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CHARACTERIZATION OF SLUDGE FROM THE TREATMENT OF DRINKING WATER AND THEIR VALUATION IN THE TREATMENT OF LEACHATE

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Abstract: The water purification plants generate hydroxide sludge. Most of this sludge is either dried or stored onsite, disposed of in landfill or discharged to various receiving environments without any prior treatment. The present work consists in the characterization of the sludge resulting from the treatment of water purification in the perspective of their environmental valorization especially in the field of decontamination of the leachates of the public dumps. The physico-chemical, mineralogical and microscopic analyzes carried out on sludge from water purification plants revealed that these sludges are silico-aluminous clays, consisting of a heterogeneous mixture of particles of irregular shapes in sheet and granular compound minerals of rounded forms, and very rich in silica, aluminum and iron. This sludge was used for the organic depollution of young and mature leachates from solid waste collection trucks and dump storage ponds, respectively. On the basis of the results of the tests carried out, it turns out that the characteristics of the sludges in question give them the properties of a good adsorbent for the organic matter with a yield of 58 % reduction for the young leachate under the conditions of *p*H = 6, a contact time of 40 min and a sludge mass of 4 $g \cdot L^{-1}$. An 86.25 % reduction yield for mature leachates under *p*H = 6 conditions, a contact time of 30 min and a sludge mass of 2 $g \cdot L^{-1}$.

Keywords: *drinking water, hydroxide sludge, leachate, organic matter, valorization*

INTRODUCTION

Coagulation is one of the most important steps in the treatment of surface water. 90% of drinking water plants use this process [1]. It consists of the addition of reagents to cause agglomeration of the suspended particles into flocculent aggregates, the whole of which forms a mass called "*floc*", the set of flocs constitutes the sludge. The reagents used are generally salts of iron or aluminum [2].

The majority of the sludge is either put to dry and stored at the treatment stations, landfilled, or applied to the irrigation water systems without any prior treatment. Several studies have shown that sludge has a negative impact on the soil because of its *p*H and its richness of metals that accumulate in the soil. The iron and aluminum hydroxides of the sludge cause the flocculation of the soil particles, they are considered as cementing agents of the soil [3].

The composition of the hydroxide sludge produced by drinking water treatment plants differs from that of biological sludge [4]. The quality and quantity of the sludge depend mainly on the quality of the water, the type of treatment and the reagents used during the potabilization process [5]. Drinking water sludge is a problem for drinking water treatment plants and its valuation is paramount.

The valuation of this sludge should go through a complete characterization of this waste. In the present work we carried out X-ray fluorescence, X-ray diffraction, infrared spectroscopy and transmission electron microscopy (TEM) analysis for sludge from the Ain Asserdoune station (Beni Mellal).

Solid waste management in Morocco and its by-products, such as leachate and gas, are among the main concerns. The leachate composition depends on several parameters, including the type of waste, *p*H, redox potential, the nature and age of the landfill, the landfill method, the nature of the site, and the climatic conditions (rainfall, air humidity and temperature) [6 – 8]. The leachate carries several types of pollutants, including heavy metals, chlorides and nitrates [9, 10]. The impact of this release on surface water and groundwater has been the subject of several studies [11 – 14] which have concluded that leachate degrades the quality of groundwater by nitric pollution and chlorides, this contamination is by two ways, a direct pathway through infiltration of leachate into the subsoil and an indirect one through surface water runoff [12].

Due to the increasing requirements of the discharge standards, several leachate treatment techniques are used. Anouzla (2014) reported that the coagulation of leachate by an Iron-rich industrial discharge gave a good yield in the elimination of COD (56 %) and BOD_5 (72 %) [15]. However, Ezzoubi (2010) reported that coagulation of leachate by ferric chloride allowed the reduction of 20 % as the minimum value and 35 % as the maximum COD value and an average BOD_5 abatement rate of 29.17 %. This indicates an elimination of organic compounds mainly by adsorption on ferric chlorides [16, 17], other researchers have been working on the treatment of leachate by nanofiltration membranes with a 70 % reduction in COD [18]. These processes are effective but costly.

The objective of this work is to treat the organic matter contained in the mature leachate by the raw hydroxide sludge from drinking water treatment.

MATERIAL AND METHODS

The supply of drinking water to the town of Beni Mellal is provided by the station of Ain Asserdoune and the station of Bin El Ouidane located in Afourer as well as the waters of the drillings of Sidi Jaber.
The operation of the two drinking water treatment plants is accompanied by the production of a large quantity of hydroxide sludge, which is the subject of our work. The sludge from the Ain Asserdoune station is discharged directly into the irrigation canals.

Samples collection

The sludge samples are taken directly from the decanters of the Ain Asserdoune drinking water treatment plant. The liquid sludge is decanted and dried and then placed in an oven at a temperature of 105 °C.
Young leachate is recovered directly from solid waste collection trucks.
Mature leachate is taken from storage basins at the public dump in Médiouna (Casablanca).

Analysis of sludge and leachate

Chemical analyzes of sludge (*p*H, conductivity, suspended matter, dry volatile matter, phosphorus, nitrates, nitrites, ammonium ions and sulfates) are carried out according to Association Française de Normalisation - AFNOR methods.
The analysis of the oxide composition is carried out by X-ray fluorescence spectrometry using a dispersion spectrometer of wavelength type Axios.
The mineralogical composition is determined by X-ray diffraction (Diffractometer type Panalytical X'Pert PRO) and by infrared spectroscopy.
Microscopic observation of the sludge is carried out by transmission electron microscopy (TEM), detector type SUTW-Sapphire.
We used hydroxide sludge for the treatment of a young leachate recovered from urban waste collection trucks and a mature leachate from the Mediouna (Casablanca) landfill.
The characterization of the young and mature leachate was carried out according to standardized standards.

RESULTS AND DISCUSSION

Characterization of sludge

The present study concerns the characterization of the sludge coming from the Ain Asserdoune station fed by the waters of the source (karst system). The coagulant used for the treatment of this water is aluminum sulfate.

Physico-chemical analyzes
The results of the physicochemical analyzes are given in Table 1. The sludge is characterized by a *p*H close to neutrality (6.84). These *p*H values are close to those

found by other studies, they varied between 6.91 and 6.38 [19]. This *p*H can lead to the precipitation of heavy metals and their adsorption on suspended or sludge materials [20].

The volatile dry matter makes it possible to evaluate the quantity of the organic matter contained in the sludge [21]. The sludge of the Ain Asserdoune station is characterized by a value of 8 %.This value may be due to the water supply source of the station (karst source of Ain Asserdoune). This volatile dry matter value is less than 30 %, so the sludge is stable and has a mineral character [19].

The determination of suspended matter is a very important criterion for optimizing the handling and recovery of sludge. The sludge from the Ain Asserdoune station contains 132.15 $g \cdot L^{-1}$ of suspended matter, other researchers found low values (23.6 $g \cdot L^{-1}$) of suspended matter in the hydroxide sludge [19]. This difference in suspended matter values is due to the station's water supply source. The station of Ain Asserdoune is fed by the waters of a karstic system whose turbidity of the raw waters sometimes exceeds 1000 NTU. So the amount of sludge produced in the station is enormous.

The chemical composition of the sludge from the Beni Mellal water purification plant using X-ray fluorescence is given in Table 2. The aluminum value (12.1 %) is due to the use of alumina sulfates during water treatment operations and the nature of the clay sludge [22]. The aluminum content allows them to be used as a plasticizer for the manufacture of building materials [23].

The high silica content may be due to the nature of the lands traversed by the feed water (52.4 %). Silica can be used as a degreaser. The sludge is also rich in Iron, which makes it possible to consider the sludge as clay with medium oxide color content for brick making [23].

The analyzed sludge is rich in oxides of calcium and magnesium; this can be explained by the fact that the source of Ain Asserdoune crosses a karst.

The low concentration of heavy metals recorded indicates that the station's water supply is far from any anthropogenic pollution.

The sludge of the Ain Asserdoune station is composed mainly of oxides of silica, alumina, iron, calcium and magnesium. It is therefore silico-aluminous clay. These characteristics allow their valuations in the field of manufacture of building materials

Table 1. *Chemical characterization of the sludge*

Parameters	Values
Temperature [°C]	15.8
Conductivity [$\mu s \cdot cm^{-1}$]	6.84
*p*H	587
Suspended matter [$mg \cdot L^{-1}$]	132.2
Volatile dry matter [%]	8
Total organic carbon [$g \cdot kg^{-1}$]	18.2
Phosphorus [$mg \cdot L^{-1}$]	0.001
Ammonium ion [$mg \cdot L^{-1}$]	0.099
Nitrites [$mg \cdot L^{-1}$]	0.82
Nitrates [$mg \cdot L^{-1}$]	0.026

Table 2. *Analysis by fluorescence X of the sludge*

Element	Concentration [%]	Element	Concentration [%]
SiO_2	52.4	Cl	0.0786
Fire loss	14.9	ZrO_2	0.0428
Al_2O_3	12.1	BaO	0.036
CaO	9.79	I	0.0183
Fe_2O_3	3.07	ZnO	0.0164
MgO	4.88	SrO	0.0075
K_2O	1.06	Br	0.008
Na_2O	0.532	Rb	0.0052
TiO_2	0.521	Y_2O_3	0.0040
P_2O_5	0.206	Ac	0.003
MnO_2	0.125	Nb	<<
SO_3	0.115	Sum	100

Sludge mineralogy analysis by X-ray diffraction (DRX) and Infrared Spectroscopy Analysis (FTIR)

In order to determine the mineralogical composition of the sludge, X-ray analysis was carried out. The mineralogical composition of the sludge was determined by X-ray diffraction on dried powders at a temperature of 105 °C for 2 h. The analysis of the diffractograms (Figures 1 and 2) allows us to observe a strong presence of free silica in the form of quartz identified by its characteristic line (d = 3.34 Å, 2θ = 26.75°).

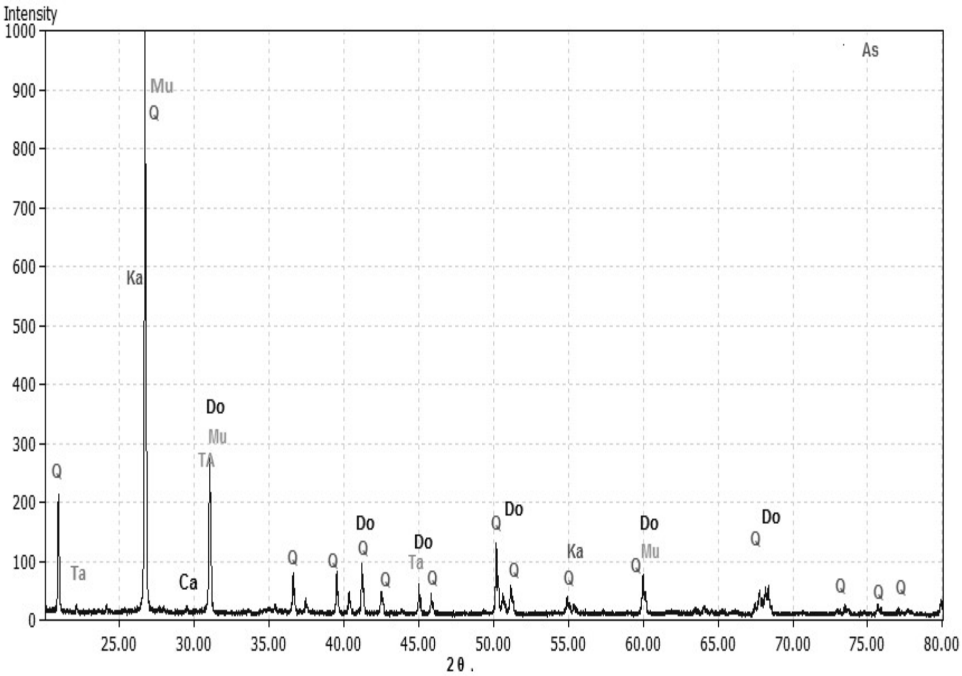

Figure 1. *DRX spectrum of the sludge*
(Mu: Muscovite, Ka: Kaolinite, Ta: Talc, D: Dolomite and Q: Quartz)

Concerning the phyllite phases, the analysis shows that the sludge presents kaolinite and muscovite therefore these sludge have a kaolinitic - Muscovite character. We also observed the characteristic line identifying the dolomite $CaMg(CO_3)_2$ (d = 2.88 Å, 2θ = 31.07°) and the Talc (d = 9.25 Å, 2θ = 9.55°).
The results of sludge analysis by X-ray diffraction are correlated with the chemical composition determined by X-ray fluorescence spectrometry.

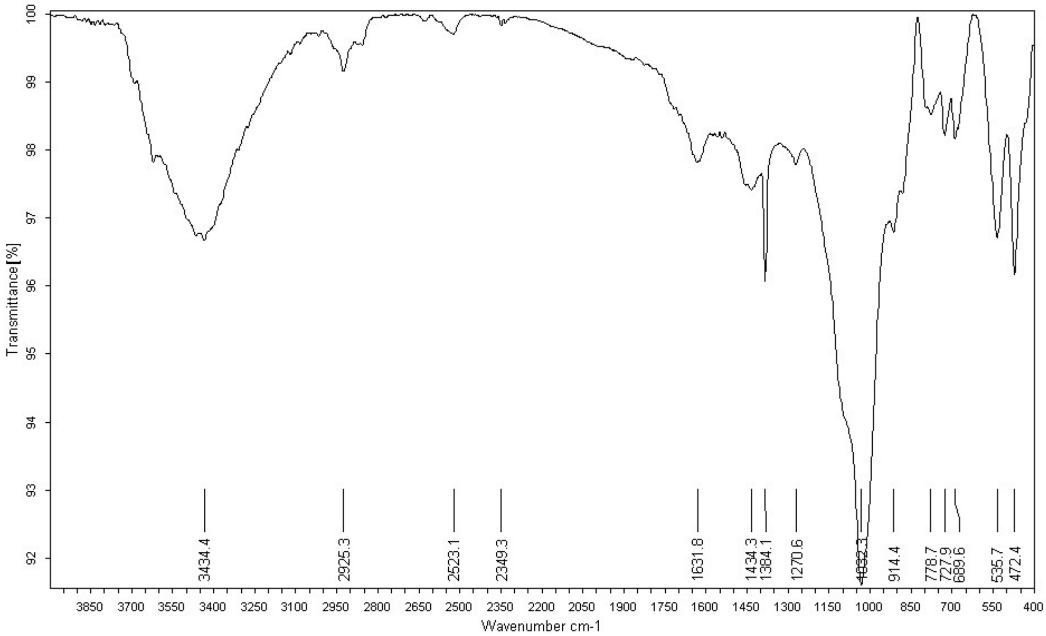

Figure 2. *Infrared spectrum of the sludge*

The results of the infrared spectra (Figure 2) are listed in Table 3.

Table 3. *Results of the infrared spectra of the sludge*

Radical	**Wave number (frequency) [cm^{-1}]**
O-H	3650 - 3600 - 3434.4 - 1631
CO_2^-	2650 - 2349.3
CO_3^{2-}	1434.4 - 1384.1 - 850
SiO_4	1270.6
SiO of kaolinite	1032.3
Al-O-H	914.4
Si-O of quartz	778.7 - 727.9 - 689.6 - 472.4
Si-O-Al	535.7

The results of infrared spectroscopy analysis confirm those of X-ray diffraction analysis.
The mineralogical composition of the sludge permits the substitution of natural clay in the manufacture of bricks. The silico-aluminous clay composition allows its use as an adsorbent.

Sludge microscopic analysis by TEM analysis

In order to complement the information obtained by the other methods of analysis on the sludge, a study was carried out by transmission electron microscopy (TEM). The photographs taken by TEM (Figure 3) show that the sludge constitutes a heterogeneous mixture of particles of irregular shapes in sheet and granular forms composed of minerals of rounded shapes. The analysis spectra of the sludge of the Ain Asserdoune station show that they are mainly composed of Al, Si, Fe, Cu and K (Figure 4).

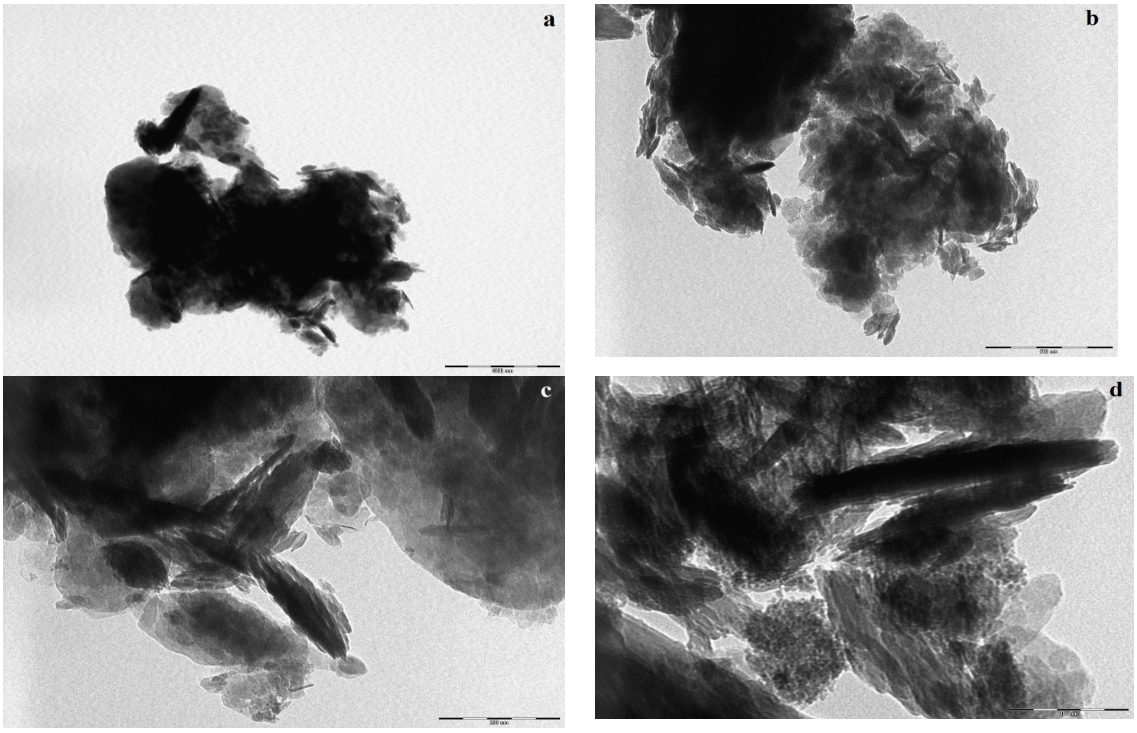

Figure 3. *TEM photos of the sludge (a). X 1000 (b). X500 (c). X200 and (d). X100*

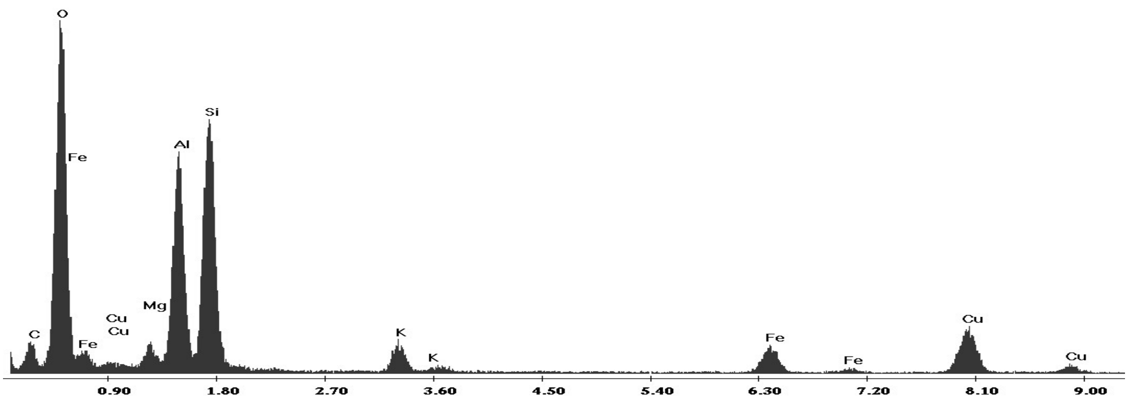

Figure 4. *TEM spectrum of the mineralogical composition of the sludge*

Characterization of the young and mature leachate

The physicochemical analysis of the young and mature leachate yielded the results listed in Table 4.

The leachate exhibits considerable variations in both flow rate and chemical composition [18] and it is therefore difficult to determine its properties with precision [5].

The studied young leachate is characterized by an acidic *p*H = 4.11, this value corresponds to the first fermentation phase; the acid phase or the leachate is rich in volatile organic compounds. The *p*H increases with the aging of leachate [1], which justifies the *p*H value found for the mature leachate of Mediouna (*p*H = 7.65). This increase is due to the consumption of acidic organic molecules, mainly volatile fatty acids (VFA) by aerobic microorganisms. Aeration of the leachate allows the release of CO_2 by the microbial activity. The produced CO_2 reacts with water to produce bicarbonate ion HCO_3^- which buffers the *p*H to neutrality (the denitrification observed during the test can also contribute to the *p*H increase by the consumption of protons) [24]. The *p*H has an effect on the mobility of metal ions because it influences the number of negative charges dissolved [18].

The leachate studied is characterized by high concentrations of COD (91200 $mg \cdot L^{-1}$) in excess of the Moroccan standard of direct discharges (500 mg $O_2 \cdot L$). This may be due to the nature of the waste generated and the age of leachate [5]. This effluent is also characterized by a very high BOD_5 (20000 $mg \cdot L^{-1}$). The mature leachate studied is highly charged with organic matter with a COD of 27000 $mg \cdot L^{-1}$ close to that of the El Kerma (Algeria) landfill and the Akaouedo landfill (Côte d'Ivoire) [6, 25] but different from other discharges [5, 7, 11, 17, 18, 25 – 28] (Table 2). This difference may be related to age, nature and quantity of waste, and climatic factors [6].

The BOD_5/COD ratio gives information on the biodegradability of leachate; it is 0.22 for the young leachate and 0.059. This ratio decreases with the age of leachate [29] which means that the biodegradability of the effluent decreases with age. The biodegradability of young leachate is low and that of mature leachate is even lower [30] since mature leachate is more stable than young leachate [31].

The analyses revealed high conductivity values indicating high mineralization due to very high levels of chloride (4970 $mg \cdot L^{-1}$), in phosphorus (4.13 $mg \cdot L^{-1}$), in nitrite (6.93 $mg \cdot L^{-1}$), in nitrates (126.2 $mg \cdot L^{-1}$) and ammonium ion (1064 $mg \cdot L^{-1}$). Conductivity increases in mature leachate, chloride values also increase in mature leachate; however the concentration of these ions does not depend on the degradation of leachate but rather on the leaching of the waste [32].

The dry matter value is 70450 $mg \cdot L^{-1}$ for the young leachate and 22741 $mg \cdot L^{-1}$ for the mature leachate, the dry volatile values for the young and mature leachate are 16320 and 704 $mg \cdot L^{-1}$. This shows the high inorganic and organic loading of young leachate compared to mature leachate [33].

The characterization of the sludge shows that they are silico-alumina clays very rich in silica, aluminum and iron; they can be used as adsorbents [34].

Table 4. *Physicochemical analysis of the young and mature leachate*

Parameters		Raw young leachate	Raw mature leachate	Discharge standard
Chemical oxygen demand (COD) [$mg \cdot L^{-1}$]	dissolved	63600	27000	1200
	total	91200		
Biological oxygen demand (BOD_5) [$mg \cdot L^{-1}$]	dissolved	7400	1600	500
	total	20000		
Nitrates [$mg \cdot L^{-1}$]	dissolved	100	22.61	
	total	126		
Nitrites [$mg \cdot L^{-1}$]	dissolved	0.9	Traces	
	total	6.9		
Ammonium ion [$mg \cdot L^{-1}$]	dissolved	781	1073	400
	total	1064		
*Ortho*phosphates [$mg \cdot L^{-1}$]	dissolved	3.12	9.43	
	total	4.13		
Total nitrogen [$mg \cdot L^{-1}$]	total	1672		
Dry matter [$mg \cdot L^{-1}$]		70450	22741	
Dry volatile matter [$mg \cdot L^{-1}$]		16320	704	
Total phosphorus [$mg \cdot L^{-1}$]	dissolved	3.2		
	total	4.8	20.88	
*p*H		4.11	7.65	5.5 - 8.5
Conductivity [$mS \cdot cm^{-1}$]		5.11	25.6	
Chloride [$mg \cdot L^{-1}$]		4970	7445	

Valorization of hydroxide sludge in the treatment of leachate

Effect of mass

In a series of agitated reactors containing raw leachate we have introduced variable masses of dry and raw hydroxide sludge. We evaluated the purification capacity of the sludge by monitoring the chemical oxygen demand.

COD monitoring of treated effluents showed a gradual decrease as the amount of dry sludge introduced was increased to an optimal dose of 4 $g \cdot L^{-1}$ with a COD reduction of 57600 $mg \cdot L^{-1}$ and a yield of 36.8 % for the young leachate (Figure 5 (a)). The optimum mass for the treatment of mature leachate is 2 $g \cdot L^{-1}$ with a COD reduction of up to 10000 $mg \cdot L^{-1}$, resulting in a removal efficiency of 62.97 % (Figure 5 (b)). The reduction of COD is explained by the insertion of the organic molecules between the sheets of silico-aluminous clay, the effectiveness of these sludges depends on their mineralogy. They are rich in silica (good adsorbent), and also contain significant quantities of aluminum and iron, which makes it possible to neutralize the negative charges of the organic matter contained in the leachate. It is noted that the optimal dose for the treatment of mature leachate is lower than the optimal dose for the treatment of young leachate, this may be due to the age and the pollutant loading of each leachate, the COD of the young leachate is 91200 $mg \cdot L^{-1}$ compared to the COD of the mature leachate, which is equal to 27000 $mg \cdot L^{-1}$.

The adsorption can be influenced by several parameters, namely *p*H and contact time.

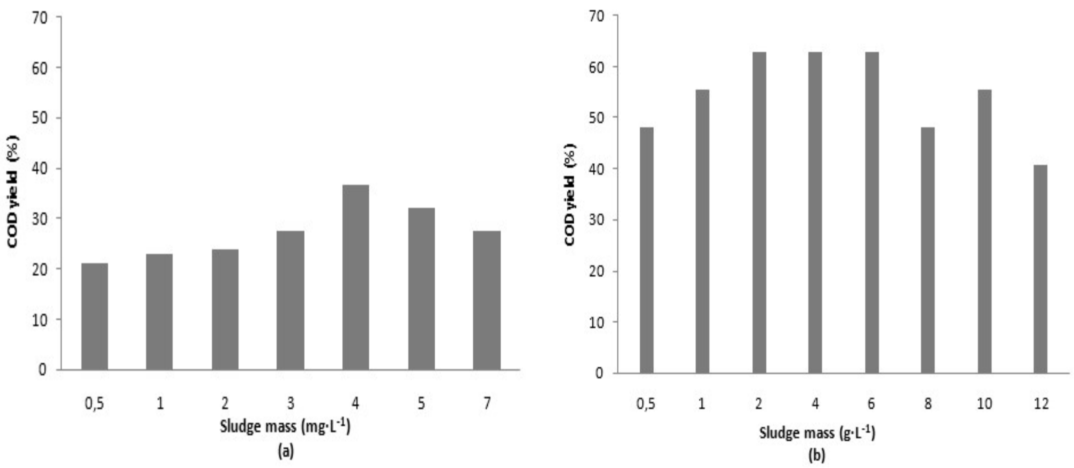

Figure 5. *Effect of raw hydroxide sludge mass on young (a) and mature (b) leachate treatment*

Effect of pH

The *p*H has a fundamental parameter in the course of the sludge adsorption reaction as it influences the surface charge of the supports [35]. In order to demonstrate the effect of this parameter, leachate adsorption tests were carried out in the presence of a 4 $g\cdot L^{-1}$ mud dose for the young leachate (Figure 6 (a)) and 2 $g\cdot L^{-1}$ for the mature leachate (Figure 6 (b)). The *p*H is adjusted in a range of 4 to 10.

The best yields are obtained for *p*H values of 6 and 10 for both leachates; there is a partial neutralization of the organic matter loads by adsorption on the sludge at *p*H 5 and 6, which favors the removal of the organic matter.

In the *p*H range 6 - 8, humic substances are adsorbed to the surface of the flocs according to two main mechanisms: one of an electrostatic type between the positively charged floc and the humic acids, the other by exchange of hydroxide floc with humate ions [36].

The *p*H influences the adsorption capacity of the sludge by modifying the surface charge distribution [37]. A study shows that for an adsorbent rich in aluminum oxide and Iron the maximum adsorption is reached at *p*H between 3.5 and 6 [38].

Effect of contact time

Contact time is an essential parameter in the optimization of adsorption treatment. It is noted that the contact time increases until a time of equilibrium which depends on the nature of the leachate; 40 min for the young leachate with a yield of 58 % COD (Figure 7 (a)) and 30 min for the mature leachate with a yield of 86.25 % (Figure 7 (b)). Beyond these times, we observe a phenomenon of desorption of the organic matter by the mud [39].

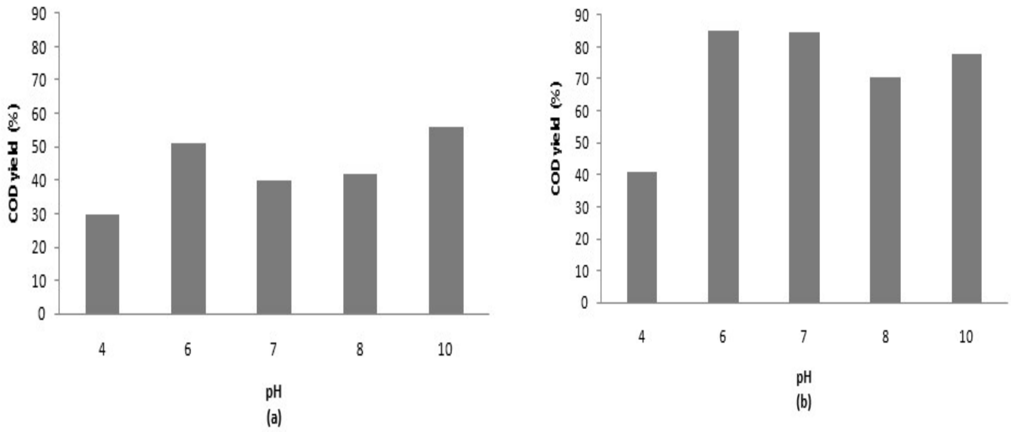

Figure 6. *pH effect on the treatment of young (a) and mature (b) leachate by raw hydroxide sludge*

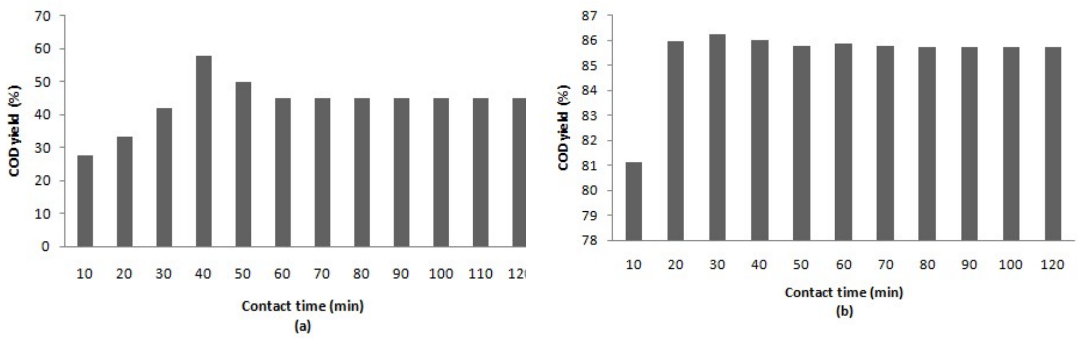

Figure 7. *Effect of contact time on the treatment of young (a) and mature (b) leachate by the raw hydroxide sludge*

CONCLUSION

The sludge characterization of the water purification plant of Ain Asserdoune in Beni Mellal shows that this sludge is very rich in silica, aluminum and iron; the same composition is approved by mineralogical X-ray diffraction analysis. The hydroxide sludge of the Ain Asserdoune station is composed of silico-aluminous clays. With a view to their environmental valorization and in particular in terms of decontamination of leachate from public landfills, this sludge is applied to the adsorption of the organic matter of the young and mature leachates from the Casablanca solid waste and the Médiouna landfill respectively. These leachates are highly charged with organic matter and have high pollution values (nitrogen, phosphorus and chlorides).
The sludge has been shown to be highly effective in reducing leachate organic pollution with yield reductions of 58 % for young leachate and 86.25 % for mature leachate.

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