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Recycling of sludge ash of wastewater treatment plants in cementitious materials; effect of rheo-mechanical characteristics

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ABSTRACT/RESUME

Abstract: *The present study is a contribution to the valorization of even the elimination of sludge storage in wastewater treatment plants (WWTPs). Indeed, after the calcination of sludge, ash obtained is incorporated into cement, for the production of ecological cements, or eco-cements.*
This approach constitutes a durable solution and unquestionable safeguarding of our ecosystem of any nuisance which can endanger the balance of our environment. The eco-cement is developed by substitution of different amount of ash (5%, 10% and 15%) obtained into the clinker. The workability of different cement paste of eco-cements developed can be characterized by the evaluation of yield shear stress (τ_y) *by using mini slump cone. Mortar samples were tested to observe the evolution of the mechanical compressive strength was according to time (for 2, 7 and 28 days). The results obtained showed a remarkable increase in yield shear stress of cement paste and an improvement in the strength of mortar in the cured state.*

I. Introduction

During the second half of the twentieth century, both water pollution and water consumption have increased together to a high level as a result of the demographic explosion. In industrialized countries the reduction and the control of water consumption is linked to the optimization of processes of industrial and domestic wastewater treatment [1].
Excess sludge treatment and disposal currently represent a rising challenge for wastewater treatment plants due to economic, environmental and regulation factors.
Algeria has set targets for 2025 in the field of spatial planning in which it integrates the concept of sustainable development [2]. This new vision is based on economic growth, social equity and protection of the environment [3].
Therefore, a fresh impetus to new appropriate strategies and technologies helpsin reducing excess sludge production in biological wastewater treatment processes [4].
There is a strong need of perspective which is technically feasible and economically favorable for WWTP managing companies, as well characterized by a positive environmental balance [5]. Several ways exist for the elimination of this sludge, but the choice remains often related to the cost of the installation, the origin of the sludge, the added-value of the products which results from this and the impacts of the solution retained on the environment. Among the solution retained to treat the sludge, we can cite the production of biogas as a source of heat and electricity on the one hand, and agricultural valorization in the production of fertilizer and compost

on the other hand. Other fields of valorization of sludge from WWTPs are explored during the last two decades. Most of the recent works explore the heat treatment to reduce the volume of the sludge and to stabilize the mineral phases [6, 7, 8, and 9]. Without denying the interest of the ways of valorization quoted above, those consistent to incorporate them in the production of ecological cements constitute an ecological and economical solution. The use of such materials which are mostly considered as industrial wastes or by products, leads to a significant reduction in CO2 emissions [10, 11, 12, 13, 14, and 15]. Meanwhile, chicken eggshells are used in enormous quantities by food manufacturers, restaurants and household and the shells are disposed of as solid waste. Indeed, world consumption of the cement strongly increases during the last twenty years, with an average rhythm of increase higher than 5% a year.

The demography, the urbanization and the economic growth, increase the needs in housing and infrastructure in the building and public works sectors. Moreover, according to researchers and cement producers, the manufacturing of cement is strongly energy-consuming and has a harmful environmental impact. The valorisation of dried sludge (dryness > 92% SM) from WWTPs could contribute to the reduction of the need in energy for cement-manufacturers. The organic matters of sludge improve the calorific power considerably. The introduction of sludge into new cement can allow reduction of CO_2 emission.

Fresh cementitious materials, as many materials in industry or nature, behave as fluids with a yield shear stress, which is the minimum stress for irreversible deformation and flow to occur. This yield shear stress is a unique material property and may, in the case of cement pastes (i.e. fine particles), be measured using conventional rheological tools [16] obtained using an analytical model.

In this study, after a detailed characterization of raw and calcimined sludge of WWTPs, various composition of the eco-cement (with increasing amount of substitution in the raw cement) was tested. To evaluate the performance of the new binders, prismatic samples made of mortars were carried out and tested in compression.

II. Materials and methods

II.1. Origin of sludge sample

The samples of the studied sludge were taken at the level as of beds of solar drying of the WWTPs known "Rochet" in the wilaya of Sidi Bel Abbes (west of Algeria, 400 km from Algiers (Algeria)). The taken samples were preserved at a temperature of - 4 ° C in order to eliminate any risk of fermentation. The volatile solids (VS), meanwhile, are measured by the loss on dried sample ignition at 550°C for 2 hours according to the standard NF EN 12879 [17].

II.2. Physicals and chemicals characteristics

Physical parameters of the sludge such as: the amount of dry volatile matters (DVM), the amount of humidity, the amount of dry matter (DM), the organic matter content (OM), the amount of mineral matter (MM), dry apparent density and the Methylene blue value (MBV), are measured in accordance with French standards as shown in Table 1.

The concentrations of Cd, Cr, Cu, Ni, Pb, Mg and Zn in the ash of study sludge samples in front of their limit values according to the French standard NF U 44 - 041 are given in Table 5.

Table 1. *Standards used to characterize the physical properties of the WWTPs sludge.*

Property	Test standard
Loss of ignition of dry mass	NF EN 12879
Particle density and water absorption	NF EN 1097-6
Dry matter	NF U44-171
Loss on ignition in waste, sludge and sediments	NF EN 15169
Methylene blue of soil by means of the stain test	NF P94-068

For the characterization of ashes resulting from the calcinations of sludge from WWTPs, analyses of X-ray diffractometry were undertaken. In these experiments, the diffractometer used is of the type OXFORD 1000 MDX (Multi-dispersive X-ray fluorescence analyzer element).

II.3. Preparation of eco-cements

For the preparation of the eco-cement; mixtures of clinker, of ash resulting from the incineration of sludge WWTPs and Gypsum are carried out. Before carrying out a chemical and mineralogical analysis of the various components and the mixtures of the finished products realized, a neat crushing was made in a micro-crusher. The duration of crushing with respect to Blaine surface was fixed to three minutes. The compositions of formulated eco-cements are given in Table 2.

Blaine surface measurements, chemical and mineralogical analysis are carried out.

Table 2. *Composition in mass proportion of the developed cements.*

Cement	Gypsum (%)	Clinker (%)	Ash (%)
ECC	5	95	0
EC5	5	90	5
EC10	5	85	10
EC15	5	80	15

II.4. Yield shear stress by mini slump test

As the rheology of cementitious material is closely related with developing performance of concrete, the rheology is considered one of the important factors for evaluate the workability.

Slump test is the most common method used in concrete industry to evaluate the workability of a fresh concrete. When rheology came in to the play, investigations have been focused on identifying the relationship between the workability measures (slump test observations) and the properties of rheology; yield shear stress and plastic viscosity.

Experimental studies have suggested a relationship between the slumps spread diameter and yield shear stress that depends on the volume fraction of the material. Furthermore, it has been found that the relation becomes less dependent on the volume fraction as the concrete becomes more workable. The obstacles against flow become less considerable with better matrix lubrication between coarse aggregates and mortar like in self-compacting concrete (SCC) [18].

Using the analytical model developed by Russel the yield shear stress can be calculated by the final spread diameter as the following equation:

$$\tau_y = \frac{225*\rho*g*V^2}{128*\pi*SD^5} \quad \textbf{(1)}$$

Where **ρ** is density of the material, **g** $(\mathbf{m.s^{-1}})$ is acceleration of gravity, **V** (m^3) is the mini cone volum and **SD** (m) is the slump spread diameter.

The characterization of paste cement rheology is based on the concept that the fresh paste can be considered as a fluid. The mini slump cone apparatus has used for study the evaluation of yield stress of the different paste cement developed, (see Figure 1). The mini slump cone geometries are giving in Table 3.

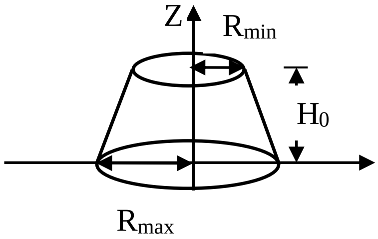


Figure 1.*Mini slump cone shape*

Table 3. *Mini slump cone geometries (NF EN 1961)*

Rmin (m)	H (m)	Rmax (m)
0.019	0.057	0.038

II.5. Mechanicals characteristics

Prismatic samples (40x40x160 mm^3) of a mortar made of the eco-cements and standard cement were tested (MCC, MC5, MC10 and MC15).

The mortar used is composed in mass, of one part of cement (or eco-cement), three parts of standard sand and a half part of water (in masse water/ cement is equal to 0.5).

Each batch for three test specimens comprises 450 g ± 2 g of cement, 1350 g ± 5 g of standard sand and 225 g ± 1 g of water.

III. Results and discussion

III.1. Physicals and chemicals characteristics

The physical characteristics of the studied sludge are shown in Table 4. From these results, it is to note the high water content, the high OM (≈ DVM) levels and a relatively low value of methylene bleu value. Compared with the characteristics of other WWTPs sludge, these values are relatively comparable [19].

Table 4. *Physical characteristics of sludge of WWTPs*

Property	Test standard
Dry matter (%)	85.11
Dry volatile matter (% DVM)	39.87
Mineral matter (% DVM)	60.13
Humidity (%)	14.88
Particle density	0.69
Methylene blue of a soil by means of the strain test	0.85

The results presented in Table 5 show that the sludge contains a low metal trace elements (MTE) compared to the thresholds in the standard NF U 44- 051.

In terms of the characterization of the eco-cements designed, Blaine specific area is presented in Table 6. It should be noted a sensitive increase of the Blaine specific area (over 20%) of cement containing ashes of calcified sludge. However, it is not possible to relate solely the increase in specific surface area to the ashes content incorporated in different cements.

Table 5. *Chemical composition of study sludge*

Elements	Limit value of MTE (ppm)	Value of MTE in ash (ppm)
Cd	10	nd
Cr	1000	100.5
Cu	1000	189
Ni	200	nd
Pb	800	nd
Mg	10	4.06
Zn	3000	142.5

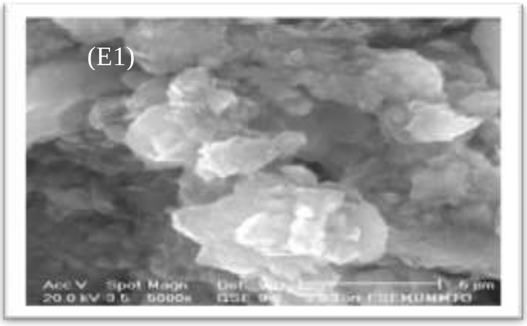
nd*: note detected and L. MTE: limit value of metal trace elements, SS (ppm): sludge study

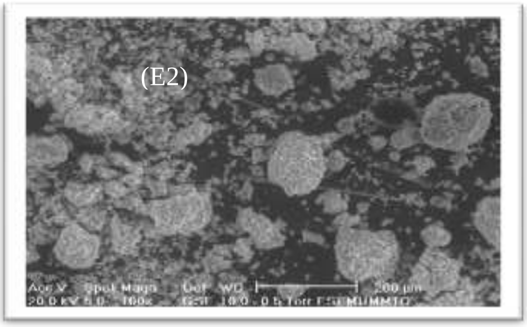
Table 6. *Blaine specific area of eco-cements developed*

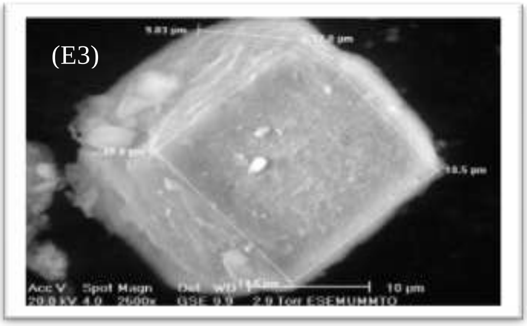
Designation	ECC	EC5	EC10	EC15
Specific area cm^2/g	3636	4497	4401	4379

III.2. Analysis by environmental scanning electronic

The scanning electron microscopic method is a good technique for showing the structure and morphology of solids. In terms of the observations using an environmental scanning electron microscope, Figure .III shows the different forms of studied sludge. The images obtained show particles of different shapes and sizes. However, an important part of these particles are in the form of plates (E3). The resulting showed non ordered structure with a very heterogeneous distribution of particles.







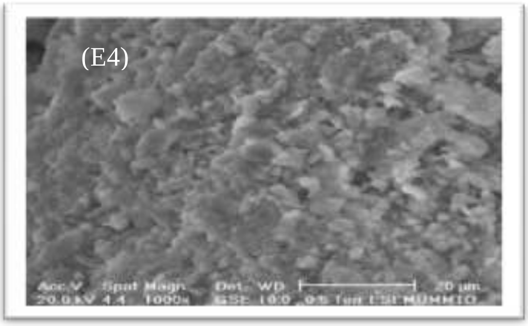


Figure 2. *ESEM photograph from E (E1, magnification 500 x; E2, magnification 2500 x; E3, magnification 5000 x; E4, magnification 1000 x) granular sludge.*

III.3. Cement pastes rheology

To study the effects of adding ash of sludge studied on the rheological properties; tests on the cement grouts were carried out by the mini-cone test measurement according to standard NF EN 196-1 giving in Figure 3.

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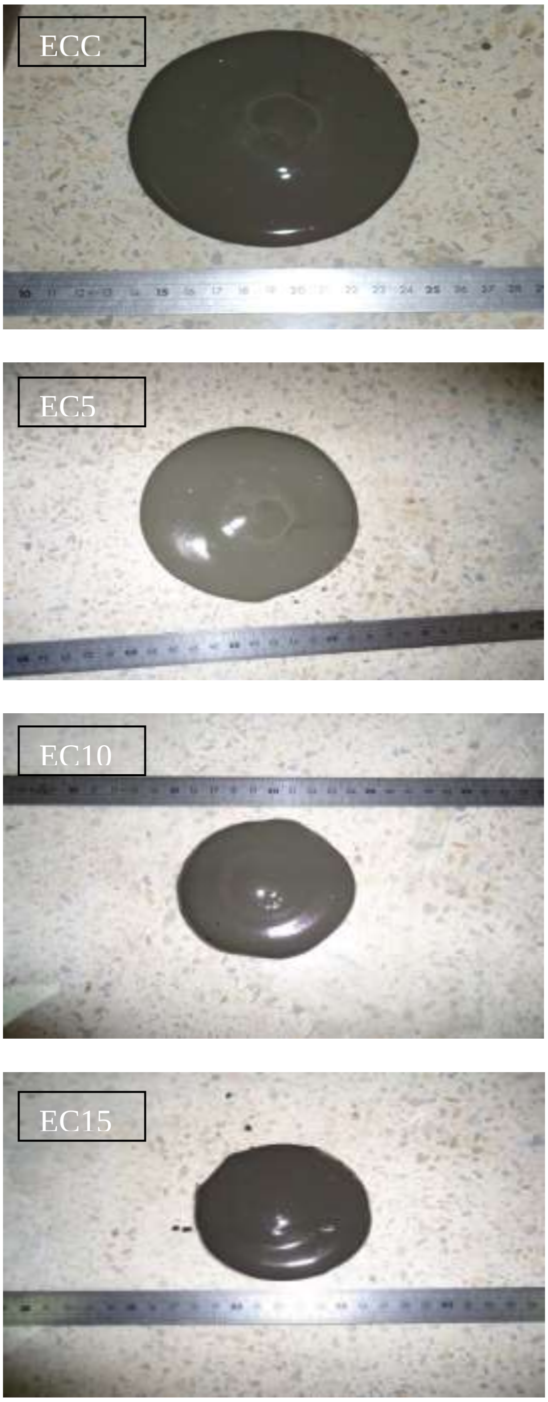

Figure 3. *Rheology of eco-cements pastes using the mini-cone (ECC, EC5, EC10 and EC15)*

Table 7. *Evolution of yield shear stress vs rate*

Designation	ECC	EC5	EC10	EC15
Ra (%)	0	5	10	15
ρ	1,7	1,68	1,64	1,61
SD (M)	0,104	0,09	0,089	0,085
τ_y (Pa)	13,42	27,34	28,22	34,87

From the results shown in the Table 7 above; a remarkable increase in the shear stress is observed between the cement paste control and all the cement paste.
Further, a power law linear function is developed to relate yield shear stress and rate of substitute ash as follows:

$$\tau_y = 1{,}3046 * R_a + 16{,}178,\ R^2 = 0{,}8732 \quad \mathbf{(3)}$$

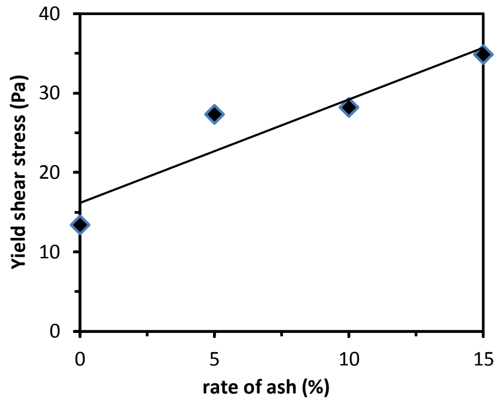

Figure 4. *Variation of yield shear stress vs rate sludge's ash content by mini slump cone*

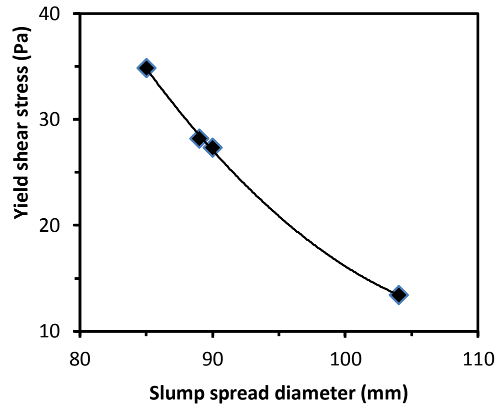

Figure 5. *Correlation between mini slump diameter and yield shear stress*

A good correlation between yield shear stress and slump spread diameter can be observed especially over the low yield shear stress values. The low function is developed to relate yield shear stress of cement paste and rate of sludge ash as follow:

$$\tau_y = 0{,}0301 * (\mathrm{SD})^2 - 6{,}8087 * (\mathrm{SD}) + 396{,}34,\ \mathrm{R}^2 = 0{,}9994 \quad \mathbf{(4)}$$

III.4. Mechanicals characteristics

The mechanical results presented in Figure 6 show that the eco-cements used make it possible to develop strengths at the early age (7days) and

higher than the strength measured on samples including the standard cement (ECC).
This increase in strength can be attributed, in regard of the measured parameters, to the specific surface developed by the ec-cements. The high specific surface area developed by the eco-cements (see Table 6) probably allowed a more important reactivity, hence, high speed of formation of hydrates silicate calcium (H-S-C) gel. The high specific area of the eco-cements could predict also finer grain size distribution of the ecological cement.
This later fact could induce an increase of the strength by increasing the compactness of developed mortars.

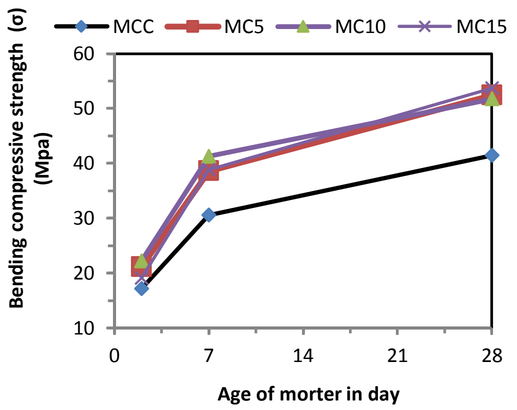

***Figure 6**. Evolution of compressive strength*

IV. Conclusion

In this study, a valuation approach of WWTPs sludge in the cement industry has been proposed. A detailed characterization of the sludge and the ashes issued from the heat treatment of the sludge, are performed. This first step has allowed comparing the physical as the chemical characteristics of the studied sludge to the results published in other countries. From this comparative study, it appears that the sludge studied is comparable in terms of physical as chemical characteristics. Moreover, the chemical and mineralogical analyzes of the induced ashes, have allowed detecting elements comparable to those present in a clinker. In terms of rheological characteristics, the study of the effect of rate ash substitute in cement on yield shear stress (workability). The results obtained from this study show that it is necessary to use a plasticizer or to improve the workability of the cement paste of the ecological cements studied. A rate proportional to the rate of the substituted ash in the cement. A good correlation between yield shear stress and slump spread diameter can be observed especially over the low yield shear stress values.
In terms of mechanical performances of samples made of standard as ecological cements developed, it appears from this study that the compressive strengths obtained at different curing times, for mortars including the eco-cements are higher than the measured values on specimens containing standard cement. It is to note that after 28 days of curing, the resistances of samples made with eco-cements exceed 50 MPa.

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