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INCORPORATION OF SLUDGE FROM A WATER TREATMENT PLANT IN CEMENT MORTARS

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Abstract

The characterization of the sludge from a Portuguese water treatment plant is presented. Dewatered sludge contains basically water, calcium, aluminium and silica, with an organic matter content of about 6%. The mineralogical constituents in the heated sludge are mainly calcite and clay minerals. Gibbsite is present only in the sludge heated at 105ºC and calcium aluminate and calcium oxide are only present in the sample heated at 700ºC. Thermal treatment does not remove the entire polymer and coagulant initially within the sludge. Sludges dewatered or heated at 105ºC inhibit the setting and hardening of the paste. Incorporation of sludge in mortars can only be feasible after treatment of the sludge at not less then 450ºC even thus with an increase of the initial setting time and a decrease of the mechanical strength.

Key words: Sludge, leachates, wastes, cement mortars, cement, pastes, chemical characterization, mineralogical characterization and infrared characterization.

1. INTRODUCTION

The effort that is being made in Portugal to fulfil the goal of serving the country with 95% by drinking water until 2006 will result in an increase of the quantities of drinking water sludge requiring disposal. In 2002 the drinking water sludge production in Águas de Portugal Group was about 16 500 tones of dewatered sludge.

This increase is mainly due to the rise in the number of households served with potable water as well as to the practical implementation of the Drinking Water Directive (98/83/EC).

According to the European list of wastes, drinking water sludge is an industrial waste of the following category: 19 09 “Wastes from the preparation of water intended for human consumption or water for industrial use”.

The Community's approach to waste management policy, established on the Waste Framework Directive 75/442/EEC (amended by Directive 91/156/EEC, by Directive 91/692/EEC and by Commission Decision 96/530/EC) as well as on the Sixth Environment Action Programme "Environment 2010: our future, our choice", is based on the guiding principle of the waste hierarchy which gives preference to waste prevention, then to waste recycling and reuse and lastly to waste disposal.

Because sludge prevention is difficult to achieve, it must be given preference to sludge recycling and reuse, provided they are technical and economical feasible.

Land filling of sludge must be done only when there is no other solution available. Council Directive 99/31/EC of 26th April 1999, on the landfill of waste, entered into force on 16 July 1999 and in Portugal this Directive was implemented by the Decree-Law 152/2002, of 23rd April 2002 [1]. This Decree-Law establishes different categories of landfills, according to waste type (hazardous waste, non-hazardous waste and inert waste). The acceptance criteria for waste disposal in the different categories of landfills are established on table 2 (value limits for waste) and table 3 (value limits for the leachate) of Annex III.

Sludge production depends on several features, such as water quality, chemicals used on water treatment, and treatment efficiency, in particular sludge dewatering processes. Moreover, sludge production is quite different in result of water quality variation with the seasons of the year.

Many studies have been made about applicability of sludge from wastewater treatment plants but very few related with sludge from water treatment plants [2].

This paper presents the results of the study that have been made about the incorporation of the sludge from a Portuguese water treatment plant in cement mortars [3].

2. CHARACTERIZATION OF THE SLUDGE

During the process of producing potable water some commercial products are added to natural water in order to assure it quality for human consumption. In the present case study the products added were calcium hydroxide, a polymer and a coagulant agent.

Sludge characterization involved the determination of its chemical and mineralogical composition and the identification of the organic products that could prevent the setting of the mortar. Also the pollution potential of the sludge was determined by characterization of the leachate composition.

It was determined the content of the major elements like silicon, aluminium, iron, calcium, magnesium, sodium, potassium, sulphate, and the content of minor elements like zinc, cadmium, copper, lead, nickel and chromium.

The leachate characterization included analysis of pH, conductivity and concentrations of zinc, cadmium, copper, lead, nickel and chromium.

Some portions of the sludge were submitted to heat treatment at temperatures between 105 ºC and 850ºC, to quantify the water content and to estimate the content of organic matter that was identified by infrared (IR) spectra.

2.1. Chemical composition

The sludge previous dried at 105ºC was melted with lithium tetra borate and the mixture dissolved with hydrochloric acid. Table 1 shows the results of the content of the major elements corresponding to the average of a duplicate analysis. The results are presented considering the sample on a calcined basis.

Table 1: Results, in percentage, for major elements

Determinations	Results (%)
Sílicon oxide, SiO_2	22,3
Aluminum oxide, Al_2O_3	26,0
Iron oxide, Fe_2O_3	4,7
Calcium oxide, CaO	38,3
Magnesium oxide, MgO	2,7
Sodium oxide, Na_2O	0,5
Potassium oxide, K_2O	1,0
Sulphates, SO_4^{2-}	0,7

Table 2 presents the results of the minor elements in sludges, as well as the limits for waste that can be deposit in inert waste landfills as are indicated on table 2 of the Annex III of the Portuguese law DL nº152/2002.

Table 2: Results, in percentage, for minor elements

Determinations	Results	DL nº 152/2002
Zinc, Zn (mg/kg)	225	8000
Lead, Pb (mg/kg)	<45,0	2000
Copper, Cu (mg/kg)	195	6000
Cadmium, Cd (mg/kg)	<5,0	50
Nickel, Ni (mg/kg)	53,0	2000
Chromium, Cr (mg/kg)	67,0	3000

Sludge water content was determined from the mass loss at 105ºC, and the organic matter content estimated from the difference between the results at 105ºC and 450ºC. Highest temperatures were tested only to verify the presence of some organic matter or refractory products. The results are presented in Table 3.

Table 3: Loss of mass of the sludge, in percentage, at different temperatures

Temperature (ºC)	Loss of mass of the sludge (%)
105	75
350	79
450	81
550	81
700	84
850	85
970	85

The results show that dewatered sludge contains 75% of water, about 6 % of organic matter, and about 4% of refractory compounds.

2.2. Mineralogical characterization

The X ray diffraction analyses of sludge samples heated at 105ºC, 450ºC and 700ºC (fig 1) were done to identify the constituents and their possible changes originated by the thermal treatment. The main constituents present in these samples are calcite and clay minerals. Gibbsite ($Al(OH)_3$) is present only in the sample heated at 105ºC and calcium aluminate (C_3A) and calcium oxide (CaO) are only present in the sample heated at 700ºC.

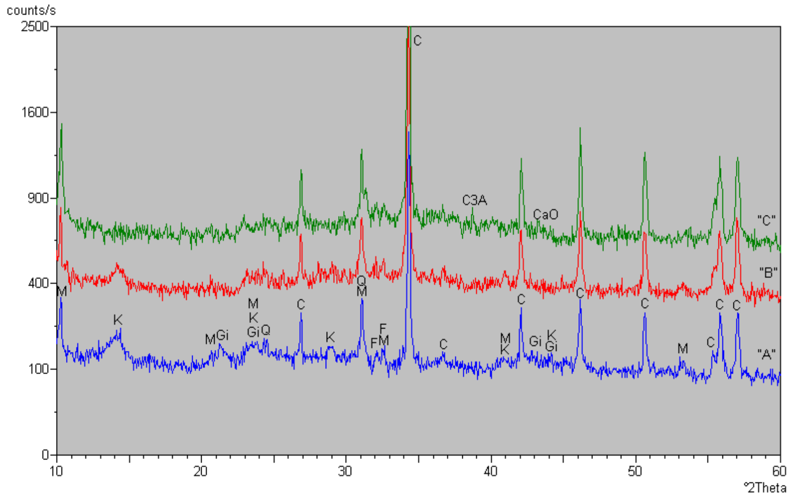

Figure 1 – X-Ray diffraction of sludges heated at (A) 105ºC; (B) 450ºC and (C) 700ºC.

2.3. Infrared characterization

Sludge and the commercial products used in the treatment of water were analysed by infrared spectrometry (IR). Hydrated lime has a pick at 3650 cm-1 and the polymer and the coagulant agent are quite similar presenting both two bands at 3400cm-1 and 1650cm-1(fig 2).

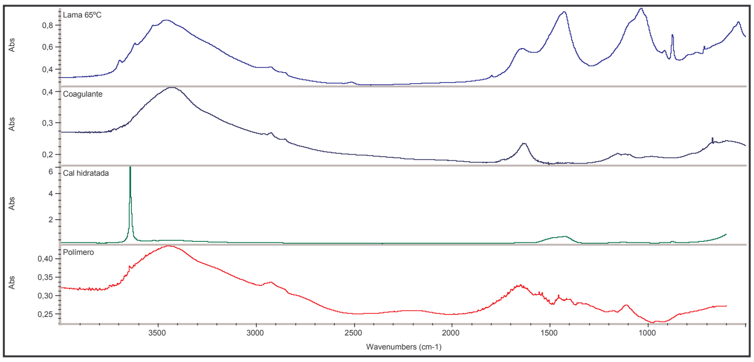

Figure 2 – Infrared spectra of the polymer (red), hydrated lime (green), coagulant agent (blue) and sludge treated at 65ºC (turquoise) [4]

The IR spectra of the sludges (fig 3) revels that the thermal treatment at 700ºC does not remove the entire polymer and coagulant initially within the sludge [4].

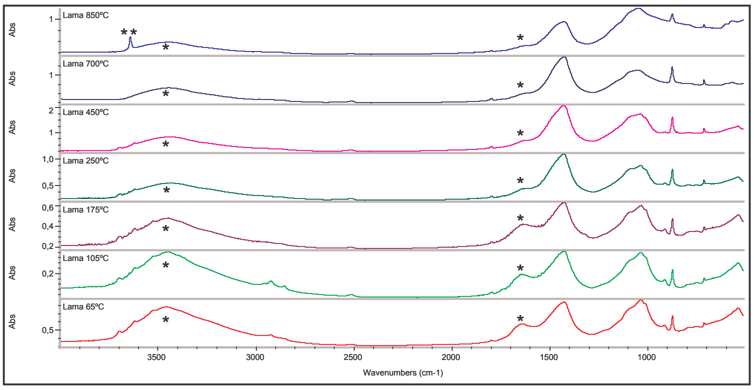

Figure3 - Infrared spectra of sludges treated at different temperatures from 65ºC (bottom) until 850ºC [4]

2.4. Leachates characterization

The leaching test was performed according to the German standard DIN 38414/4 Part 4 [5] using one extraction only. The concentration of the elements in the leachates was done by Inductively Coupled Plasma Spectrometry (ICP), being the result the average of two replicates.

Table 4 presents the results obtained from the leachate characterization and the limits established in table 3 of the Annex III from DL nº 152/2002 [1] to compare the results.

Table 4: Results of the characterization of sludge's leachate.

Determinations	Results	DL nº 152/2002
pH	9,93	5,5 to 12
Conductivity, 25ºC ($mScm^{-1}$)	0,99	6 to 50
Zinc, Zn (mg/l)	<0,02	2
Lead, Pb (mg/l)	<0,09	0,5
Copper, Cu (mg/l)	1,2	2
Cadmium, Cd (mg/l)	<0,01	0,1
Nickel, Ni (mg/l)	0,25	0,5
Chromium, Cr (mg/l)	<0,06	0,5

Table 4 shows that the concentrations of pollutants are lower than the limit established on the Portuguese Decree.

3. PASTES AND CEMENT MORTARS WITH SLUDGE

A set of preliminary tests was done with cement pastes and mortars exchanging 5%, 10% and 20% of the mass of cement by dewatered sludge and by sludge after thermal treatment at 105ºC.

Tests were carried out also with treated sludge at higher temperatures of 450ºC, 700ºC and 850ºC as described below.

3.1. Sludge treated at 105ºC

The standard mortar was prepared with the cement:sand:water ratio of 1:3:0,5 using natural siliceous sand. The mortars were prepared exchanging 20% of the mass of cement by sludge as it was received (containing 75% of water) and previously dried at 105ºC until constant mass (Table 5). During the heat treatment at 105ºC sludge suffered agglomeration and it was necessary to grind it before the tests. The quantity of water added was only to keep the flow consistency constant.

Table 5: Composition and flow of mortars in the preliminary tests

Component	Standard mortar	Test mortar with 20% sludge	
		Original sludge with 75% of water	Sludge heated at 105ºC
Cement–I 42,5 R (g)	450	360	360
Sand (g)	1350	1350	1350
Sludges (g)	0	153	90
Water (ml)	225	192	255
Flow (mm)	207	187	218

For each mortar, three specimens with (4x4x16) cm^3 were prepared. The specimens did not presented any setting after several days. These results were confirmed by the tests of setting time in pastes with the same composition of the binder.

3.2. Sludge treated at 450ºC, 700ºC and 850ºC

With the aim of verify the influence of the organic matter in the setting time of the mortar, three portions of 1000g each of sludge treated at 450ºC, 700ºC and 850ºC were tested.

Standard mortar was prepared with a cement:sand:water ratio of 1:3:0,5 using CEN standard sand and test mortars with 5% and 10% of the cement replaced by sludge heated at 450ºC; 700ºC and 850ºC. Table 6 presents the composition of the mortars prepared.

Table 6: Composition of the mortars with sludge heated at 450ºC, 700ºC and 850ºC

Component	Standard mortar	Mortars with 5% sludge	Mortars with 10% sludge	Mortar with 10% sludge*
		All temperatures	All temperatures	700ºC
Cement-I 42,5 R (g)	450	427,5	405	405
Sand (g)	1350	1350	1350	1350
Sludges (g)	0	22,5	45	45
Water (ml)	225	225	225	252
Flow (mm)	247	nd	nd	242

nd- not determined

* Mortar prepared with addition of water to achieve the standard mortar flow.

For each mortar, three specimens with (4x4x16) cm^3 were prepared to determine the mechanical strength at the age of 7 days according to the standard NP EN 196-1 [6].

Table 7: Mechanical strength of the mortars with sludges at the age of 7 days

Mortars	Compressive strength (MPa)
10% sludge at 450ºC	40,4
5% sludge at 700ºC	42,0
10% sludge at 700ºC	39,7
10% sludge at 700ºC*	36,2
10% sludge at 850ºC	37,9
Standard mortar	46,4

*Mortar prepared with addition of water to achieve the standard mortar flow.

The results on Table 7 show that introduction of any kind of treated sludge leads to a decrease of the compressive strength of the mortars. When the content of sludge is was changed it was verified that the compressive strength decreases with the increase of sludge content and also with the increase of the temperature treatment.

As it was expected, the mortar with 10% of sludge at 700ºC and with supplementary water addition, presents the lowest mechanical strength.

Considering the results of the setting time of the pastes in Table 8 determined according to NP EN 196-3 [7], the introduction of sludge in cement pastes requires a supplement of water of about 7,5% when we use 5% of sludge and a supplement of water of about 21,5% when we use 10% of sludge. Considering the content of sludge of 5% the setting time decrease from 185min. to 145min., as we use sludge treated at temperature at 450ºC or at

850ºC. If we added 10% of sludge the setting time decreases from 255min. to 140min. However the setting time is slightly lower for sludge treated at 700ºC and 850ºC. So, an increase of temperature of treatment corresponds to a decrease of the setting time and the setting time of the paste with 10% of sludge submitted to 850ºC has the same value of the standard paste.

Table 8: Setting time of the pastes with sludge after heat treatment

Paste	Water of the paste (%)	Setting time (min)
Standard	28,8	140
5% sludge at 450ºC	30,8	185
10% sludge at 450ºC	35,6	255
5% sludge at 700ºC	31,6	165
10% sludge at 700ºC	34,4	160
5% sludge at 850ºC	31,6	145
10% sludge at 850ºC	35,4	140

It was also tried to determine the setting time of a paste with a content of 20% of sludges treated at 700ºC. However, this test was not feasible because five minutes after mixing, the paste hardened very quickly with a temperature rise until 30ºC.

The results of setting time could be justified with the rise content of lime and calcium aluminates that are formed as the temperature of sludge treatment increase. The formation of lime is also believed to decrease the compressive strength of the mortars.

4. CONCLUSIONS

- Dewatered sludge contains basically water, calcium, aluminium and silica, with an organic matter content of about 6%;
- Leachate of the sludge is alkaline and few mineralised, and the zinc, lead, cadmium, nickel, copper and chromium concentrations, are lower than the limits established in the Portuguese Decree for inert wastes;
- The mineralogical constituents present in sludge heated at 105ºC, 450ºC and 700ºC are mainly calcite and clay minerals. Gibbsite is present only in the sample heated at 105ºC and calcium aluminate and calcium oxide are only present in the sample heated at 700ºC;
- Sludge dried at 105ºC or at higher temperatures agglomerates, so it has to be grinded before it can be used in cement mortar;
- Sludge dewatered or heated at 105ºC inhibit the setting and hardening of the paste and mortar, and so they should not be incorporated in cement mortars;
- In sludge treated at temperatures of 700ºC occurs the formation of lime and calcium aluminates, which might be the cause for the observed decrease of the initial setting time;
- The incorporation of thermally treated sludge in mortars promotes the increase of the consistence and consequently a supplementary water need to keep the same workability;
- The incorporation of sludge in mortars it can only be feasible after treatment at not less then 450ºC leading to an increase of the initial setting time but a decrease of the mechanical strength;

- Tests with mortar sludge heated at 450ºC are foreseen to verify the results of mechanical strength at later ages.

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