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ORIGINAL RESEARCH PAPER

CHARACTERIZATION AND THERMAL STUDY OF SEWAGE SLUDGE FROM THE MUNICIPAL WASTEWATER TREATMENT PLANT OF EL EULMA, ALGERIA

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Abstract: The continuous produce of sewage sludge generated by the treatment of wastewater in wastewater treatment plants poses a serious environmental problem. The characterization of sewage sludge before its reuse in safe way becomes very important. The aim of this study is the characterization of sludge and calcined sludge in different temperatures in the range of 200 °C - 1100 °C. Qualitative and quantitative analysis were achieved using X-ray diffraction (XRD) and X-ray fluorescence (XRF). Thermal behavior of sludge was studied using thermo gravimetric analysis (TGA), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). Loss in mass, carbonates rate, *p*H, chemical oxygen demand (COD) and nitrate contents are also measured. Results show that major mineral constituents in our sludge and calcined sludge are, Al_2O_3, SiO_2, $CaCO_3$ and Fe_2O_3. Thermal behavior of sludge indicates three important losses in mass, first with a value of 8.24 % in temperature lower than 250 °C, the second with a value of 43.79 % between 230 °C and 700 °C and above 700 °C until 1100 °C a loss of 13.77 %. *p*H measurements values of different calcined samples are in the range from 6.3 to 11.1. Measurement of COD and nitrates contents reveals the values 2474 $mg \cdot L^{-1}$ and 134 $mg \cdot L^{-1}$ respectively.

Keywords: *calcinations, sewage sludge, sludge valorization, traces elements, X-ray diffractions*

INTRODUCTION

The rapid population growth in the world increases the demand of water in domestic, agricultural and industrial sectors. The reuse of wastewater using wastewater treatment plants (WWTP) is one of the most effective solutions for finding other sources of water. The treatment of water in this kind of installations constitutes another major problem with the increase and continuously produces of sewage sludge. The safe disposal of sewage sludge generated from wastewater treatment plants become one of the major environmental problems [1 – 4]. According to the Algerian National Sanitation Office (Office National de l'Assainissement - ONA), 153 WWTP were installed destined for treating 10 million person-equivalent (PE), and about 21 million m^3 per month which imposes the search for promising solutions for the reuse of sludge. In general, sludge is characterized by a considerable variability of organic and inorganic fractionation and their percentage depends on the quality of the wastewater sources and treatment process. The application of sewage sludge in different areas such as, fertilizer in agriculture, production of bricks, ceramics and cements are the most common solutions for sludge disposal [5 – 8]. In our country, this sludge has so far been mainly used in agricultural spreading and landfill [9]. Agricultural valorization of sludge is one of the techniques most used in the world (60 %) [10], because of its high organic matter content [11], and it's beneficial to soil with macronutrients existing in the sludge which serve as good sources for plant nutrients [12, 13]. However, new routes of elimination of sludge include incineration, which is one of potentially beneficial techniques used in recent years because of its applicability in industrial sectors [14]. On the other hand the use of sludge as fertilizer or in industrial sectors must be carefully controlled because sewage sludge contains many toxic and dangerous contaminants like heavy metals, organic compounds, and pathogens [15], therefore the characterization of sludge before its application is very crucial as studying the toxicity rate can ensure correct and the safe way for reuse purposes.

In this work, we investigate the characterization of the mineral fraction in sewage sludge and sludge ash after calcinations at different temperatures from 200 °C to 1100 °C using XRD, XRF, and TGA/DSC analysis. Loss in mass, carbonates rate, *p*H, COD and nitrates contents are also measured.

MATERIALS AND METHODS

Sample collection and preparation

The sewage sludge analyzed in this study was collected from the municipal wastewater treatment plants of Bazer Sakhra - El Eulma, Setif, Algeria, localization of this WWTP is 36°05'18.0"N latitude 5°41'28.2"E longitude. This WWTP use an aerobic activated sludge process designed to treat 20000 m^3/day (about 230000 PE).

At first, our sludge was dried during 48 h in air at 80 °C. After that sludge was crushed in Retsch PM 100 planetary ball mills (Germany) for 3 hours with 200 rpm speed to obtain a uniform particle size. In a Nabertherm P330 (German) furnace, 10 samples of 25 g from the previously obtained powder were placed in alumina crucibles and calcined for 3 hours in different temperature from 200 °C to 1100 °C with step of 100 °C with heating rate of 5 °C/min [16].

Characterizations

The measurement of mass before and after calcinations allows us to determine the loss in mass. For XRF analysis, 10 g from each calcined sludge was pressed under 10 tones with 30 mm diameter in a pellet press Retsch PP25 (Germany), the XFR analysis were carried out using Panalytical Epsilon 3 spectrometer (Netherlands). For accurate analysis, XRF data of all samples was recorded under different excitation conditions of ddp and current, 5.00 kV and 1000 µA, 30.00 kV and 300 µA, 12.00 kV and 519 µA, 20.00 kV and 450 µA, respectively. For XRD analysis, 1 g from each calcined simple was analyzed using an X'pert-PRO Panalytical powder diffractometer (Netherlands), all spectra were recorded in the range from 5° to 80° with a step of 0.013° using copper (Cu) anode. Thermal behavior of sewage sludge was carried using TA SDT Q600 analyzer (Canada), with heating rate of 10°C/min from room temperature to 1200 °C. Carbonates rates were determined by measuring the amount of releasing CO_2 after reacting sludge in HCl medium. *p*H, COD and nitrates were determined as indicated in literature [1, 11]. In our case, 10 g of sewage sludge was stirred with 100 mL of ultra-high quality (UHQ) water for 60 min. The solution obtained after centrifugation was mixed in a sulfochromic medium at 148 °C to oxidize all the organic matter. The measurement of the amount of the residual oxidant using ultraviolet spectrophotometer allows us to determine the amount of oxygen necessary for the oxidation of the soluble organic matter containing in our sludge, this measurement was carried in a Hach DR/2000 at 345 nm. The determination of the nitrate concentration was measured also using UV-Vis spectrophotometer at 420 nm after reacting the nitrate containing in the previous centrifuged solution with sodium salicylate to obtain sodium paranitrosalicylate colored in yellow. The determination of *p*H was carried using a *p*H-meter JENWAY 3505 (England). All products used in our manipulations are from Sigma-Aldrich: H_2SO_4 (≥97 %), $K_2Cr_2O_7$ (99 %), $HgSO_4$ (≥98 %), Ag_2SO_4 (≥99 %), benzoic acid (≥99 %), HCl and sodium salicylate (≥99.5 %)

RESULTS AND DISCUSSIONS

Loss in mass

Figure 1 shows the mass loss of the sewage sludge during calcinations at different temperatures from 200 °C to 1100 °C.

Results show that there is a significant loss in mass of 38 % from room temperature to 300 °C, which can be explained by the evaporation of moisture, structural water, biodegradable matter and semi-volatile compounds [17 – 19]. From 300 °C to 600 °C, we observe that the mass loss was 25 %, this loss is also significant, but with lower rate, this loss is most probably attributed to the decomposition of non-biodegradable organic matter, dead bacteria and organic polymers [20]. The maximum loss of 64 % was at 800 °C due to the decomposition of all organic matter and the transformation of carbonates into oxides with releases of CO_2 [21].

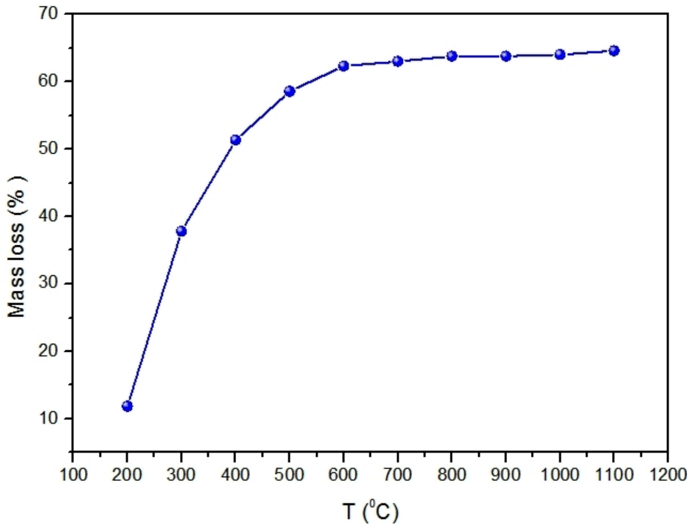

Figure 1. *Evolutions of mass loss in sludge with temperatures*

Carbonates rate

Figure 2 represents the carbonates rates in calcined sewage sludge from room temperature to 1100 °C.

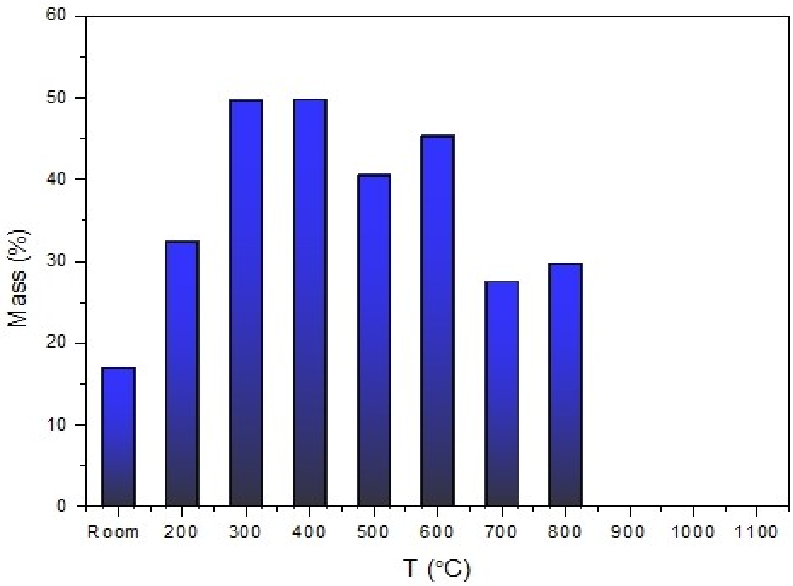

Figure 2. *Evolution of carbonates percentages with temperature*

The initial process from room temperature to 400 °C started with a significant increase in the rate of carbonates from 17 % to 49.8 %, this increase can be explained by the evaporation of the water existing in sludge and the degradation of the organic matter [22]. However, the carbonates level decreases above 400 °C to 800 °C due to the beginning of the decomposition of $CaCO_3$ into CaO with the release of CO_2, the increase of percentage of carbonates at 600 °C is due to the evaporation of all organic matter, in this temperature the mass loss of the sludge was 63 % which concentrates the mineral fraction in the sludge. The last interval of temperature from 900 °C to 1100 °C we notice the extinction of all carbonates due to the total transformation of metallic carbonates into oxides [23].

XRF analysis

XRF analysis (Figure 3) shows the evolution of the percentage of the elements present in sludge and sludge ash after calcinations in different temperatures.

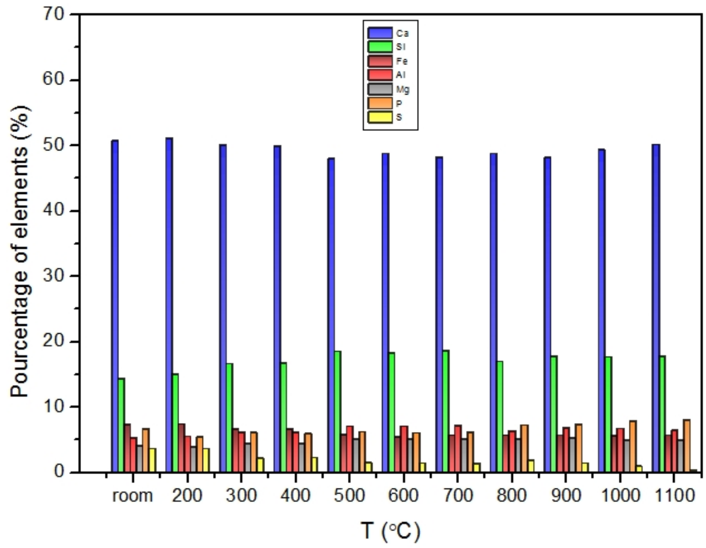

Figure 3. *Evolution of the amount of elements present in sludge and sludge ash with temperature*

According to previous results [24 – 26], the presence of some elements with important percentage such as Ca, Si, Fe, Al and Mg is absolutely normal because treated water carries a significant quantity of clays, and also the employ of ferric salts during treatment of water [7, 27]. The presence of phosphates is mainly due to the use of phosphorus in agriculture and domestic detergents [28, 29]. Sulfur also is present in significant amount in raw sludge; sources of sulfur species in sewage sludge are divided in two major categories: organic sulfur such as sulfone, sulfoxide, thiophene and mercaptan, and inorganic form such as sulfate and sulfite [30]. Results show that there is no significant change in the rate of metals in sludge from room temperature to 1100 °C, this result indicating that the most part of metals are in mineral or inorganic form. The amount of certain trace elements in WWTP of Bazer Sakhera - El Eulma and their comparison with the Algerian regulations NA 17671 [31] (adapted from French regulations NF U44-0411985) are shown in Table 1, results indicated the presence of Cr, Cu, and Zn with high concentrations that exceed regulations limits which make this sludge toxic for the use as fertilizer in agriculture.

Table 1. *Concentrations of trace elements and limit values admitted in agricultural spreading (NA 17671) in the sludge of wastewater treatment plant of Bazer Sakhera - El Eulma*

Elements	Limit values [mg·kg^{-1} DM*]	Concentrations values in sludge of Bazer Sakhera [mg·kg^{-1} DM*]	Maximum cumulated in 10 years [g·m^{-2}]
Cd	20	-	0.03
Cr	1000	3150	1.5
Cu	1000	1620	1.5
Hg	10	-	0.015
Ni	200	80	0.3
Pb	800	730	1.5
Zn	3000	5290	4.5
Cr + Cu + Ni + Zn	4000	10140	6

*DM - dry matter

The study of the evolution of trace elements (ETM) containing in sewage sludge and calcined sludge (Cr, Cu, Pb, Zn, Ni) from room temperature to 1100 °C is represented in Figure 4.

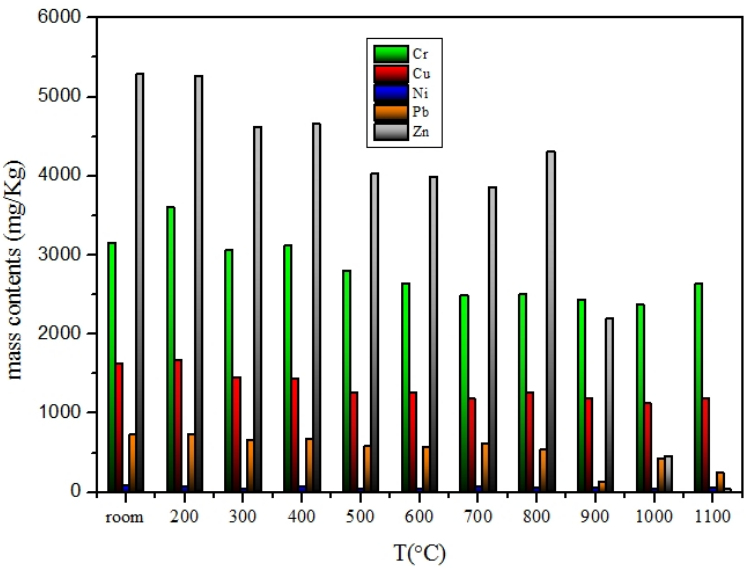

Figure 4*. Evolution of the amounts of ETM with temperature*

Results show that the percentage of ETM decreases slightly and gradually from room temperature to 1100 °C, with the exception in 400 °C. At this temperature we notice an increase in the contents of all ETM, which can be explained by the degradation of significant amount of organic matter and water [30]. The minimum concentration of ETM in calcined sludge was at 1000 °C, in this temperature the amount of Cr, Cu, Pb and Zn decreases with 34.17 %, 32.33 %, 42.46 %; respectively, also we observed that the amount of nickel remains stable during calcinations with a content varying between 50-70 mg/g. The concentration of ETM as a function of temperature can help us selecting the appropriate and the safe way for the reuse of the sludge, such as in cements or ceramics manufacturing [32, 33].

XRD analysis

Figure 5 shows the collected data from X-rays diffraction of sewage sludge and calcined sewage sludge at different temperatures.
Results of analysis of crystalline mineral fraction indicate the presence of Al_2O_3, SiO_2, $CaCO_3$ and CaO corresponding to Powder diffraction files (PDFs) numbers 00-004-0878, 00-001-0649, 00-085-1108, 00-028-0775 respectively.

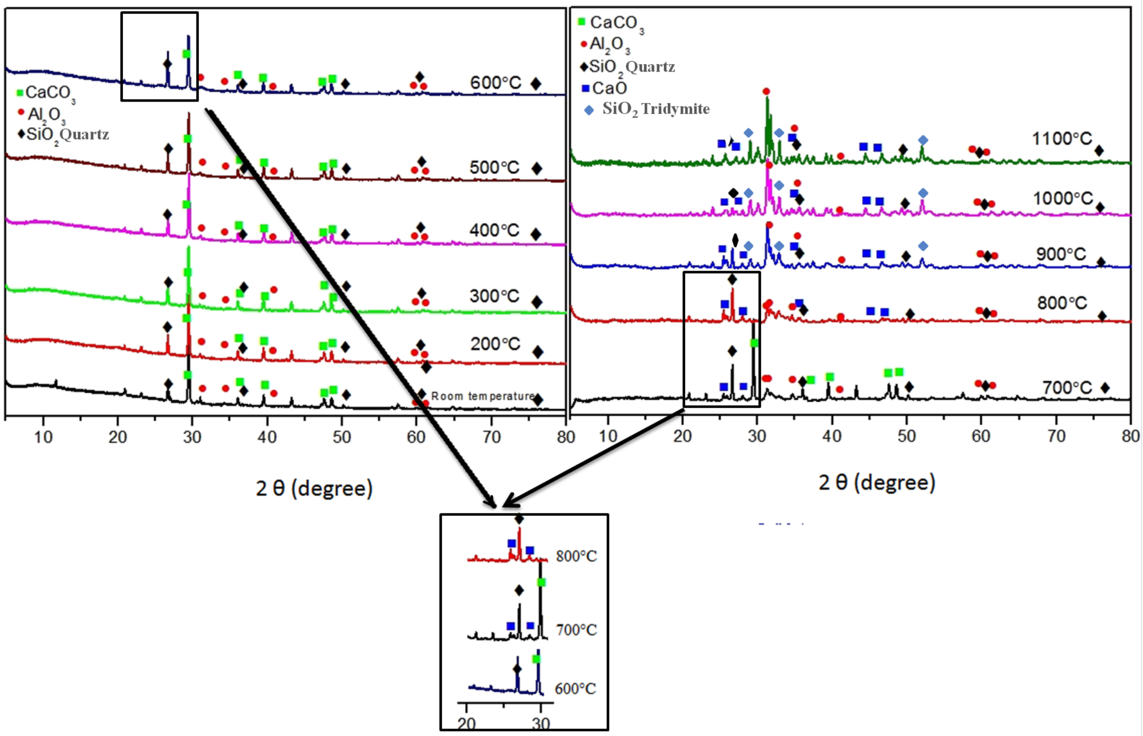

***Figure 5.** XRD of the sludge and calcined sludge*

For Al_2O_3, the intensity of diffraction peaks in spectrum from room temperature to 700 °C is low, due to the nature of the existing phases of aluminum oxide, as Al_2O_3 below 700 °C is a mixture of amorphous and ϒ-Al_2O_3 phases [34, 35]; at 800 °C the peaks become more intense because of the transformation of existing phases to α-Al_2O_3, which has a spinel cubic structure with high peaks intensity [36]. The decomposition of $CaCO_3$ has been well defined in literature, previous studies showing that the decomposition of calcium carbonate begins above 600 °C giving CaO with release of CO_2 [37]. The results extracted from our X-ray diffraction diagrams confirm that the decomposition of $CaCO_3$ begins around 700 °C. The first appearance of small new peaks attributed to CaO was observed at 700 °C and at the same temperature we notice the start of the disappearance of $CaCO_3$ peaks, the new peaks of CaO remain stable up to maximum temperature. The intensity of characteristic peak of quartz form of SiO_2 at 2θ = 26.6° remain stable until 800 °C, at 900 °C a decrease in this intensity due to the transformation of SiO_2 quartz form to SiO_2 tridymite form which known in literature to starting at 870 °C [38, 39] and the appearance of new peaks at 900 °C 1000 °C 1100 °C at 2θ = 27.9°, 33.9°, 53.1° with weakness in intensity of characteristic peak of quartz confirm the phase transition.

Thermal analysis

Both thermal analysis techniques, differential thermal thermogravimetric analysis (DT/TGA) and differential scanning calorimetry (DSC) were used for the study of thermal behavior of sludge (Figure 6).

a)

b)

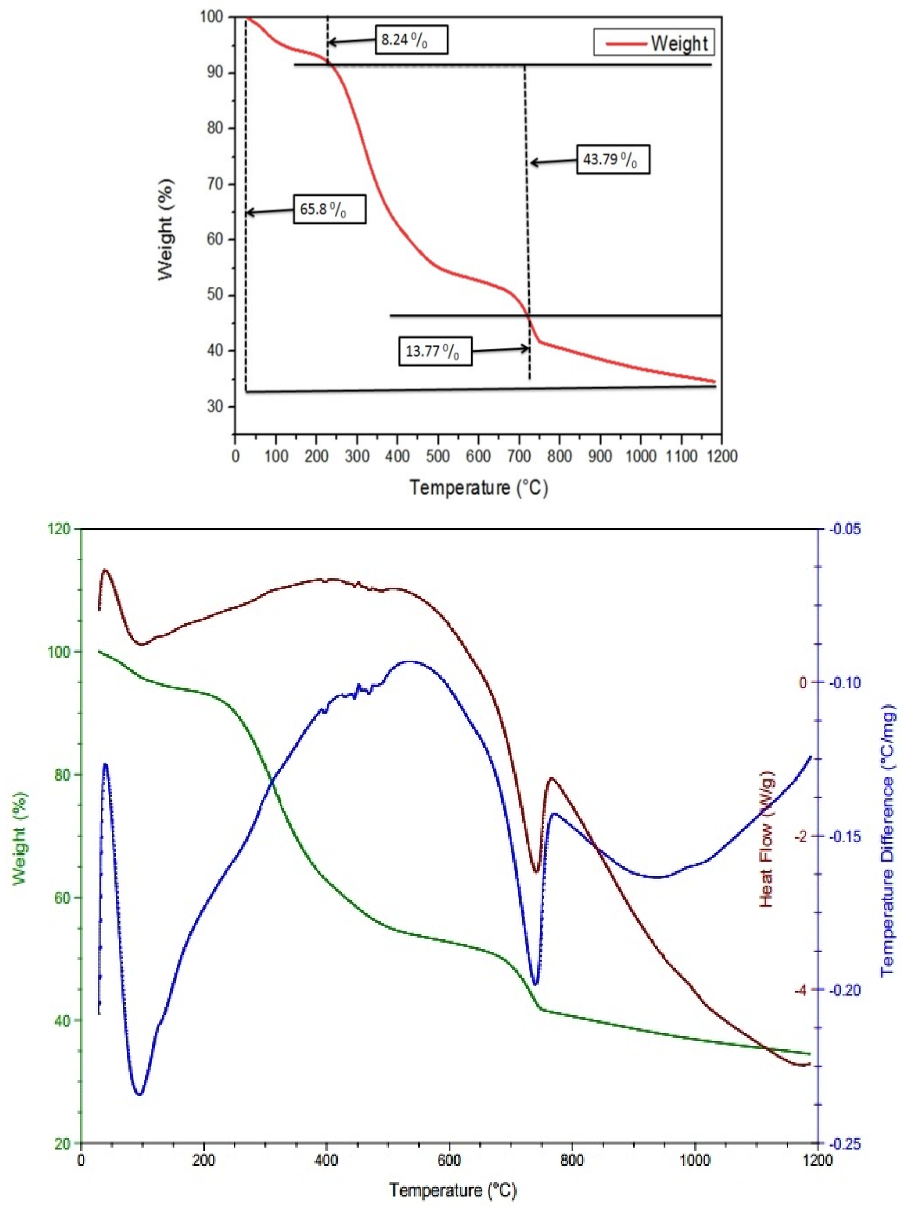

***Figure 6.** Thermal behavior of sludge: a) DT/TGA and b) DSC*

A loss in mass of 8.24 % was observed from room temperature to 230 °C (Figure 6a) accompanied with an endothermic peak (Figure 6b) which could be related to the evaporation of the remaining moisture. Between 230 °C and 700 °C the weight loss was of about 43.79 % with exothermic peak which can be related to the oxidation of organic matter [40, 41]. The third weight loss with value of 13.77 % was in the range from 700 °C to 1100 °C with an exothermic peak due to the decomposition of carbonates with the release of CO_2 [42].

*p*H measurements

pH results of different calcined samples are listed in Table 2. Two main parameters control the *pH* values during calcinations; the decomposition of organic matter and the carbonate level [8]. Results show that *p*H was almost neutral and slightly decreased from 7.20 to 6.27 in the range from room temperature to 300 °C, however, in the range of 400 °C to 1100 °C *p*H was basic with values in the range of 7.73-11.13.

Table 2. *pH values of sludge and calcined sludge*

T [°C]	Room temperature	200 °C	300 °C	400 °C	500 °C	600 °C	700 °C	800 °C	900 °C	1000 °C	1100 °C
***p*H**	7.20	6.82	6.27	7.73	9.28	9.34	10.82	11.04	11.13	9.52	9.45

The increase in basicity with the increase of calcinations temperature can be explained by the formation of basic metal oxides [43, 44].

COD and nitrates

The values of measurements of COD and nitrates of sludge are 2474 $mg \cdot L^{-1}$ and 134.23 $mg \cdot L^{-1}$ respectively.

CONCLUSION

The characterization of the sludge from WWTP of Bazer Sakhra - El Eulma, Setif, Algeria, indicated that the agricultural valorization is dangerous and toxic, considering the high rate of ETM. However, the good way for the reuse of this sludge can be in manufacturing materials. For instance, sludge studied in present study contains nearly 60 % of organic matter which is consistent with results found in literature and the concentration values of the dissolved ions nitrate and the COD of sludge are 134 $mg \cdot L^{-1}$ and 2474 $mg \cdot L^{-1}$ respectively. The evolution of the mineral fraction of the sewage sludge in different temperatures from 100 to 1100 °C was also studied; the obtained results from XRD, XRF show the presence of large number of metals, some of those metals are in crystalline form such as Al_2O_3 $CaCO_3$ SiO_2 and CaO. The measured *p*H of the sludge and calcined sludge range between 6.27 and 11.13, it's depending to organic matter and metals basic oxides contents in sludge. Thermal behavior of sludge was studied using DT/TGA and DSC; obtained results show that total mass loss is near 65 % according with results of mass loss and the total decomposition of carbonates was between 700 °C - 800 °C with the presence of exothermic peak between which confirm results of XRD analysis.

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