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Characterization of the sludge of Batna (Algeria) waste water treatment plant for its valorization

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Abstract
The treated waste waters of the city of Batna (Algeria) hold for about half of the needs of the land farmers in water in this area. The treatment of these waters generates a sludge which is rich in nitrogenous and phosphorous compounds and in organic matters. The purpose of this study is the characterization of this sludge in order to use it as a fertilizer. The X-ray fluorescence analysis allowed detecting the following element in this sludge : Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Sr, Y, Zr, Mo, Cd, Ba, Pb. The mass of the previous elements hold 38.52% of the dried sludge total mass. In this sludge, the X-ray diffraction analysis showed the existence of the following phases: $(Mg_{0.03}Ca_{0.97})CO_3$, SiO_2, *ZnO*, Cu_2O *and* $BaPbO_3$. *The trace metals which are present in Batna waste water treatment plant, and the content of which is limited, are the following: Cd, Cr, Cu, Ni, Pb and Zn. Their contents are 40 mg/Kg, 240 mg/Kg, 70 mg/Kg, 830 mg/Kg and 3890 mg/Kg, respectively. An extraction in aqueous solution was used for determining the amount of dissolved ions nitrate, chemical oxygen demand (COD) and pH. The results obtained are 530 mg/Kg, 850 mg/Kg and 7.28, respectively.*
Keywords: Batna, sludge, element trace, landspreading, sewage treatment plant.

1. Introduction

The climate of Northern Algeria is semi-arid. It is characterized by a strong rainfall irregularity. The water resources are insufficient and their exploitation has been intensified by the development of the Country [1-3]. Therefore the agricultural demand can be complied with only by using the storage of regular water [4] especially for the west of Algeria [5]. The search for other sources of water as the re-use of the treated waste waters as a supplement in irrigation is the ultimate solution [6]. The total amount of the used waste waters collected in Algeria is at the rate of 1.2 billion m^3/year [3]. Nearly 170 waste water treatment plants (most of which are mechanized) are used for treating 35% of this amount.

The city of Batna runs a mechanical and biological water treatment plant by activate sludge. It is designed for treating 28 000 m^3 (230 000 EH) of liquid discharge per day. The treated waste waters stand for approximately half the needs of the farmers in water in this area. The treatment of the waste waters generates a sludge rich in nitrogenous and phosphorous compounds and in organic matters. Therefore they are used as fertilizers. This sludge may consist of pathogenic microbes [7-11] and potentially dangerous chemical contaminants for human health and for the environment. Trace metals stand for the largest part of these compounds [12]. Above a certain concentration, the sludge from the water treatment plants is not recoverable anymore in agriculture. The organic matters contained in the sludge buried in the soil can be degraded [13]. The use of sludge contaminated by trace metals impairs the biodegradation of some organic compounds [14]. These trace metals are trapped in the sludge, dragged by lixiviation [15] or adsorbed by the plants [16, 17]. This adsorption incorporates them into the food chains. A very high concentration in trace metals as Pb, Zn and Cd prevents revegetation [18]. The continuous production of sludge in contaminated filtering plants will eventually end in landfills, incineration or composting. The cost of the disposal of the sludge from a filtering plant can reach 60% of the operation charge [19, 20].

The characterization of the sludge from the filtering plant is imperative before its valorization in agriculture. Many techniques can be used for the soil analysis. The obtained results depend on the technique used [21]. In our study, we used X-ray fluorescence and X-ray diffraction on powders for the metal analysis of the sludge from Batna waste water treatment plant. Carbon and nitrogen were chemically dosed after their extraction in aqueous solution.

2. Materials and methods

The sludge samples were collected in the drying beds of Batna waste water treatment plant. They were dried at 60°C. The entirely dried samples were crushed small enough to guarantee a perfect homogeneity. This powder was used directly in the X-ray diffraction analysis. A pad of 30 mm diameter and 10 gram weight was realized under a 10-ton pressure to be used for the X-ray fluorescence analysis.

The X-ray fluorescence analysis was performed by a 9-watt power Panalytical Epsilon 3 spectrometer. The X-ray fluorescence range was recorded four times under different excitation conditions. The first range was performed with a 5.00-kV ddp and a 1000-µA current. It allows exploring the energy zone up to about 4 keV. The second range was performed with a silver filter with 100 µm thickness, a 30.00 kV ddp, and a 300 µA current. It shows high energy peaks. The peaks are highly mitigated under such conditions. The first range was recorded with a 50 µm thick aluminum filter with a 12.00 kV ddp and a 500 µA current. The fourth range was recorded with a 200 µm thick aluminum filter, 20.00 kV ddp and 450 µA current. The use of an aluminum filter allows exploring the spectrum zone ranging between 1 and 20 keV.

The diffractogram was achieved with an X'pert-PRO panalytical powder diffractometer. The data recording was carried out within an angle range from 5° to 120° with a 0.013° tread for a 560 s step time. The voltage and current of the tube were 45 kV and 40 mA, respectively. The device uses a 0.154056 nm wavelength radiation with a tube using a copper-made (Cu) anode. The diffraction data were analyzed with a PANalytical X'Pert HighScore Plus software program. The crystalline phases were determined compared to registered models of the PDF2 database.

The analysis of carbon and nitrogen by X-ray fluorescence is challenging. We measured their concentrations chemically after an extraction in aqueous solution. The nitric nitrogen was dosed by visible UV spectrometry, carbon by COD and acidity by pH-meter.

The extraction was performed by agitating 10 gram sludge in 100 ml UHQ water for an hour. The solution obtained after centrifugation served for determining the chemical oxygen demand (COD), ions nitrate concentration and pH.

The COD determination was carried out by oxidation of the organic matter in sulfochromic medium at 148°C. The decrease in the oxidant concentration (potassium dichromate) is proportional to the amount of organic matter contained in the sample. Such decrease is determined by spectrometric quantification at 345 nm of the initial and final concentration of the oxidant. It is proportional to the amount of oxygen necessary for oxidation of the organic matter present in the sample. The COD-meter is a Hach model 45600 brand. The spectrometer is a Hach DF/2000 brand.

The concentration in ions nitrate was determined after reaction with sodium salicylate. This reaction allows obtaining sodium paranitro-salicylate, yellow-colored and likely to have a spectrometric 420 nm measurement. The measurement of optic density was performed by JENWAY brand visible UV spectrometer.

The measurement of pH was performed with a JENWAY 3505 brand pH-meter.

3. Results

The X-ray fluorescence spectra under different conditions are presented in figure 1. The position of the peaks informs us on the metallic composition. The interpretation of the spectra indicates the presence in the analyzed sludge the following elements : Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Sr, Y, Zr, Mo, Cd, Ba, Pb. The total mass of the previous elements holds 38.52 % of the total mass of the dried sludge.

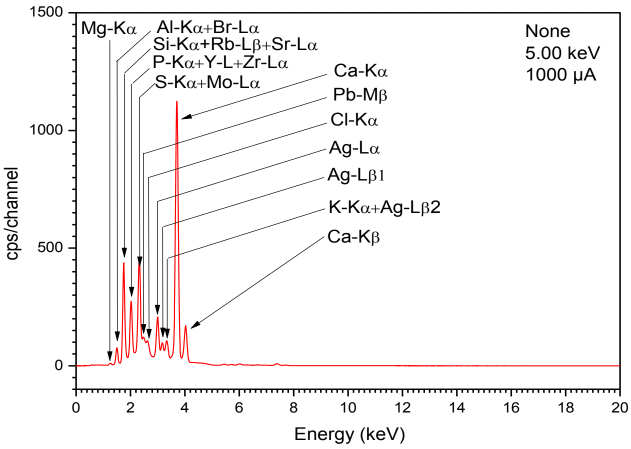

***Figure 1a.** XRF spectrum of sludge of Batna waste water treatment plant achieved without a filter, 5.00 kV, 1000 µA*

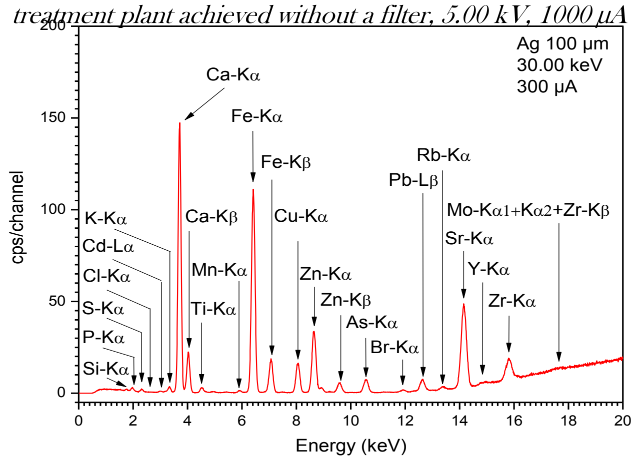

***Figure 1b.** XRF spectrum of sludge of Batna waste water treatment plant achieved with a silver filter of 100 µm, 30.00 kV, 300 µA*

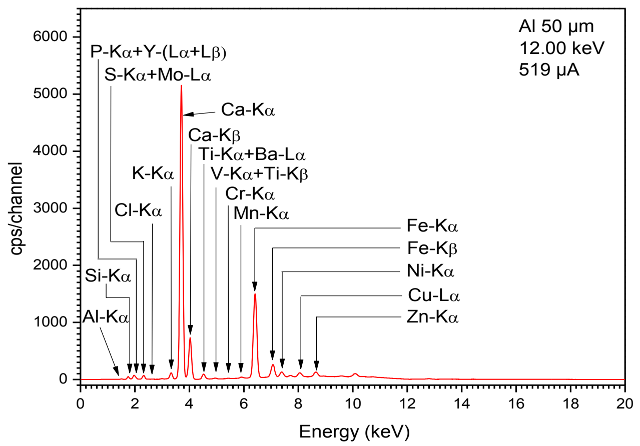

Figure 1c. *XRF spectrum of sludge of Batna waste water treatment plant achieved with an aluminum filter of 50 µm thickness, 12.00 kV, 519 µA*

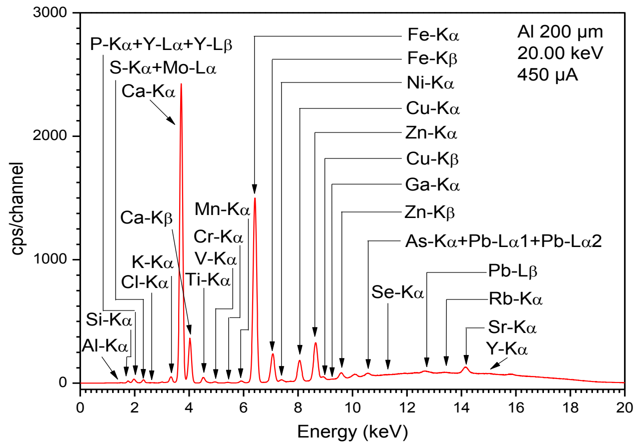

Figure 1d. *XRF spectrum of sludge of Batna waste water treatment plant achieved with an aluminum filter of 200 µm thickness, 20.00 kV, 450 µA*

The area of the X-ray fluorescence spectrum peak is proportional to the concentration of the element factor of the peak. The exploitation of this feature allowed us to quantify the concentration of the polluting elements in the sludge; table 1.

Table 1. *Trace elements present in the sludge from Batna waste water treatment plant and the quality limit of the sludge admitted in agricultural spreading ant the flow speed limits over 10 years of trace metals brought by the sludge (*French regulations; bylaw 08/01/98)

Compound	Cd	Cr	Cu	Hg	Ni	Pb	Zn	Cr+Cu+Ni+Zn
Concentration (mg/Kg MS)	40	240	2740	0	70	830	3890	2520
Content limit (mg/kg MS)	10	1000	1000	10	200	800	3000	4000
Limit flow limit (kg/ha/10 years)	0.15	15	15	0.15	3	15	45	

The X-ray diffraction analysis of the spectra (figure 2) of the sludge from Batna water treatment plant showed the existence of the following phases: magnesium calcium carbonate: $(Mg_{0.03}Ca_{0.97})CO_3$ (PDF number 01-089-1304), silicone oxide SiO_2 (PDF number 01-085-1054), zinc oxide ZnO (PDF number 03-065-0523), copper oxide Cu_2O (PDF number 00-001-1142), barium lead oxide $BaPbO_3$ (PDF number 01-081-0904).

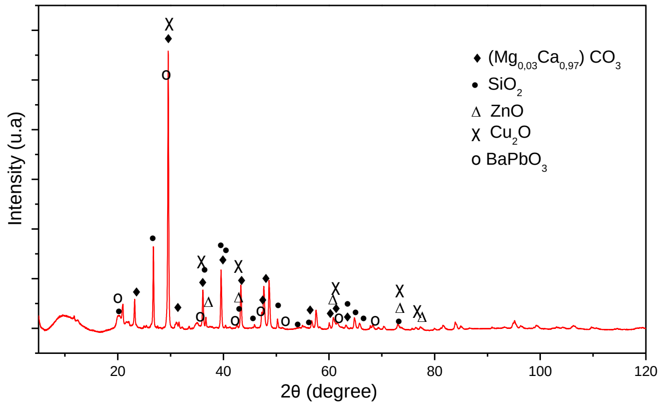

Figure 2. *XRD spectrum of the sludge from Batna waste water treatment plant*

The measurement of the concentration of dissolved ions nitrate gave us a value of 0.53 mg/g (0.53 Kg/ton)

The measurement of the COD in the sludge delivered a value of 0.85 mg/g (0.85 Kg/ton).

The measured pH of the sludge from Batna waste water treatment plant is 7.28.

The measured pH of the sludge from Batna waste water treatment plant is innocuous during its spreading. The concentration values of dissolved ions nitrate and that of the COD are very low. Therefore several hundreds of tons per hectare must be spread to cause a risk for the environment.

The X-ray fluorescence analysis showed the presence of a large number of metals in the sludge from Batna treatment plant. This large number of metals does not necessarily imply pollution. Certain trace metals in very low concentrations as iron, zinc, magnesium, copper, sodium, potassium, boron, molybdenum are indispensable to cellular biology [22]. The biological treatment cannot be done without them. These metals are mainly found in the drinking water [23] and the urban dust but at very small concentrations [24, 25].

The presence of cadmium, chromium, copper, mercury, nickel, lead and zinc at high concentrations, is on the other hand, synonymous with pollution. It is the consequence of their presence in the urban effluents [26]. The impact of the treatment was to make them concentrate in the sludge. The sludge contaminated by a high concentration of these elements cannot be spread over. The regulations also limit the flow of these elements over ten years; table 1. The concentrations of copper, lead and zinc present in the sludge from Batna waste water treatment go beyond the norms. This sludge must be subjected to treatment before being spread [27]. Without an adequate treatment, the sludge from the water treatment of Batna must be incinerated or stored. The origin of pollution must be quickly determined in order for the future sludge to be valorized by agricultural spreading.

The origin of sludge pollution of Batna water treatment plant can be natural or entropic. The soil of the Batna region is known for its richness in copper, lead and zinc. This can account partly for the richness of the sludge of the Batna treatment plant in these metals. The automobile traffic is another major source of lead [28]. Lead tetraethyl is still used in Algeria as an anti-knock additive in gasoline-propelled car engines. The copper contained in the piping can be dragged by the water. This segment of the copper is in relation to the stagnation time in the water of the pipes, the dissolved oxygen concentration, pH, hardness and water temperature in the water. Cotitex-Batna textile industry uses chromium, copper, nickel, lead and zinc. This industry can be partly the cause of pollution of the waste waters of the city of Batna. [29].

A part of the pollutants trapped in the sludge is only mixed to it. The remaining part of these pollutants was processed by the bacteria [30]. The part which is not digested by bacteria is amorphous or crystalline. The one which is found in crystalline form can be characterized by XRD (X-ray diffraction). This identification of the phases of the pollutants can help track back the origins and propose techniques of treatment. The sludge of Batna waste water treatment plant contains the following crystals: magnesium calcium carbonate, silicon oxide, zinc oxide, copper oxide and barium lead oxide. These crystals can probably be collected by simple cleansing of the sludge.

4. Conclusion

The measured pH of the sludge from Batna waste water treatment is 7.28. It is harmless at land spreading. The concentration values of the dissolved ions nitrate and that of the COD are 0.53 mg/g and 0.85 mg/g, respectively. They are very low and show no risk for the Environment. This sludge consists of a large number of metals. Some of these do not necessarily imply pollution. At low concentration as iron, zinc, magnesium, copper, sodium, potassium, boron, molybdenum... are indispensable to cellular biology. These metals come from drinking water, city dust, etc. The presence of cadmium, chromium, copper, mercury, nickel, lead and zinc at high concentrations on the other hand means pollution. This is the consequence of their presence in the urban effluents. The impact of the treatment is to make them concentrate in the sludge of the waste water treatment plant. The concentrations of copper, lead and zinc present in the sludge from Batna waste water treatment plant exceed the norms. This sludge must be subjected to a treatment before being spread over. The cause of the pollution of the Batna treatment plant sludge can be natural or entropical. The soil of the Batna region is known for its richness in copper, lead and zinc. This can partly account for the richness of the sludge of Batna waste water treatment plant in these metals. The automobile traffic is another major source of lead. The copper contained in the piping can be dragged by the water. Cotitex-Batna textile industry uses chromium, copper, nickel, lead and zinc. This industry can be responsible, in part, for the pollution of the waste waters of the city of Batna. The sludge of Batna waste water treatment plant contains the following crystals: magnesium calcium carbonate, silicone oxide, zinc oxide, copper oxide and barium lead oxide. These crystals can probably be collected by a simple sludge cleansing.

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