Journal of Materials and Environmental Sciences
ISSN : 2028-2508

JMES, 2017 Volume 8, Issue 8, Page 2800-2806

http://www.jmaterenvironsci.com/

2'JP2E, 24-25 Oct. 2016, FP-Taza

Effect of incineration of sewage sludge on the evolution of physicochemical characterization and mineralogical properties

M. Aadraoui[1*], M. Elbaghdadi[1], J. Rais[1], A. Barakat[1], W. Ennaji[1], L.A. Karroum[1], H. Oumenskou[1]

[1]*Laboratory of Geo-resources and Environment, Faculty of Sciences and Technics, University Sultan MoulaySlimane, BéniMellal, Morocco.*

Received 8 Jan 2017
Revised 25Mar 2017
Accepted 28Mar 2017

Keywords

- ✓ Sewage sludge,
- ✓ Characterization,
- ✓ Calcination,
- ✓ Recycling,
- ✓ Construction materials.

m.aadraoui@usms.ma, mohamedgat2012@gmail.com

Phone: (+212)15704525

Abstract

Recently, the protection of the environment through the ecological management of waste has become a major preoccupation of the modern societies. Therefore, our study aims to present the physicochemical and mineralogical properties of sewage sludge calcined at temperatures between 105°C and 950°C by using the technics of the ICP–AES method, X-ray diffraction, Bernard calcimeter test and Bartington MS2 dual frequency sensor. These parameters allow us to arrive at a clear determination of the composition in heavy metals and mineralogical, chemical elements such as calcium, sodium and potassium, the carbonate (CaCo3) and magnetic susceptibility of sewage sludge ashes in order to valorize the use of these ashes in the cement manufactories.

The obtained results showed that sewage sludge ashes contain an important quantity of iron, magnesium, calcium, sodium, zinc, and potassium with a total destruction of organic matter, and the appearance of several mineralogical phases such as britholite, merrillite, and andradite. Concerning the calcite, the values are around 20% and down to 8%. Hence, decreased after incineration of sewage sludge at 550°C and 950°C, calcite decomposes into calcium oxide CaO. The Magnetic susceptibility increases with higher temperatures in accordance with the high content of oxides and metals.In general, the ranges of the magnetic susceptibility measured are around 0.02×10^{-5} to 0.88×10^{-5} SI.

1. Introduction

In the last few years, the increasing demand of water in domestic, agricultural and industrial sectors and repetition of dryness phenomena, has led us to think again about the consideration of wastewater as an important resource to cover that lack observed. In Morocco, the situation is not different from other countries in the world; the volume of rejected wastewater was estimated at 470 Mm^3/year in 1994 and might reach 900 Mm^3/year in 2020 [1], which means that a huge quantity of wastewater needs to be re-used or it will present a high risk to the environment in Morocco. For this reason, the national program for wastewater treatment aims to install over 250 wastewater treatment plants by 2015 [2]. These plants produce an important quantity of solid sewage sludge which is growing more and more with the increasing number of wastewater treatment plants. [3, 4]. Therefore, in Morocco, it is estimated at 320,000 tons per year in 2025 [2]. This is considered as big problem [5], because it contains some toxic and hazardous substances which can be harmful to the public health [6]. According to several studies [7-9], the origin of wastewater, the period of the year, type of adopted treatment, and the condition practiced for the treatment of muds within the purification station, are the principal parameters that can have an influence on the composition of the sewage sludge. Currently, the conventional management of waste sludge is in general the throwing in discharge savages without any treatment or control [10]. The valorization in agriculture by spreading it on the grounds remains limited in Morocco [11,12]. In the industrial sector, the valorization could be by the incineration in cement factories as asource of energy [13-15], because the calorific value of the sewage sludge is 8293 J/g [16].

Several studies have been carried out by various researchers to find out the favorable uses of sludge in the construction materials [17, 18], and they have approved it ability to be used for the instance as a filler in

asphaltconcrete applications [19,20] bricks and tiles [21,22], raw material in a cement kiln [23,24], and in the manufacturing of lightweight aggregates [18, 21,25-27]. Hence, the main aim of this study is to determinate the influence of the temperature change in the incineration process between 105°C and 950°C in the physical, chemical and mineralogical properties of sewage sludge ash [28], to arrive at the recycling of the sewage in an environmental and sustainable construction materials.

2. Material and methods

2.1. Origin of the ashes used

The sample of the sewage sludge used in this work was sampled from the wastewater treatment plant located in the Khouribga city, Morocco, which treats its wastewater by the biological method of the activated sludge. The household waste water of 220000 of inhabitants is treated in this plant.

2.2. Methods

The samples collected were dried in a temperature less than 105°C during 48 hours**,** and further pulverized in a porcelain mill until the particles pass through a 200µm mesh metallic sieve. **At** the level of the laboratory, the calcination was carried out by the introduction of 3 g of the sewage sludge in an electric kiln at different temperatures 105°C, 250°C, 550°C and 950°C, for 2 Hours [29]. After the heating, the calcined sewage sludge was cooled in a desiccator to room temperature of the laboratory to investigate analyses [30].

2.3. Analyses

The characterization of the sludge ashes was carried out in the following analysis:

- The mineralogical composition: performed based on The X-ray diffraction (Bruker X-ray diffractometer equipment) in a radiation of half an hour scanning, which explores in the interval of 2Θ angles from 10° to 90°, at different temperatures 105°C, 250°C, 550°C 950°C.
- Heavy metal concentration: The heavy metal concentration in the sludge ashes were analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES) equipment after dissolving sample of ash sludge in water with the small quantity of nitric acid and hydrochloric acid.
- Sensibility magnetic: was measured a Bartington MS2 dual frequency sensor in order to determine the mineral matters of the sample in the sludge ash.
- The determination of the carbonate rate contents in samples were realized by the carbon dioxide evolution (CO_2), afterwards the reaction was observed in diluted hydrochloric acid, by the Bernard calcimeter method. The carbonate was calculated by the difference between the initial and the final volume of the sample. [31]
- Loss of weight: The percentage of mass loss at different temperatures (original, 250°C, 550°C and 950°C) was calculated according to the equation below [32]:

  $$\text{Weightloss (\%)} = [(m_i - m_a)/m_i]*100$$

  With m_i is the initial mass (g); m_a is the mass after calcination (g).

3. Results and discussions

3.1. Weight loss of sewage sludge during the calcination

The weight loss analysis of calcined sludge in different temperature studied, are presented in figure 1.

From the room temperature to the average of 105C°, the loss of weight had an intense decrease rhythm; which can be explained by the elimination of moisture and water absorbed. [33-36].

From 250°C to about 550°C, the loss of weight keeps the same decrease rhythm, the losses most likely are attributed to the combustion of organic matter, the complex nonvolatile organic matter and species of hydrocarbons and inorganic materials of structural water, the sewage sludge composition contains various substances that volatilize in boiling temperature[33, 35, 36]. In the interval from 550 to 950°C, it can be seen that the elimination of the carbonaceous matter present in sewage sludge by thermal decomposition caused a total disappearance of organic matter [33,36]. In more than 950°C temperature, no more loss of weight can be seen, because all the substance is dehydrated.

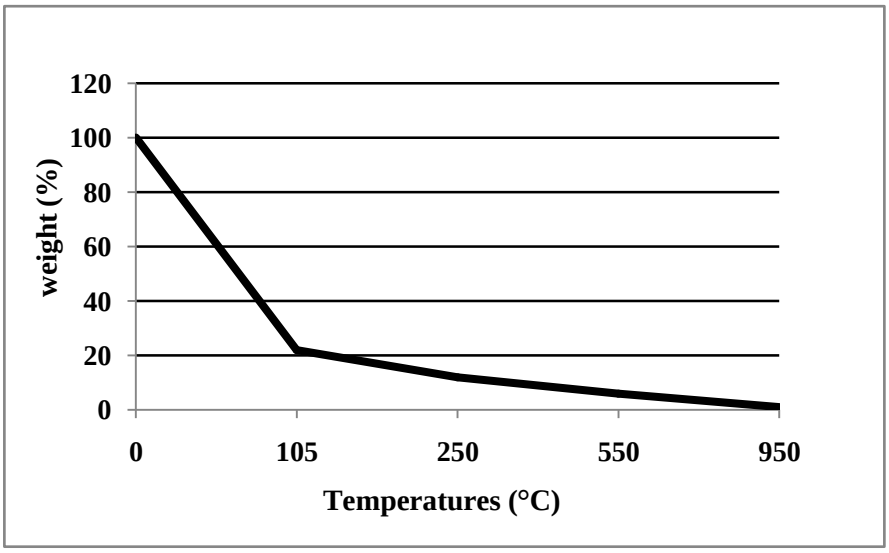

Figure 1: Weight loss of sewage sludge during the calcination at 105°C – 950°C

3.2. Magnetic susceptibility

The magnetic susceptibility of different samples is presented in figure 2 which gives an increasing result of the magnetic susceptibility with the temperature. The ranges of metal contents measured are around 0.02×10^{-5} to 0.88×10^{-5} SI.

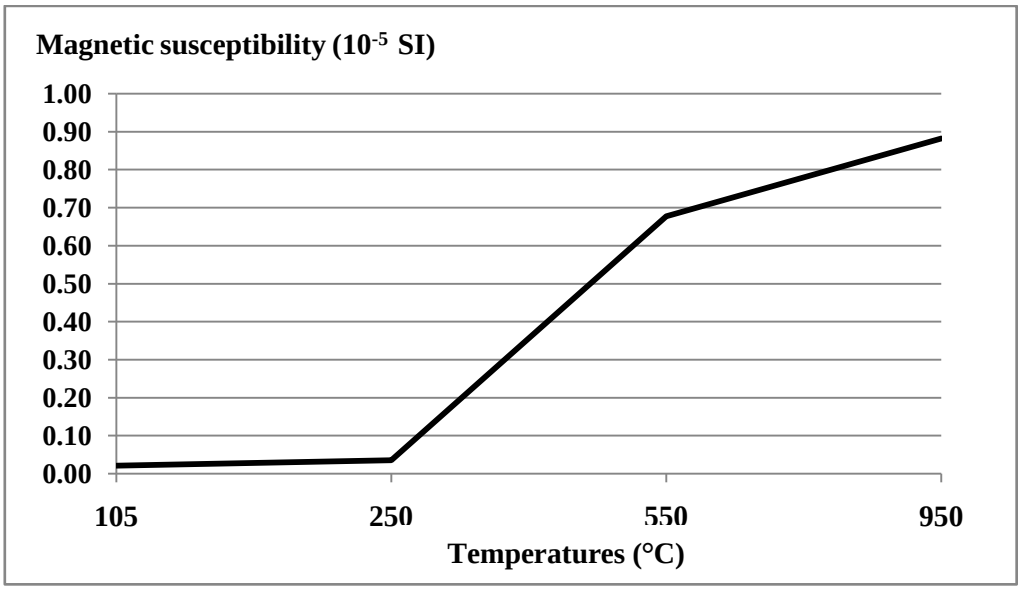

Figure 2: Variation of magnetic susceptibility according to the calcination temperatures

From the diagram above, the magnetic susceptibility in a temperature between 105°C and 250°C stayed in an almost weak rhythm, which is due to the water absorption by the incineration temperature. From the two ranges 250–550°C and 550–950°C, the magnetic susceptibility had a similar increase with the temperature in an intense way, which can be described by the total change of the organic matter statement to a dry volatile one that contains a strong portion of the mineral matters.This result is will be confirmed with the chemical analysis using the ICP method.

3.3. Carbonate rate of sewage sludge during the calcination

The evolution of the carbonate quantity during thermal treatment is given in the following figure 3:

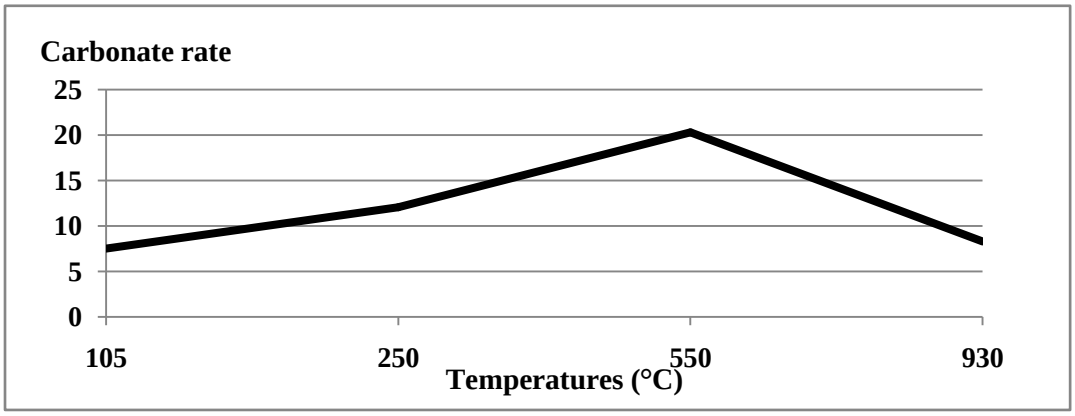

Figure 3: Variation of carbonate calcium according to the calcination temperatures**.**

The rate of carbonate had a major increasing between 105°C, 250°C, and 550°C temperatures. After that, in a temperature range of 500°C–950°C (sewage sludge incineration), the calcite is diminishing due to its decomposition in a proximate temperature of 700 °C. This result was proved by several studies which showed that the decomposition of carbonate is near 750°C to 800°C [3,31,36-39]. To give more significance to thisresult, we will confirm it by the X-ray diffraction analysis.

3.4. Chemical composition of sewage sludge ash

The table 1 below represents the results of major elements analysis of different sludge residues in calcination of 105°C, 250°C, 550°C and 950°C temperatures, which have been realized by the spectrometry of emission (ICP-AES). The obtained results revealed the presence of toxic and nontoxic elements in all residues.

Table 1: Major and some minor elements present in sewage sludge ash calcined at 105- 950°C

Elements(mg/Kg)	**105 °C**	**250 °C**	**550 °C**	**950 °C**
Major elements in excess (> 10000 mg / kg)				
Ca	51000	98390	136700	161900
Fe	5830	9947	15150	19470
Mg	5273	9355	13470	16770
Major Elements (100 - 10000 mg / kg)				
Cu	168,5	188,3	292,5	347,1
K	1823	2207	3367	5359
Mn	93,30	153,9	201,7	271,2
Na	1913	2781	4100	4894
Pb	61,59	219.8	146,6	119.5
Zn	1218	2229	1645	3313
Al	4667	7091	ND	ND
Ba	220,1	390,7	501,7	663,5
Trace Elements (<100 mg / kg)				
Cd	4,248	11,96	7,036	8,797
Ni	15,82	30,50	28,17	41,59
Co	2,350	2,571	4,928	5,882
Cr	31,68	52,17	79,11	93,28
P	ND	ND	ND	ND

ND: not detected due to the detection limits of ICP.

The results of chemical analysis given in table 1 showed the major chemical components of sewage sludge ashes like iron, magnesium, calcium, sodium, zinc, and potassium. According to results obtained by [7,28,40-

42], the high concentration of the calcium and iron in the sewage sludge ash, is owing to the employ of ferric salts during wastewater treatment [34, 36], and the existence of alumina and silica ought to be assigned to materials brought by pluvial waters. The high percentage of phosphates is probably due to domestic detergents [31,34,36]. In addition, a few elements such asPb and Cd are highly volatile at a higher temperature, in consequence, concentrations in sewage sludge ash decrease around 250°C to 950°C, and this result is similar to the one obtained by Naamane et al, [28]. In general, the concentrations of chemical elements in sewage sludge increase with the temperature elevation in the calcination process, this may be attributed to the fact that during the calcination these elements react with oxygen gas to form its oxides. Otherwise, the minor constituents are with low concentrations (< 100 mg/kg); such as Cd, Co, Ni, and Cr. The results are in a good agreement with [28] and the other metals are negligible e.g, Al and P. Moreover, various elements such as Mn, Na, Ba, Zn and Cu are relatively slowly volatile, thus their concentrations in sewage sludge ash stay stable or grow around 250°C to 950°C. This result encourages the incorporation of the sewage sludge ash in the cement [33].

3.5. Analyzed RDX

The figure 4 presents the mineralogical analysis performed by the x-rays diffraction of sewage sludge calcined at different temperatures (105°C, 250°C, 550°C, 950°C) and during 4h, and allows characterizing the nature of the mineral phase's crystallized present in sewage sludge ashes. The main constituents found are represented bellow:

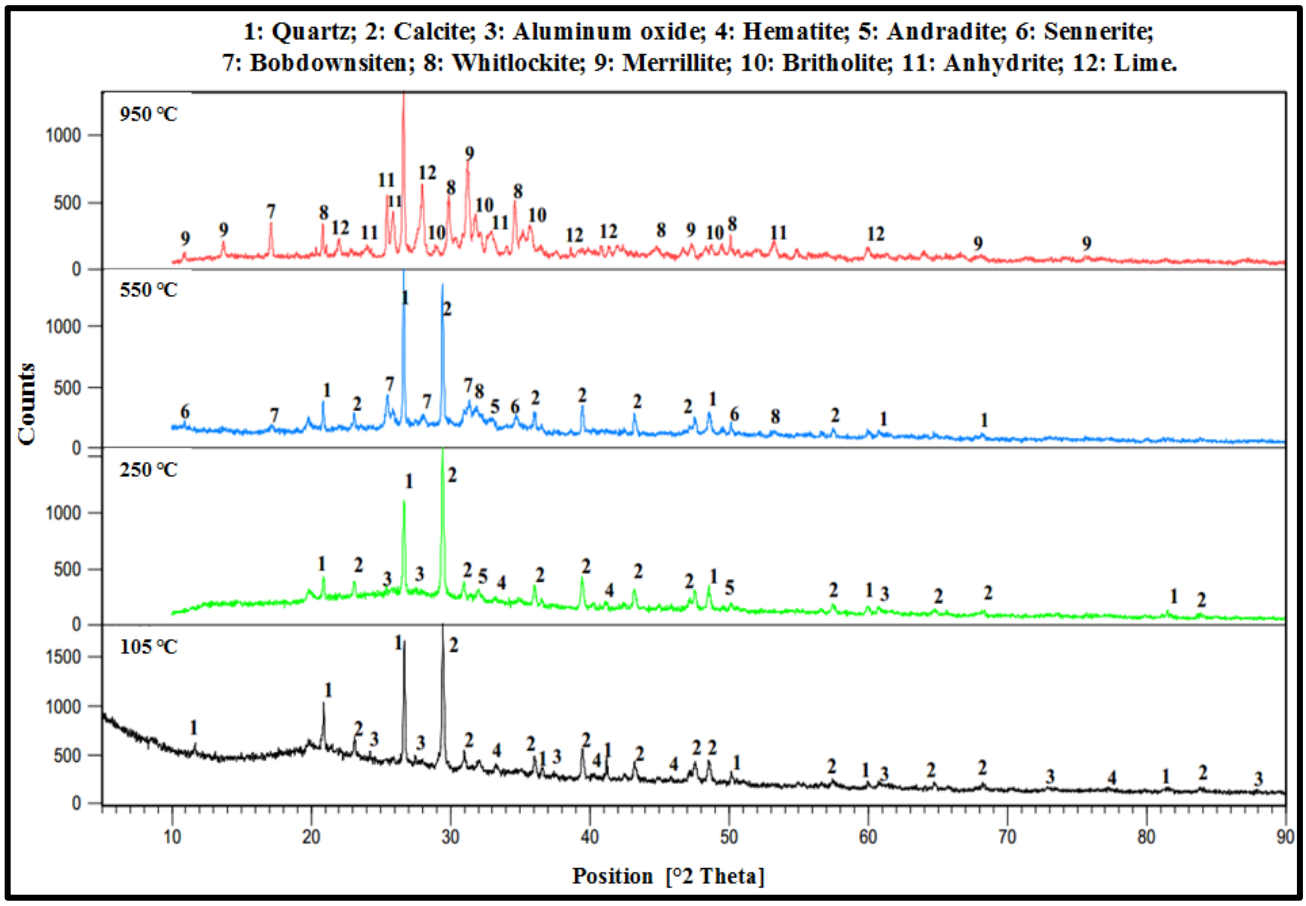

Figure 4: X-ray diffraction diagrams of sewage sludge ashes

The sample calcined between the ranges of 105 °C to 250 °C: revealed the presence of the following crystalline phases: quartz (SiO_2), calcite ($CaCO_3$), hematite (Fe_2O_3), and the presence of aluminum oxide (Al_2O_3). These compositions are similar with other obtained by some researchers, in particular [30, 31, 33,36, 42,43]. The temperature around 550°C characterized by the presence of quartz, hematite, and andradite [$Ca3Fe_2$ (SiO_4)3] [36],bobdownsite [$Ca9(Mg)(PO_4)6(PO_3F)$],and calcium phosphate (whitlockite -[$Ca9(Mg, Fe)(PO_4)6PO_3OH$]) [44]. During thermal treatment, the iron is transformed into hematite [31, 36]. The calcite identified as a component in the original sample, is not present after thermal treatment at 950 °C because it decomposes about 800 °C, at atmospheric pressure, to give's the lime (CaO) and CO_2 [36, 44], and characterized by the presence

of theMerrillite[Ca9NaMg $(PO_4)_7$],Britholite [(Ce,Ca)5(SiO_4,PO_4)$_3$(OH,F)] and calcium sulfate (Anhydrite-[$CaSO_4$]) presence.
The main mineralogical compounds of sewage sludge ashes after incineration in 105 and 550 are calcite, quartz, hematite and aluminum oxide, and also, after thermal treatment at 550 °C and 950 °C, the main compounds occurring in thermal treatment are lime, whitlockite, britholite and Anhydrite, these compounds are similar to those of cementitious materials. In addition, these compounds are responsible for the pozzolanic activity in cementitious materials [17, 42, 43, 45, 46].

Conclusion

In this research, the evolution of the characteristics of the sewage sludge in different temperatures of 105°C, 250°C, 550°C 950°C, gives leads us to the following conclusions summarized below:

- After incineration of sewage sludge at 550 and 950 °C, calcite is not present, because it decomposes into lime CaO
- Sewage sludge principally contains concentrations such as calcium, iron, magnesium, sodium, and potassium becomes higher with the increase of the calcination temperature. Generally, the majority of elements concentrations increase with the calcination temperature.
- The main mineralogical compounds in the sewage sludge ash are SiO_2, Fe_2O_3, CaO, and Al_2O_3 those components are similar to that of ordinary Portland cement i.e. responsible for the pozzolanic activity in cementitious materials. Moreover, the thermal treatment provokes the formation of several mineralogical phases: andradite, bobdownsite, merrillite, britholite, and Anhydrite and whitlockite

Considering these results, the re-use of sewage sludge ash in cementitious materials as a primary or secondary adjuvant showed its highest possibility. This will take part in the safeguarding of the natural resources and the valorization of the harmful solid waste against the pollution of the environment.

References

1. AMIR S., Doctoral dissertation, *Thèse Doctorale Sciences Agronomiques*–L'Institut National Polytechnique de Toulouse. Toulouse, (2005).
2. El Fels L., *Doctoral dissertation, École Doctorale Sciences de l'univers, de l'environnement et de l'espace* Toulouse, 159341302 (2014).
3. Valderrama C., Granados R., Cortina J-L., *Chemical Engineering Journal*, 232 (2013) 458–467.
4. Fang P., Tang Z-J., Huang J-H., Cen, Zhi C-P., Tang X., Chen X., *Fuel Processing Technology*,137(2015)1–7.
5. Werther J., Ogada T., *Progress in Energy and Combustion Science*, 25 (1999) 55–116.
6. Naamane S., Rais Z., Chaouch M., *J. Mater. Environ. Sci*., 5 (S2) (2014) 2515-2521.
7. Chen M., *Thèse de doctorat, L'Institut National des Sciences Appliquées de Lyon*, N. d'ordre : 2012ISAL0046, (2012) 270p.
8. Jard E., Mansuy L., Faure P., *Journal of Analytical and Applied Pyrolysis*, 68-69 (2003) 331-350.
9. Singh K.P., Mohan D., Sinha S., Dalwani R., *Chemosphere*, 55 (2004) 227–255.
10. Ambkin L., Nortcliff D., White S., *TrAC Trends in Analytical Chemistry*, 23 (2004) 704–715.
11. Peters G. M., Rowley H.V., *Environ. Sci. Technol*., 43 (2009) 2674-2679.
12. Rogers M., Smith S.R., *J. Water Environ*., 21 (2007) 34-40.
13. Cartmell E., Gostelow P., Riddell-Black D., Simms N., OakeyJ., MorrisJ., Jeffrey P., Howsam P., Pollard S.J., *Environ. Sci. Technol*., 40 (2006) 649-658.
14. Malerius O., Werther J., *Chemical Engineering Journal*, 96 (2003) 197–205.
15. Wauthelet M., *Séminaire International sur la réutilisation des eaux usées traitées et des sous produits de l'épuration, Tunis*, Tunisie, (2003). 24-25.
16. Husillos-Rodríguez N., Martínez-Ramírez S., Blanco-Varelaa M. T.,Donatello S., Guillem J. P. C, Fos M.,Larrotcha E., Flores J., *Journal of Cleaner Production*, 52 (2013) 94 -102.
17. Cyr M., Coutand M., Clastres P., *Cement and Concrete Research*, 37 (2007) 1278–1289.
18. Tay J-H., Yip W-K., 1989, *Journal of Environmental Engineering*, 115 (1989)56-64.

19. Wegman D.E., Young D.S., *67th Annual Transportation Research Board Meeting, Washington DC*, 23 (1988).
20. Al Sayed M.H., Madany I.M., Buali A.R.M., *Construction and Building Materials*, 9 (1995) 19–23
21. Tay J.H., Show K.Y., *Conservation and Recycling*, 6 (1992) 191–204.
22. Lin D.F., Luo H.L., Sheen Y.N., *Journal of the Air and Waste Management Association*, 55 (2005) 163–172.
23. Lin K.L. Chang K.Y., Lin C.Y., *Cement and Concrete Research*, 35 (2005) 1074–1081.
24. Lin K.L., Lin C.Y. *Cement and Concrete Research*, 35 (2005) 1999–2007.
25. Bhatty J.I. , Reid K.J., *Waste Management and Research*, 7 (1989) 363–376.
26. Wang K.S. , Chiou I.J. , Chen C.H., Wang D., *Construction and Building Materials*, 19 (2005) 627–633.
27. Huang C. H., Wang S-W., *Construction and Building Materials*, 43 (2013) 174–183.
28. Naamane S., Rais Z., Taleb M., *Construction and Building Materials*, 112 (2016) 783–789.
29. Naamane S., Rais S. et Taleb M., *Matériaux & Techniques*, 101 (2013) 703.
30. Lin K. L., Chang K-Y., LinD-F., *Journal of Hazardous Materials*, 128 (2006) 175–181
31. Merino I., Arévalo L. F., Romero F., *Waste Management*, 25 (2005) 1046–1054.
32. Karayildirim T., Yanik J., Yuksel M., Bockhorn H., *Fuel*, 85 (2006) 1498–1508.
33. Naamane S., Rais Z., Lachquar M., Taleb M., *Mater. Environ. Sci*., 5 (2014) 2212-2216.
34. Karayildirim T., Yanik J., Yuksel M., Bockhorn H., *Fuel*, 85 (2006) 1498–1508.
35. Lynn C. J., Dhira R. K., Ghataora G. S., West R. P., *Construction and Building Materials*, 98 (2015) 767–779.
36. Tantawy M. A., El-Roudi A. M., Abdallah E. M., and Abdelzaher M. A., International Scholarly Research Network, *ISRN Chemical Engineering*, 2012 (2012) 8.
37. Chahidi ELOuazzani D.., Bouamrane A., Mansouri K., Fokam C. B., J. *Mater. Environ. Sci*., 3 (2012) 628-635.
38. Bouamrane A., Chahidi ELOuazzani D., Tiruta BarnaL., Mansouri K., *J. Mater. Environ. Sci.*, 5 (2014) 605-614.
39. Chen M., Blanc D., Gautier M., Mehu J., Gourdon R., *Waste Management*, 33 (2013) 1268–1275.
40. Cyr M., Coutand M., Clastres P., *Cement and Concrete Research*, 37 (2007) 1278-1289.
41. Hui-Sheng Shia, Kan L. L., *Journal of Hazardous Materials*, 164 (2009) 750–754.
42. Naamane S., Rais Z., Mtarfi N.H., El Haji M., Taleb M., *Phys. Chem. News*, 74 (2014) 44-50.
43. Cyr M., Klysz G, Julien S. et Clastres P., *Déchets sciences & techniques*, 29 (2003) 22-29.
44. Cyr M., Idir R., Escadeillas G., *Journal of Hazardous Materials*, 243 (2012) 193– 203.
45. Chen H. X., Ma X., Dai H., *Cement & Concrete Composites*, 32 (2010) 436–439.
46. Lin Y., Zhou S., Li F., Lina, Y. *Journal of Hazardous Materials*, 213– 214 (2012) 457– 465.

(2017) ; http://www.jmaterenvironsci.com/