Pure clay minerals, particularly naturally occurring bentonite has been used as a favoured adsorbent for removal of various metals from aqueous medium. Uses of bentonite for adsorptive removal of Zn(II) [11], natural and Na-exchanged-bentonite for removal of Cr(III), Ni(II), Zn(II), Cu(II) and Cd(II) from water [12] and even kaolinite for Mn(II), Co(II), Ni(II), Cu(II) have yielded significant adsorption [13]. Other important results include adsorption of Cu(II) and Zn(II) on surfactant modified montmorillonite [14].

Bentonites are highly valued for their sorptive properties that could possibly be improved by chemical treatment or by heat treatment, which stem from their high surface area and their tendency to absorb water in the interlayer sites. Acid activation increases the surface area [15], [16], [17]; [18], and modifies the structure of smectites [18], [19], [20], [21].

The most important physical changes in activated smectites is the increase of their specific surface area and the average pore volume [17], [19], [22-25]. The extent of these changes depends on the acid strength and time of treatment [17], [19], [26-28], as well as on temperature [29].

In our work we did two types of treatment to clay sample: A chemical treatment with sulfuric acid at various concentrations, a heat treatment under various temperatures. The choice of these modes of activation was determined by the results of a previous work [29-30], that predict an increased capacity of adsorbing bentonites. Which points to an increase in the adsorption capacity of the bentonite.

II. EXPERIMENTAL

A. Reagents

A stock solution containing 100 mg of Ni(II) per litter was

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prepared by dissolving $Ni(NO_3)_2.6H_2O$ in distilled water and was used to prepare the adsorbate solutions by appropriate dilution.

B. Clay adsorbents

Bentonite was obtained from Maghnia (ROUSSEL) Algiers. It was cleaned, dried and sieved into sizes of 100 µm.

C. Adsorbent characterization

XRD measurement

to «X'PERT PRO MPD» Philips X-ray spectrometer using Cu Kα radiations was used for characterizing the adsorbents.

FTIR measurement

Infrared spectrograms were recorded with a Perkin Elmer FT-IR using KBr pallets.

TGA measurement

Thermal analysis was carried out between 20 °C and 1100 °C using an apparatus: derivatographe.

Fluorescence spectroscopy

Fluorescence spectroscopy analysis was performed according to NF P 15-467 standard at CETIM, using the Philips Analytical device.

Specific surface area

Specific surface was calculated by using the method BET, in the field $0.02 \le P/P_o \le 0.35$. The volume of the micropores, the specific surface of the micropores and external surface, mainly the surface of the average pore was determined by the method t-plot. The difference between specific surface BET and external specific surface is indicated as being the specific surface of micropores.

Isotherms of adsorption of N_2 on bentonite samples were obtained on an apparatus TRISTAR 3000 (Micromeritics). Roughly 0.74 g of sample was degassed with 200°C during 24 hours under a high vacuum. An isotherm was then obtained at the temperature of liquid nitrogen (77.3K). The size of the pores is given starting from the phase of adsorption of the isotherm of adsorption of nitrogen (method BJH).

Cation exchange capacity

Method used to determine cation exchange capacity is SCHOLLENBERGER. This method consists in moving all the cations by ammonium a normal acetate solution (pH=7) from which excess is eliminated by a solution of ethanol with 95%. In the second percolation, salt used is the potassium chloride 1N.

D. Adsorption experiments

The adsorption experiments were made by batch technique at room temperature (25°C) with known amount of the adsorbent and 200 mL of metal solution at fixed concentrations of metal. The Erlenmeyer were kept under constant agitation (250 rpm). Afterward, samples were taken out from the shaker at regular contact time intervals, and the clay was separated by filtering. The concentration of remaining nickel in the solution, after shaking, was analysed by using atomic absorption spectrophotometer (AAS).

The adsorbed nickel amount (q_e) per unit absorbent mass was calculated as follows (Equation 1):

$$q_e = \frac{(C_0 - C_e)}{m}.V \qquad (1)$$

Where C_o is the initial Ni^{2+} concentration, C_e is the concentration of Ni^{2+} at equilibrium time (mg/L), m is the clay mass (mg) and V is the solution volume (L).

The following conditions were maintained for different sets of experiments:

Thermic activation: Clay 5 g/L, Ni(II) = 100 mg/L, pH =7.66, time = 6h, temperature = 100 to 300 °C;

Acid activation: Clay 5 g/L, Ni(II) =100 mg/L, pH =7.66, time = 6h, temperature = 98 °C, $[H_2SO_4]$= 3 to 36 % mass;

Kinetics: Clay 5 g/L, Ni(II) = 100 mg/L, temperature 289 K, pHi=7.66, interaction time: from 0 to 180 min;

Effects of pH: Clay 5 g/L, Ni(II) = 100 mg/L, temperature 289 K, interaction time180 min, pH 1.47 to 8.6;

Effect of adsorbate amount Clay 5 g/L, temperature 295 K, pHi=7.66, interaction time 180 min, Ni(II) = 10 to 300 mg/l.

III. RESULTS AND DISCUSSION

A. Adsorbent characterization

XRD and FT-IR study

XRD show intense peaks characteristic of montmorillonite, with the values (d = 4.46Å, 2θ =19.91°), (d =3.19Å, 2θ =27.77°) and (d= 3.76Å, 2θ = 23.60°).

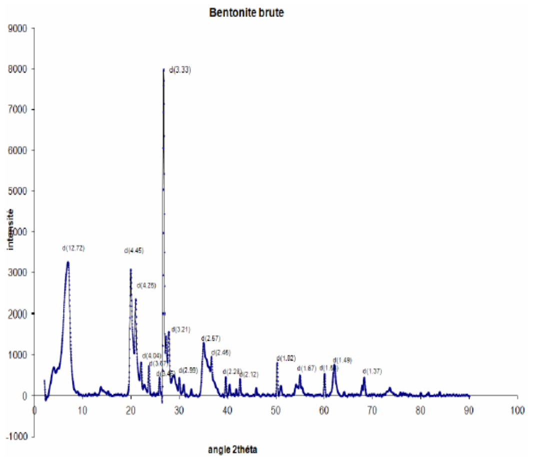

Fig. 1: XRD patterns for bentonite.

A FT-IR spectroscopic study for bentonite is shown in Fig. 2. As we can see there are absorption bands in the range of 3200-3800 cm -1, 1600-1700 cm-1.

The bands in the range of 1600-1700 cm^{-1} are due to lattice OH and bound water stretching vibrations. A strong and sharp band is detected at 1040 cm^{-1} which is related to Si–O stretching vibrations. The weak absorption bands at 528 and

478 cm^{-1} can be attributed to the Si-O-Al(VI) et Si-O-Mg(VI) bending vibrations. The band in the range of 3200-3800 cm^{-1}, with peaks at 3625 and 3440 cm^{-1} characterizing montmorillonite, corresponds to stretching vibrations of OH-.

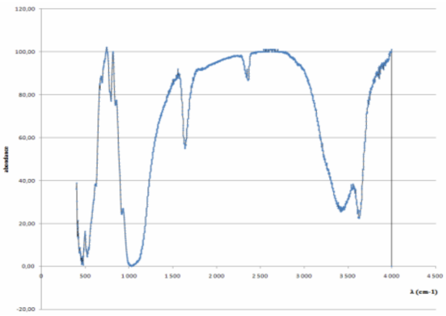

Fig.2: FT-IR (Fourier Transform Infrared spectrophotometer) Spectra of the natural bentonite.

TGA/ATD study

The interpretation of the DTA-TGA curves of bentonite leads to the following results:

- An endothermic peak at 100 0C, due to the removal of the hygroscopic water interlayer.
- An endothermic peak between 550 and 650 0C which corresponds to the loss of hydroxyl group from the clay mineral structure (clay dihydroxylation).
- An endothermic peak between 820 and 920 0C shows the formation of a phase at high temperature characteristic of aluminium ferifere smectite.

In addition to these peaks, other peaks appear in the DTA curve of the crude sample corresponding to the transformation of the quartz, at 650 0C.

Curve of TGA allow us to follow the weight loss of bentonite. These curves show three losses of weight:

•The first loss of weight occurs between 20 and 1800C. These transformations are due to the removal of adsorbed and interlayer water from the clay mineral.

•The second loss of weight occurs between 400 and 550 0C due the removal of water composition from the clay minerals. The third loss of weight between 600 and 700 0C. This loss can be attributed to the dihydroxylation of bentonite.

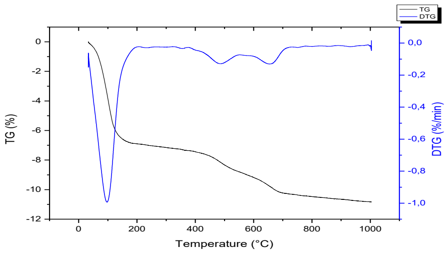

Fig. 3: DTA-TGA curve of bentonite.

Table. I

CONTENTS OF MAJOR ELEMENTS IN SAMPLE BENTONITE

Compounds	SiO_2	Al_2O_3	CaO	MgO	MnO
Content (%)	48.09	12.80	0.52	8.22	0.21
Compounds	Fe_2O_3	TiO_2	P_2O_5	Na_2O	K_2O
Content (%)	12.60	0.36	0.02	2.02	0.83

Fluorescence spectroscopy study

X-ray fluorescence (XRF) of sedimented fine (less than 2µm) Maghnia bentonite shows a significant percentage of silicon oxide (SiO_2) and aluminium oxide (Al_2O_3), and a low percentage K_2O (Table 1) which approves that it is a bentonite which belongs to the families of montmorillonite.

Surface area

We plot a graph Q_m the amount adsorbed as a function of relative pressure P/P0. The shape of the curve is shown in Fig. 4.

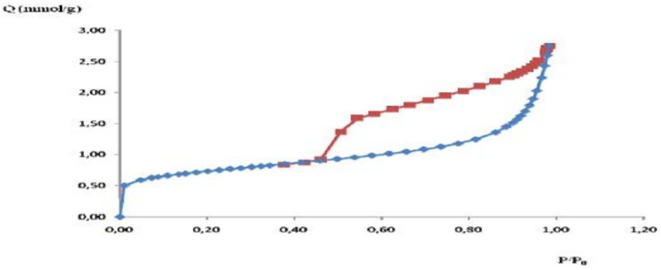

Fig. 4: Adsorption and desorption isotherms of nitrogen vapor at 77°K.

The nitrogen adsorption isotherms at 77°K for our clay (Fig. 4) are Type IV isotherms [31], the hysteresis effect on desorption being an indication of the presence of pores in the intermediate size range.

The results obtained show that our material is a mesoporous because we have a low microporous volume of $24.3*10^{-3}$ cm^3/g and a specific surface equal to 56.44 m^2/g.

Cation exchange capacity (CEC)

For the divalent cations, magnesium is the most dominating element (46.0 meq/100g). It is four times quantity of calcium (11.0 meq/100g). For monovalent cations, the most dominating element is sodium (36.7 meq/100g). Thus, the dominating replacement parts in the bentonite sample are Magnesium and Sodium.

Table.2

CATION CHARACTERISTICS AND AMOUNT OF VARIOUS CATION COMPOUNDS IN BENTONITE.

Cation	Sample meq/100g
Magnesium	46.0
Sodium	36.7
Calcium	11.0
Potassium	27.0
Exchangeable cation content (S)	120.7
Cation exchange capacity (T)	122.3

Capacity of total exchange (T) is higher than the sum of the exchangeable cations (S). The difference (T-S) is 1.4

meq/100g of dry product (table 2).

This difference (T-S) can be due maybe with:

- The used method (dynamic method),
- Salts employed whose dimensions of molecules are relatively high,
- The probable presence of other cations such as Cu^{++}, Fe^{++}, Mn^{++}, …etc.

B. Adsorption of Ni^{+2}

Thermic and acid activation

Fig. 5 shows that the activated bentonite in a temperature between 120 and 160°C is the one which represents the biggest capacity of exchange. This increase can be justified by the elimination of a part of the molecules of adsorbed water. This departure of water results the liberation of positions of exchange. The decreasing values of the activity of the clay, which we obtain afterward, would be possibly the direct consequence of the progressive destruction of the crystalline structure of the clay with the rise of temperature. When this material is heated at temperature higher than 160°C, it loses more and more its adsorption properties.

For the rest of our work, it will be economic to use a bentonite dried just at 105-1100C, because there is a small difference between the quantity of nickel fixed on bentonite at 105^0C ($Q_e = 18.44$ mg.g^{-1}) and that fixed on activated clay in a range of temperature 120-160^0C ($Q_e = 18.60$-18.65 mg.g^1). According to the Fig. 5, (18.44 mg.g-1) represents the maximum quantity of nickel adsorbed by natural bentonite. The Ni^{2+} removal efficiency of bentonite was approximately 92 %.

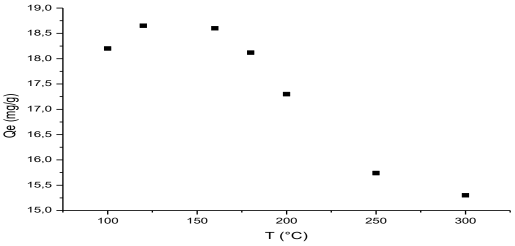

Fig. 5: Removal of Ni^{2+} by thermic activated bentonite at equilibrium.

According to researchers, the acid activation of the clay is due to the development of pores after uniform dissolution of the structure so that it has exactly one silicium atom for one of aluminium atom. When the solution is saturated with silicium, aluminium continues to dissolve and disappear from the network, causing the collapse. This explains the decrease in adsorption capacity of bentonite due to the rapid collapse of the structure after activation.

As we can see (Fig. 5 and Fig.6), the two types of activation of bentonite (chemical and thermic) have a negative impact on the ability of adsorption of our clay. That is why we continue our research with natural Bentonite just dried at 105-110 °C.

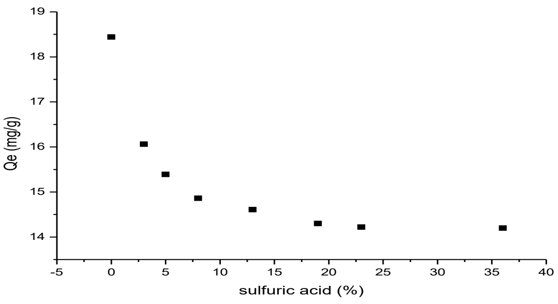

Fig. 6: Removal of Ni^{2+} by acid activated bentonite at equilibrium.

Effect of pH

Fig. 7 shows that adsorption of Ni(II) increase from 67.1% to 76.5% in the pH range of 1.47 to 6.31 under the experimental conditions.

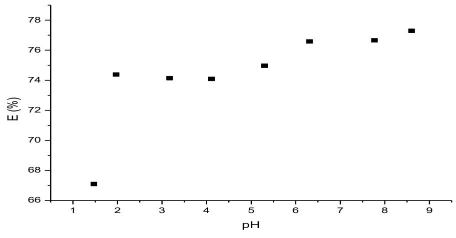

Fig. 7: Influence of pH on adsorption of Ni(II) on bentonite.

Then variation of adsorption remains constant until pH close to 9. At pH = 8.08, we have the rate of the highest elimination of nickel, (79.06%).

When pH is close to 7, the mechanism of adsorption by ionic exchange is supported. At these values of pH, exchangeable cations present on the sites of exchange (i.e. Na^+, K^+, Ca^{2+} et Mg^{2+}) are exchanged with the cations Ni(II) present in solution [32, 33].

If the solution is basic, adsorption by complexation of surface is favoured. The principal mechanism passes from the ionic exchange to the complexation of surface with the increase of pH. When pH increase, the dominant aqueous species of nickel changes into Ni $(OH)_2$, $Ni(OH)_{-3}$ et $Ni(OH)_4^{2-}$ with weak charge, these species have a weaker competitiveness for the ionic exchange [34, 35]. In general, the influence of pH is negligible because it does not improve much the yield. (An increase of 10%).

Effects of adsorbate amount

Fig. 8 shows that the amount adsorbed per unit mass, qe, increase with the initial concentration of Ni(II), up to 100mg/l quantity adsorbed remain constant. This may be explained by the fact that in the diluted solutions, the mobility of the ions nickel is high, so his interaction with the adsorbent increases.

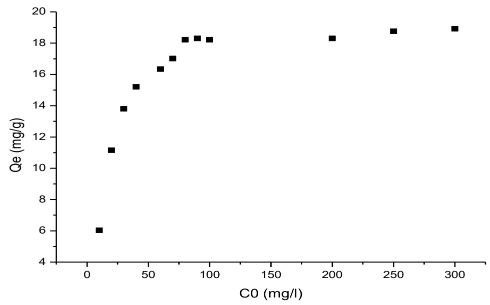

Fig. 8: Effects of Ni(II) concentration for adsorption on bentonite.

According to some authors [36, 37] at low initial metal ion concentration would have first the fixing of the cations on sites of strong chemical reactivity with side surface.

With an increase in Ni(II) concentration, a second site of lower chemical reactivity with stronger surface (side surface) would intervene whereas the sites of strong chemical reactivity would be almost saturated.

Kinetics of Ni(II) adsorption

The curve characteristic of the adsorption of nickel is respectively represented on Fig.9. This kinetics is characterized by a fast fixing; after only 3 minutes, the bentonite absorbed 80% of nickel. Beyond 140 min, the quantity of nickel fixed does not vary.

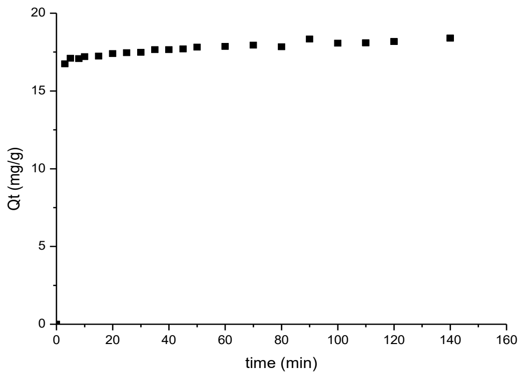

Fig. 9: Effects of interaction time on adsorption of Ni(II) on bentonite (clay 5/L, initial Ni(II) 100 mg/L,).

Different models have been used to test the kinetics of clay-Ni(II) interactions. The pseudo first order curves are obtained by plotting log [$q_e(q_e–q_t)$] vs. time. The first order rate constant (obtained from the slopes) equal to 2.53.10-2 min−1 (Table 3).

However, linearity of pseudo first order plots does not necessarily assure a first order mechanism. The large deviations have led to verifying the kinetics of the interactions further by using the second order equation.

Table. 3

RATE COEFFICIENTS FOR ADSORPTION OF NI(II) ON BENTONITE.

Pseudo first order		Second order	
k_1(min)	R^2	K_2(g/mg.min)	R^2
$2{,}53.10^{-2}$	0,677	$7{,}10^{-2}$	0,999

The kinetics of Ni(II) adsorption on clays is very close to second order kinetics.

Adsorption isotherm

The Langmuir plots based on equation 2 [38] :

$$\frac{C_e}{q_e} = \frac{1}{(b*q_m)} + \left(\frac{1}{q_m}\right) C_e* \qquad (2)$$

The Freundlich plots, based on equation 3 [39]:

$$q_e = K_F + {C_e}^{1/n}* \qquad (3)$$

The values of the adsorption coefficients obtained from the plots are given in Table 4.

It arises from the table 4 that it is the equation of Langmuir who models at best the contact solution nickel-clay chosen.

Table.4

FREUNDLICH AND LANGMUIR COEFFICIENTS FOR ADSORPTION OF NI(II) ON BENTONITE

Langmuir	Q_m(mg g^{-1})	19,61
	b (L^{g-1})	0,10
	R^2	1,00
Freundlich	K_F ($mg^{1-1/n}L^{1/n}\,g^{-1}$)	5,46
	n	4,03
	R^2	0,74

Thermodynamic parameters

Thermodynamic parameters, i.e. heat of adsorption, ΔH^0 and entropy change, ΔS^0, for the sorption of Ni(II) on bentonite were calculated for each system by using the following equations (Equation 5 and Equation 6)

$$lnK_d = (\Delta S/R) - \left(\frac{\Delta H}{R*T}\right) \qquad (5)$$

$$K_d = (Q_e/C_e) \qquad (6)$$

The values of ΔH^0 and ΔS^0 are obtained from the slope and the intercept of each plot. The free energy for the specific adsorption, ΔG^0, was calculated by using the equation 7.

$$\Delta G^0 = \Delta H^0 - T*\Delta S^0 \qquad (7)$$

In general, the phenomenon of adsorption is always accompanied by a thermal process [40, 41] which can be either exothermic ($\Delta H<0$) or endothermic ($\Delta H>0$). Measurement of adsorption heat is the main criterion for differentiating chemisorption from physisorption.

The values of the thermodynamic parameters for the sorption of Ni(II) on bentonite are given in Table 5.

The positive value of ΔH (Table 5) show that the sorption of nickel ions on bentonite is an endothermic process. The value of AG indicates the spontaneity of the process These results show that sorption of Ni(II) on bentonite is an endothermic process.

According to some authors [42, 43] the sorption reactions of the monovalent cations are exothermic, resulting in a sorption that tends to decrease with temperature. Conversely, the sorption reactions of bi and trivalent cations are endothermic, which therefore leads to an increase in retention when the temperature rises.

The positive value of ΔH confirms that the adsorption of nickel on the clay is an endothermic process. This value is less than 40 K Joule / mole. This indicates that it is a physical adsorption, even the negative value of the free enthalpy given in Table 5 shows that the process of adsorption of nickel on bentonite is a spontaneous process [44].

IV. CONCLUSION

The fixing of Ni(II) on Bentonite increases with pH in the interval considered $1.47 \leq pH \leq 8.60$. The maximum effect is reached for pH = 7. This clay fixes Ni(II) in a very short time (t=3 minutes, 80% of Ni(II) retained). Equilibrium is reached at time t < 10 minutes. This kinetics is very good because most of the factors are favourable (speed of agitation, temperature, ray of the particles, etc.…). This behaviour is described by a monolayer Langmuir type isotherm. The adsorption process is found to be a second order kinetics. These results show that bentonite can be used effectively for the removal of Ni(II). Considering the abundance and low price of bentonites and their physical and chemical characteristics, they are materials to be efficiently applied in the environmental industry. They can be used in the treatment of mining and/or industrial effluents with metallic contents above the standard values considered by the corresponding legislation.

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