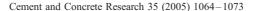


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Durability of concrete with addition of dry sludge from waste water treatment plants

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Abstract

This paper evaluates the use of dry sludge as an additive for concrete, for which it must be guaranteed that the resulting concrete has the appropriate mechanical strength and durability.

In earlier work on the subject, it was shown that the addition of sludge reduces the mechanical strength of concrete. With the addition of 10% sludge in proportion to the amount of concrete, the mechanical strength decreases significantly, making it unsuitable for medium-to high-strength reinforced concrete.

One possible area of application would be in the preparation of low-strength mass concrete that could be used for bases and subbases of roads with light traffic, as filler, etc.

We subjected the concrete specimens to different types of accelerated attack in order to evaluate long-term performance and compare them with the reference concrete (not containing sludge).

The following tests were made:

- Combined wet-dry cycles using fresh water, seawater and water containing 5% sulphates
- Accelerated ageing in an autoclave
- Accelerated carbonation

The performance of the concrete containing sludge was acceptable and comparable to the results obtained for the reference concrete not containing sludge.

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Keywords: Durability; Stability; Sulphates attack; Waste management; Carbonation

1. Introduction and objectives

This study aims to evaluate the addition of dry sludge to mass concrete. The sludge used is a dry sludge from Sabadell (Barcelona) produced by anaerobic biological digestion. In the final phase of the waste treatment process, it undergoes a thermal drying process, which substantially reduces its volume, thus facilitating handling and eliminating any pathogenic microorganisms that could be present. But, the sludge had a residual humidity (about 15%), the dried industrial process was not complete.

Addition of sewage sludge to concrete slows the curing process and reduces the mechanical strength of the concrete, particularly in the short term [1,2]. In addition to these changes, we wished to determine whether the addition of sludge alters the durability of the concrete.

In order to do this, we subjected the concrete to various types of accelerated aggression to predict its long-term performance.

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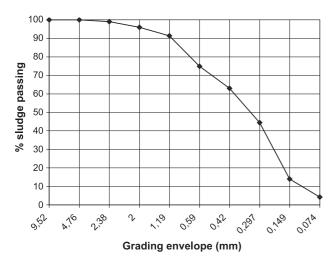


Fig. 1. Dry sludge granulometry from the Sabadell (Barcelona) treatment plant.

The specimens were subjected to the following trials:

- Combined wet–dry cycles using fresh water, seawater and water containing 5% sulphates
- Accelerated ageing in an autoclave
- Accelerated carbonation

Following these tests, we studied variations in saturated weight with dry surface and hydrostatic weight after each wet–dry cycle, and the mechanical strength of the specimens at the end of the tests.

We also studied the development of the by-products of hydration in concrete with and without sludge by means of X-ray diffraction.

2. Characterization of dry sludge

2.1. Physical characterization

The granulometric curve of the sludge is equivalent to that of fine sand (see Fig. 1). The dry sludge is added to the concrete as an additive mixed with the cement.

2.2. Chemical characterization

The dry sludge has a high content of organic matter, a neutral pH and relatively low concentrations of metal ions, with the exception of nickel [3], whose content is slightly above maximum permitted levels (see Tables 1, 2 and 3).

Table 1 Sabadell dry sludge characteristics

рН	7.08
Organic matter—500 °C	41.5-52%

Table 2 Main elements of Sabadell dry sludge determined by XRF

	,	-	
Na ₂ O	$1.11 \pm 0.07\%$	CaO	22.7±0.2%
MgO	$2.73 \pm 0.08\%$	Cr_2O_3	$0.24 \pm 0.02\%$
Al_2O_3	$12.9 \pm 0.2\%$	Fe_2O_3	$10.1 \pm 0.1\%$
SiO_2	$29.7 \pm 0.2\%$	NiO	$0.13 \pm 0.010\%$
P_2O_5	$12.4 \pm 0.2\%$	CuO	$0.23 \pm 0.02\%$
SO_3	$3.22 \pm 0.08\%$	ZnO	$0.84 \pm 0.04\%$
C1	$0.20 \pm 0.01\%$	SrO	$0.29 \pm 0.02\%$
K_2O	$1.83 \pm 0.06\%$	ZrO_2	$0.16 \pm 0.01\%$

2.3. Mineralogical characterization

X-ray diffraction was used to determine the main types of crystalline minerals present. Significant amounts of only quartz and calcite were detected. No clays were detected (see Fig. 2).

3. Method

We prepared an HM-25/B/20/1 type concrete, in accordance with Spanish standard UNE 83-301 [4] and UNE 83-313 [5], this corresponds to a mass concrete of 25 MPa with characteristic strength, a soft consistency and a maximum aggregate size of 20 mm for low-aggression type-I surroundings.

Since the aim of this study does not involve attaining high performance from this concrete through addition of sludge, we used a CEMII/A-L 32.5 R cement, in accordance with European standard EN [6], see Table 4.

The dried industrial process was not complete, then this dry sludge containing a small moisture content of the sludge allowed us to add fines to the concrete that do not create a demand for water, thus maintaining a soft consistency for the concrete, which did not require a higher proportion of liquid water and had the same consistency as the reference concrete (see Table 5). This characteristic of the sludge constitutes an advantage for its practical use, since the technological processes relating to the concrete remain unchanged.

3.1. Tests

The following tests were carried out:

- Wet–dry cycles in fresh water, seawater and water containing 5% K₂SO₄
- Accelerated ageing cycles in an autoclave
- Accelerated carbonation

Table 3 Heavy metals concentration in mg/l by standard DIN 38414-S4 [9] of Sabadell dry sludge

Ba	0.75 ± 0.02	Mn	0.23 ± 0.01
Zn	1.23 ± 0.06	Cd	< 0.01
Ni	1.058 ± 0.003	Cr	0.05 ± 0.01
Pb	< 0.05	As	< 0.1
Cu	0.31 ± 0.07		

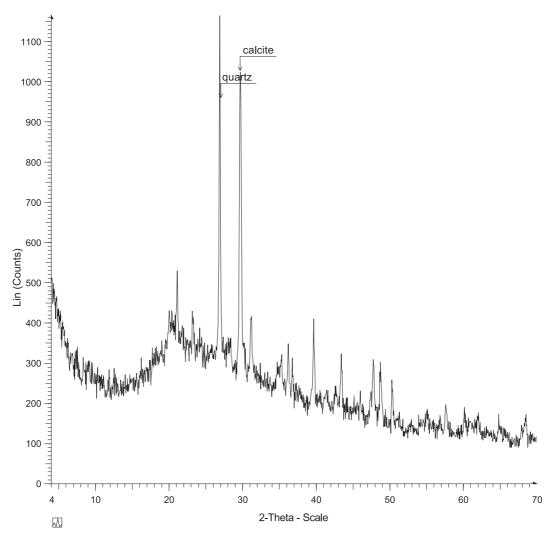


Fig. 2. XRD dry sludge, without grading.

3.1.1. Wet-dry cycles

The wet-dry cycles were carried out in fresh water, seawater and a 5% solution of K₂SO₄.

We chose to carry out wet-dry cycles rather than having the specimens submerged constantly, thereby accelerating the potential chemical reactions and intensifying the changes in the specimens.

The same procedure was followed with the three different solutions and consisted of 7 cycles of 28 days

Table 4 Dosage concrete (HM-25/B/20/I)

Mass concrete-25/B/20/I		
Material	1 m ³ concrete	
Sand 0/5	847 kg	
Sand 0/2	200 kg	
Melcret PF-75 (superplastified additive)	1.68 1	
Water	160 1	
Aggregate 5/12	85 kg	
Aggregate 12/18	816 kg	
Cement II/32.5-R	287 kg	

each, in the following pattern: 28 days after manufacture, the specimens were removed from the moisture chamber (21 °C and 95% humidity) and allowed to dry for 2 days in the laboratory. They were then submerged in containers with the appropriate type of solution and left for 5 days. After 5 days, they were removed from the containers and left to dry for 2 days in the laboratory and then submerged in the solutions again for another 5 days. After the second submersion, they were left to dry for 2 days and then submerged in the solutions again for 12 days.

We left the specimens in this last submersion for 12 days to ensure that they were saturated, so that we could

Table 5 Slump according sludge percentages

% Sludge	Slump (cm)
0%	10
2.5%	12
5%	13
10%	13

determine their saturated weight with a dry surface and their hydrostatic weight after each cycle.

These cycles were repeated seven times, after which the specimens were broken under simple compression.

4. Results

4.1. Saturated weight with dry surface and hydrostatic weight

Hydrostatic weight and saturated weight with dry surface both increased in the specimens submerged in the sulphate solution [7], and this increase was greater with a greater number of cycles and a higher proportion of added sludge.

In the specimens submerged in seawater, the results were similar, but the weight increases were not as great as in the case of the sulphate solutions.

The weight variations in fresh water were less substantial and tended to diminish with each successive cycle. This decrease was more substantial with a higher proportion of sludge and a greater number cycles.

The differences observed can be attributed to two types of process: (1) the hydration of the cement that had not reacted is completed; (2) the sulphates present in this type of solution react with the C_3A and become fixed, increasing the weight of the specimen through formation of ettringite, and the salts present in the solution can crystallize in the pores of the concrete. All three of these circumstances give rise to increased weight.

In addition, the ettringite formed in this way and the salts that crystallize in the pores of the concrete reduce its porosity.

Also, soluble components are subject to leaching processes, giving rise to weight loss in the specimens. This weight loss was greater in the specimens submerged in fresh water and, in the long term, the weight lost through leaching was greater than the potential weight increases owing to hydration reactions. It is important to say that in other study we saw that the environmental impact attributable to the addition of the sludge is negligible. The concentrations of the heavy metals in the leachate are so low that in most cases they were undetectable by the analysis technique [8].

Weight loss through leaching was greater in proportion to the sludge content and increased with the number of cycles carried out (see Fig. 3).

The apparent volume of the specimens can be determined from the values for hydrostatic weight and saturated weight with dry surface.

The decrease or increase of these magnitudes occurs concurrently and the apparent volume of the specimens remains practically unchanged.

Since the mass increases and the apparent volume remains unchanged, the density of the specimens increases, which would explain the increase in their mechanical strength.

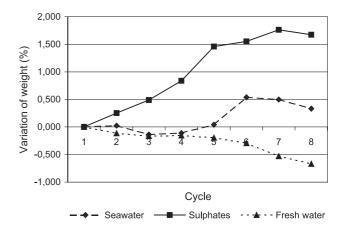


Fig. 3. Variation saturated weight with dry surface based on the attack solution.

4.2. Mechanical strength

After completion of the various wet-dry cycles, compressive strength test was carried out to perform a mechanical evaluation of the results of the treatments [9].

All the treatments increased the compressive strength of the concrete in comparison with the reference specimens, which remained in the moisture chamber throughout the experiment. This increase was greater in the specimens submerged in the sulphate solution.

In the case of the specimens not containing sludge, upon completion of the treatment after seven cycles the strengths of the reference specimens, the ones submerged in seawater and the ones submerged in fresh water were practically the same, with a substantial increase in the specimens submerged in the sulphate solution (see Fig. 4).

The specimens containing sludge and subjected to treatment cycles showed greater strengths than those left in the moisture chamber, the specimens submerged in the sulphate solution showing in all cases the greatest strength.

The poorest mechanical strengths were obtained consistently for the specimens with a 10% sludge content subjected to standard curing, which reached strengths of 20 MPa after seven months, with a 25% increase in this value for those treated with sulphates.

Since the treatment time and acceleration of aggression mechanisms were both relatively high, we can assume improved performance particularly for the specimens treated with sulphates, though to a lesser extent for the other treatments, although we cannot be sure that problems with excessive expansion will not arise in the long run.

4.3. Accelerated ageing in autoclave

One of the aims of this study was to determine whether the addition of sludge to concrete gives rise to changes in size, and we therefore prepared specimens for trials using an autoclave.

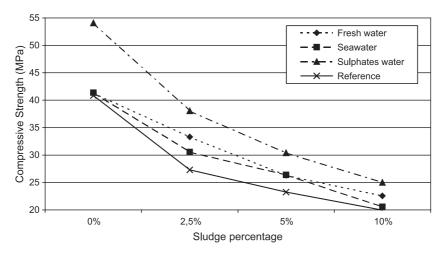


Fig. 4. Compare of compressive strength according to its treatment and sludge percentage.

After 28 days of curing in the moisture chamber, we subjected the specimens to heating in an autoclave at a pressure of 0.2 MPa.

We measured one of the dimensions of the specimens after curing for 28 days and then soaked them in water for 48 h before placing them in the autoclave for 4 h. After heating, we left the specimens to cool for 20 h and then measured the dimensional change using a comparator with an accuracy of 0.01 mm.

The procedure was repeated three times with measurement of the same dimension of the specimen.

Shrinkage was observed in all cases, the difference being very small in the concrete not containing sludge or with only 2.5% sludge and increasing in proportion to the sludge content (see Fig. 5).

Shrinkage of the specimens was basically plastic and occurred in the phase of transition from fresh concrete to hardened concrete during the initial hours. After 28 days'

curing in the moisture chamber, the shrinkage was minimal.

In spite of the drastic conditions of the test, no expansion of the concrete was observed, allowing us to assume that under normal conditions of use these concretes containing sludge would not cause any significant problems due to change in volume.

4.4. Accelerated carbonation

Carbonation is a normal process in concrete consisting in the reaction of atmospheric carbon dioxide with the calcium hydroxide that is produced in the hydration of Portland cement, to form calcium carbonate.

This reaction has negative consequences for reinforced and pre-stressed concretes, since it lowers the pH of the concrete and reduces the alkaline protection of the reinforcing steel, allowing it to oxidize.

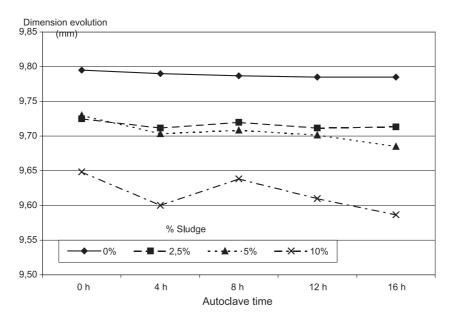


Fig. 5. Dimension evolution of specimens using a comparator.



Fig. 6. Image carbonation equipment.

For mass concretes, this process is positive, since it reduces the proportion of calcium hydroxide, which is somewhat soluble, and replaces it with calcium carbonate, which fills the pores and is relatively insoluble.

Since the carbonation process is slow, we accelerated it by using a mixture of 80% air and 20% carbon dioxide (the normal atmospheric concentration of carbon dioxide is approximately 0.03%).

After 28 days in the moisture chamber, we placed the specimens in a plastic container in which we had made two openings on opposite sides with stopcocks to control the entry and exit of a carbon dioxide-rich mixture, and we closed and sealed the cap so that only the carbon dioxide-rich mixture could enter.

We opened both stopcocks to allow circulation of the mixture for 3 min to replace the air in the containers. At this point, we closed both stopcocks and the specimens were therefore immersed in a carbon dioxide-rich atmosphere.

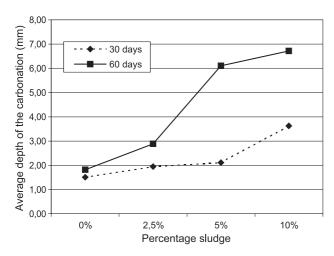


Fig. 7. Depth of penetration of the carbonation front based on the percentage of sludge after 30 and 60 days.

Table 6
Area noncarbonate and depth of penetration of the carbonation

% Sludge	Time (days)	Area noncarbonated (mm²)	Depth of carbonation (mm)
0%	30	9407	1.51
	60 (1)	9341	1.68
	60 (2)	9235	1.95
2.5%	30	9238	1.94
	60 (1)	8920	2.78
	60 (2)	8840	2.99
5%	30	9175	2.11
	60 (1)	7729	6.04
	60 (2)	7685	6.17
10%	30	8602	3.63
	60 (1)	7643	6.29
	60 (2)	7345	7.15

We repeated this process every 3 days to attain an atmosphere with a relatively constant concentration of carbon dioxide.

After 30 and 60 days of immersion in this atmosphere (more or less 60% humidity and a mixers of 20% CO₂ and 80% O₂), we removed the samples and broke them using steel bars under splitting tension, and we painted the surfaces of the breaks with a pH indicator, phenolphthalein (see Figs. 6 and 7 and Table 6).

The carbonated areas have a lower pH. Under these conditions, the phenolphthalein is colourless and the colour observed is that of the concrete itself. The totally or partially uncarbonated areas have a higher pH, and here the pinkish colour of the indicator is noted.

Comparison of the colourless and coloured areas allowed us to determine the rate and depth of the advance of carbonation in the samples.

In the specimens submitted to the same test conditions, the depth of carbonation depended on the porosity of the concrete, see Fig. 8.

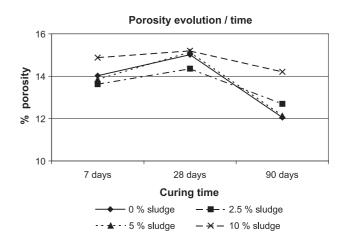


Fig. 8. Porosity varied according to the sludge content with three different curing times.

As stated above, porosity increases with a higher concentration of sludge. The carbonation test confirms this, since the carbonation was highest in the specimens containing 10% sludge.

Carbonation also depends on exposure time and it was in fact higher in the specimens subjected to this type of atmosphere for 60 days.

4.5. X-ray diffraction

Lastly, we used X-ray diffraction to study the specimens subjected to wet–dry cycles with sulphates, seawater and fresh water. A comparative study of the diffractograms shows that the presence of sludge has no effect on the crystalline substances formed in the hydration of the

cement, and that the only noticeable difference is a slower rate for the hydration reactions, indicated by the presence of residual C₂S (Larnite) in the samples containing sludge (see Figs. 9, 10 and 11).

5. Conclusions

As demonstrated in previous work [8,10,11] and confirmed by this study, the addition of sludge reduces the mechanical strength of concrete, and this makes sludge an unsuitable additive for short-term high-strength concrete and for reinforced and pre-stressed concrete.

Having said this, from the standpoint of durability and response to the different types of aggression dealt with in

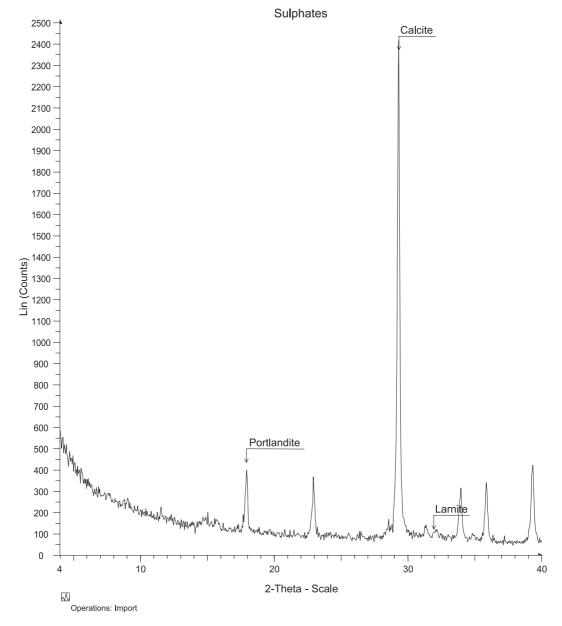


Fig. 9. XRD of sample with 10% sludge after sulphates attack.

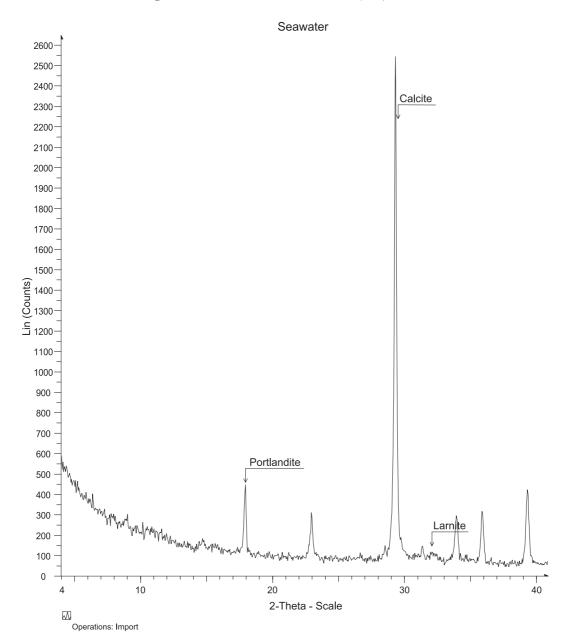


Fig. 10. XRD of sample with 10% sludge after seawater attack.

this paper, concrete containing sludge shows a performance similar to that of the reference concrete.

The residual moisture of the sludge allows it to be added to concrete without increasing the water requirement.

Of the three attacks by wet-dry cycles using fresh water, seawater and a sulphate solution, it was the latter that caused the greatest variation in the properties of the concrete, making it the one with the greatest increase in saturated weight with dry surface and hydrostatic weight. This is explained by the formation of ettringite and the crystallization of salts that are deposited in the pores of the concrete, filling them and reducing the porosity of the concrete.

These phenomena also provide an explanation of the substantial increase in the compression strength of the

specimens subjected to attacks in comparison with the specimens kept for the duration of the experiment in the moisture chamber, owing to the reduced porosity of the concrete.

Changes in the length of the specimens immersed in seawater and freshwater were substantially less notable than in the specimens immersed in the sulphate solution. In these cases, the leaching of components of the hydrated cement and the changes in the organic matter present in the sludge caused by the basic medium play an important role, giving rise to increased porosity.

The action of the cycles in an autoclave produced no significant changes in the concrete, with no expansion processes and no significant retraction. However, the shrinkage process occurs mainly during the first few hours

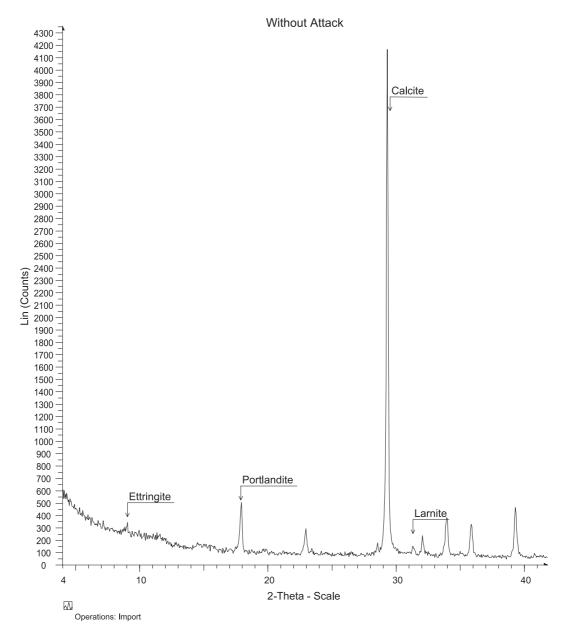


Fig. 11. XRD of sample with 10% sludge without attack.

and days, and in this study the treatment commenced when the specimens were already 28 days old.

This minimal shrinkage increases with the concentration of sludge. No expansion processes were detected.

The accelerated carbonation test shows that the porosity of the concrete increases with a higher concentration of sludge and that the concrete with the highest concentration of sludge showed the greatest penetration by the carbonation front. It was also observed that penetration by the carbonation front increased with the duration of the action of the carbon dioxide.

Study of the crystalline components of the concrete by X-ray diffraction shows that under these new conditions no crystalline substances other than those found in the reference samples are produced, and the only difference

noted is a slowing of the hydration process, shown by the presence of unhydrated C₂S in the concretes with higher concentrations of sludge.

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