Physicochemical properties of activated sludge ash as an additive in mortars

Christian Seal[1] and *Patrica* Mery[1*]

[1]Departamento de Ingeniería Civil en Obras Civiles, Universidad de Santiago de Chile, Avda. Ecuador 3659, Estación Central, Santiago, Chile

Abstract. Wasted activated sludge is a by-product of the treatment of wastewater, that historically been considered a waste and disposed in landfill. This has resulted in the search for alternatives in its disposal and possible reuses of these wastes. This study focuses on the use of activated sludge ashes from wastewater treatment plants as an additive in mortars and the impact on its mechanical resistance. For this purpose, it was determined the physical and chemical properties of the ashes and the mechanical properties of different percentage of ash content used in the fabrication of mortar prisms. The physical-chemical properties were centred in the percentage of ash versus temperature and the pozzolanic characteristics of the ash generated. Therefore, the determination of the ash percentage by thermogravimetric analysis was performed. Two temperatures were employed in the generation of ash (550 ºC and 750 ºC) and measured its pozzolanic characteristics by a rapid determination of pozzolanic activity of materials method. As a result, the ash generated at a higher temperature had a stronger pozzolanic characteristics, than the ash generated at the temperature used for determining volatile content of the sludge. Afterwards, sludge was calcinated in presence of oxygen at 750 ºC, and the ash was used in the manufacture of mortar prisms. Due to the variation in the size of the particles generated in the calcination processed, it was proceeded to separate them into two sizes (mesh sieve Nº 4 and Nº 100). The percentage by weight of the ash employed in the fabrication of the prism were 0%, 5, 10 and 15%. The prisms were tested for flexure and compression at 10, 21 and 28 days, and compared with a standard mortar. With regards of the consistency of the mortar, it was observed that the addition of ash had an impact on this property. The greatest loss of consistency was observed with the 15% of ash content and mesh sieve Nº 4, decreasing by 26%, while the consistency of the mortar with 15% of ash content and mesh sieve Nº 100 decreased up to 16.6%. With regards of the mechanical properties, it was observed that the addition of ash as an additive for mortar, observing an increase in flexural strength of up to 11% at 28 days for the mixture of 10% ash content and mesh sieve Nº 4. In the case of compression, the resistance after 21 days of curing, increased by 82% for additions of 10% and 15% of ashes content for the mesh sieve Nº 100. Therefore, it is possible to use ashes from wasted

* Corresponding author: patricia.mery@usach.cl

sludge of activated sludge wastewater treatment plant as an additive in mortars, and this can be employed as an alternative to disposal in landfills.

1 Introduction

In Chile wastewater treatment was introduce extensible in the beginnings of the 2000. With the treatment of wastewater, large quantities of wasted sludge are generated. Traditionally wasted activated sludge has been considered as a by-product and is discarded on non-agricultural land or landfilling. With the incorporation of more restrictive regulations and the constant increase in soil value, it is necessary to look for alternatives for its disposal [1]. As an alternative to the traditional methods of sludge disposal, energy recovery or incineration of the sludge can be used, and because of these processes activated sludge ash (ASA) is generated. Several studies have shown that ASA can be used as an additive in building materials such as cement-based materials [2-4]. Research has shown that the resulting ash that have high concentration of SiO_2 and Al_2O_3 [5], that could imply its pozzolanic characteristics.

It is also important to mention that the manufacturing of cement consumes large amounts of energy and generates considerable greenhouse gas emissions (GHG) into the environment [6]. As a result of the above, there is a need for the reduction of the emissions and develop process that produced lower emissions of GHG [7-8]. Therefore, the use of sludge ash as an additive in mortars or cement can lead to energy recovery and reduce the volume of sludge that goes to landfill. Additionally, the use of waste products that would otherwise be disposed in landfill, and used as biofuels for commercial or industrial use would provide a sustainable approach for minimizing waste and reducing environmental pollution [9].

Different studies have showed that temperatures between 550 to 950 ºC can activate clay minerals to obtain alumina and silica rich phases with partially disordered structures, which have pozzolanic reactivity [10-11]. Therefore, this work analyses the feasibility of using ash from wasted activated sludge as an additive for mortars as an alternative for its disposal. For this purpose, initially the pozzolanic characteristics were determine at two different calcination temperature and afterwards the mechanical properties for different ash mixture were measured.

2 Material and methods

2.1 Sludge sample and origins

This research examines the use of different concentration of ASA in mortar and its impact in the physical and mechanical characteristics by testing mortar prism. For this purpose, six sample of dried digested sludge from an activated sludge wastewater treatment facility were analysed. Each of the sample consisted of approximately 250 grams of wasted activated sludge from a sludge treatment centre. Four of the samples corresponded to sludge that was dried using bio-drying and the other two samples corresponded to solar drying beds. The solar drying method is used between October and April and the sludge is stacked in rows of 0.5 m of height that are periodically turn until the desired humidity is reached (40%).

In the case of the bio-drying, this method is employed during May to September, which corresponds to the colder month of the year. The stacks are composed of a mixture of the new sludge with solar dried sludge and a structural material (wood chips). The stacks are periodically turned for assuring aerobic conditions and to promote the drying process.

2.2 Physical sludge properties

The physical properties measured for each of the sludge samples where: the water content of the sample [12], ash content [13], and the resulting density of the ash generated at the two tested temperature [14]. The equipment employed for this analysis corresponded to a DHG 9053A drying oven, Kern Abj analytical balance, and a custom-made vacuumed chamber.

2.3 Thermogravimetric properties

The thermos gravimetric analysis was performed for a recently digested sludge sample. The equipment employed corresponded to a TA Instrument SDT Q600. The analysis was conducted under a nitrogen atmosphere with a flow rate of 50 mL/min. The temperature ranges between 50 to 900 ºC, and the temperature ramp corresponded to a constant ramp of 20 ºC/min. An alumina cup of 90 microliter capacity was employed and the sample weighted 8.2630 mg.

2.4 Measuring of the pozzolanic activity

The pozzolanic activity of the ash was determine by comparing the electro conductivity to a partial saturated solution of calcium hydroxide, using the methodology for rapid determination of Pozzolanic Activity of Materials [15]. The principle used in this method is adsorption and fixation of Ca^{2+} on the surface and matrix of the pozzolanic material [15]. The equipment used for measuring the electrical conductivity (EC) corresponded to a conductivity meter DSS-307, the solution was mixed in a 1 litter glass flask with a magnetic stirrer (Variomag Electronichüher and its regulator Telemodul 20P), and the pH meter corresponded to a Hanna HI9811-5. The calcium hydroxide was laboratory grade and the water used corresponded to distil and deionize water. It is important to note that the electro-conductivity of the solution is very temperature dependent, therefore the temperature during the experimental phase was maintained at 25 ºC.

2.5 Measuring of the mechanical properties

The class of cement used in the research was pozzolanic cement, common grade according to Chilean legislation [16]. The ASA employed in the determination of the mechanical properties was 750 ºC, which had a higher pozzolanic activity. The ash was classified into two sizes with the help of two mesh, sieves Nº 4 and Nº 100. The mix proportion of the mortar specimens can be seen in the following Table (Table 1). The dimensions of the prism employed were 40-40-160 mm, and the mould brand CONTROLS, model 65-L0010/A. With regards to the mechanical properties, the compressive strength [17] and flexure [18] of each of the mixture were determined at the following dates: 10, 21 and 28 days. The equipment employed in the determining the flexure strength corresponded to a SOILTEST, model CN 472 and for the compression a SOILTEST, model VERSATESTER. Additionally, the consistency of each mixture was determined by using a flow Table [19], for this purpose a flow Table CONTROLS, model L0040/A was employed.

Table 1. Mix proportion of mortar specimens.

Mortar Mix (Mesh size/Ash %)	**Cement (g)**	**Sand (g)**	**Water (ml)**	**Ash mesh Nº 4 (g)**	**Ash mesh Nº 100 (g)**
none/none	500	1500	305		
Nº 4 / 5%	500	1500	305	25	
Nº 4 / 10%	500	1500	305	50	
Nº 4 / 15%	500	1500	305	75	
Nº 100 / 5%	500	1500	305		25
Nº 100 / 10%	500	1500	305		50
Nº 100 / 15%	500	1500	305		75

3 Results

3.1 Physical sludge properties

The humidity and ash content from the different samples are shown in Table 2. There is a wide difference between the sample's humidity, the drying method doesn´t have a significant impact in the resulting humidity. With regards to percentage ash content it is possible to observe that higher temperature tends to generate lower ash content and the difference between both temperatures is 7.2%.

A representative sample from each ash temperature range performed, resulting in an average density 0.6071 g/ml for the ash sample generated at 550 ºC, and for the ash generated at 750 ºC an average density of 0.7167 g/ml. Therefore, at high temperature it is possible to expect an increase in the density of the resulting ash.

Table 2. Humidity and ash content of the sludge.

Sample	**Humidity %**	**Ash % at 550 ºC**	**Ash % at 750 ºC**
Bio-dying	4.6	61.6	53.2
Bio-drying	30.2	54.0	49.6
Bio-drying	19.13	63.8	56.1
Bio-drying	15.6	61.3	55.0
Solar drying	60.2	51.4	40.4
Solar drying	16.0	44.8	39.4
Average		56.1	48.9
SD		7.36	7.36

3.2 Determination of the pozzolanic activity

For this characteristic, the loss of electrical conductivity of the calcium hydroxide ($Ca(OH)_2$) solution with 20 grams of ash was measured. Initially a standard calcium hydroxide solution was generated, which had to be calibrated to obtain a concentration that would result in a EC in that did not have a measurement variation. Once the concentration for the standard calcium hydroxide solution was obtained, a 20 g. sample of each ash was mixed with the standardized solution. The difference in the EC was recorded over time for each sample until there was no variation of the EC. If the sample mix did not present a significant loss or its EC increases over time, it is considered as a non-pozzolanic material. The results of the analysis can be seen in Table 3. For the ash generated at 550 ºC, only one sample presented a significant loss of the EC. In contrast, the samples generated at 750 ºC all presented a notable loss of EC. Therefore, the experimental temperature selected for the mechanical properties was 750 ºC, due to that the ash presents a higher pozzolanic activity.

Table 3. Summary of the analysis for the determination of pozzolanic activity.

Sample	Average Variation of EC (mS/cm)	Standard deviation
550 ºC	-1133	2776
750 ºC	-5394	253.83

3.3 Thermogravimetric analysis

The thermogravimetric analysis allowed the analysis of the degradation process of the digested sludge while the temperature increased, for this purpose an inert atmosphere of nitrogen was employed avoiding the combustion of the sample. From the analysis it is possible to observe the curves for the mass loss (TG) and the degradation rate (DTG) (Fig. 1). It is possible to observed that between 170 and 550 ºC there is a significant mass loss of 37.14% of the sample. However, between 550 ºC and 720 ºC there is small loss of mass 4.75%. Therefore, the two temperatures that are employed in the generation of ash are 550 and 750º C.

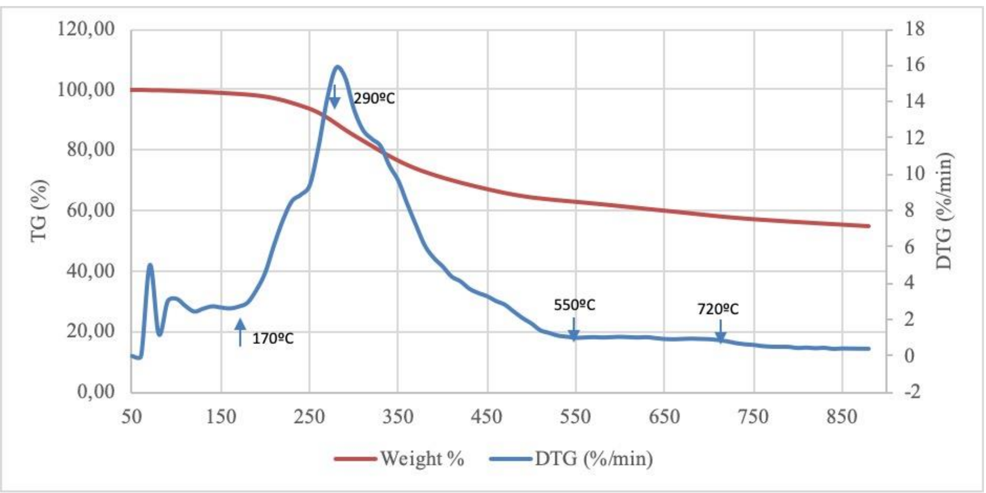

Fig. 1. Thermogravimetric analysis of sludge sample.

3.4 Mechanical properties

The mortar consistency was determined for each of the mixture three times (Table 4). It is possible to observe that an increase of the ash content in the mortar mixture has a significant impact in the consistency, for the ash sieve Nº 4 the consistency lowers between 11.7 to 31.4%, and for the sieve Nº 100 the variation is between 8% to 19.2%. Therefore, it is possible to conclude that the addition of ash impacts the mortar consistency, and in the case of size sieve Nº 4 decreases more the consistency than sieve Nº 100.

For the different mortar mixture, the mechanical properties of flexural and compression were measured at 10, 21 and 28 days. The number of samples employed in the flexural test were 3 (Table 5), and afterwards each side of the prism were tested for compression (Table 6).

Comparing mean between the standard mortar and the results for the ash from sieve Nº 4 for the flexural analysis, it was not possible to conclude that the addition of ash had a significant effect. In contrast the ash from sieve Nº 100 had a higher resistance than the standard mortar for the 10% and 15% ash content, 11.1% and 8.9% increase respectably.

With regard to the compression results, it is possible to conclude that the addition of ash increases the compression capacity of the mortar. Compering the average results for each day it is possible to observe that sieve Nº 100 tends to have a higher capacity than sieve Nº 4. The increase in the capacity for day 28 for sieve Nº 4 was 8.9% (10% ash content) and for sieve Nº 100 was 16.8% (10% ash).

Table 4. Consistency results.

Mortar Mix (Mesh size/Ash %)	**Average (mm)**	**SD (mm)**
none/none	213.7	14.3
Nº 4 / 5%	193.7	0.3
Nº 4 / 10%	176.0	13.0
Nº 4 / 15%	158.0	63.0
Nº 100 / 5%	202.0	4.0
Nº 100 / 10%	197.7	74.3
Nº 100 / 15%	178.3	4.3

Table 5. Results from the flexural analysis.

Mortar Mix (Mesh size/Ash %)	**Day 10**		**Day 21**		**Day 28**	
	Average (MPa)	SD (MPa)	Average (MPa)	SD (MPa)	Average (MPa)	SD (MPa)
none/none	0.565	0.4851	0.598	0.0265	0.794	0.2440
Nº 4 / 5%	0.620	0.0039	0.451	0.6938	0.963	0.0412
Nº 4 / 10%	0.656	0.0353			0.671	1.6101
Nº 4 / 15%	0.359	0.0755	0.678	0.0363	0.742	1.7591
Nº 100 / 5%	0.519	0.4988	0.653	0.0069	0.841	2.2912
Nº 100 / 10%	0.719	0.9202	0.847	1.1946	1.056	1.0682
Nº 100 / 15%	0.733	0.5527	0.866	0.1401	0.991	0.5449

Table 6. Results from the compression analysis.

Mortar Mix (Mesh size/Ash %)	**Day 10**		**Day 21**		**Day 28**	
	Average (MPa)	SD (MPa)	Average (MPa)	SD (MPa)	Average (MPa)	SD (MPa)
none/none	0.988	0.1968	1.174	0.1310	1.648	0.2724
Nº 4 / 5%	1.156	0.1491	1.369	0.1499	1.753	0.3050
Nº 4 / 10%	1.322	0.1781	1.485	0.1554	2.083	0.1893
Nº 4 / 15%	1.235	0.3160	1.313	0.1676	2.029	0.2435
Nº 100 / 5%	1.268	0.2274	1.512	0.1694	1.602	0.2298
Nº 100 / 10%	1.467	0.2256	2.138	0.2342	2.277	0.1828
Nº 100 / 15%	1.513	0.2185	2.019	0.2965	2.287	0.3929

4 Conclusion

The use of ash from digested activated sludge as an additive for mortars is a viable alternative to the traditional disposal of this waste. It was observed that the temperature has a significant impact in the pozzolanic activity of the sludge and due to its biological nature, there is high variability with respect to this characteristic.

With regards to the consistency of the mortar, the addition of ash hinders the fluidity but can be compensated with additives. It is possible to observe that the size impacts the consistency therefore smaller size particles are recommended. The use of ash sieve Nº 100 had a higher increased the mechanical properties for the tensile strength in bending. The difference in the flexural strength with respect to the standard mortar at 28 days between the two types of ashes is at least 16% and occurs when adding 10% of ash content.

The granulometry of the ash used in the confection of the mortar affects the resistance to compression. This can be seen for the ash sieve Nº 100 that produces a higher resistance to compression than sieve Nº 4. The maximum increase in the compression with respect to the standard mortar was 82.1% for the 10% ash content and sieve Nº 100 at day 21. Afterward this difference decreased to 38.2 percent at day 28.

References

1. C.J. Lynn, R.K. Dhir, G.S. Ghataora, R.P. West, Sewage sludge ash characteristics and potential for use in concrete. Constr. Build. Mat. **98**, 767-779 (2015). https://doi.org/10.1016/j.conbuildmat.2015.08.122
2. C. Gu, Y. Shuang, Y. Ji, H. Wei, Y. Yang, Y. Xu, R. Qian, D. Cui, H Zhou, Effect of environmental conditions on the volume deformation of cement mortars with sewage sludge ash. J. Build. Eng. **65**, 105720 (2023). https://doi.org/10.1016/j.jobe.2022.105720
3. T. Zdeb, T. Tracz, M. Adamczyk, Physical, mechanical properties and durability of cement mortars containing fly ash from the sewage sludge incineration process. J. Clean. Prod. **345**, 131055 (2022). https://doi.org/10.1016/j.jclepro.2022.131055
4. A.K. Prabhakar, P. Krishnan, S.S.-C. Lee, C.S. Lim, A. Dixit, B.C. Mohan, J.H. Teoh, S.D. Pang, D.C.W. Tsang, S.L.-M. Teo, C.-H. Wang, Sewage sludge ash-based mortar as construction material: mechanical studies, macrofouling, and marine toxicity. Sci. Total Environ. **824**, 153768 (2022). https://doi.org/10.1016/j.scitotenv.2022.153768
5. L.G.G. de Godoy A.B. Rohden, M.R. Garcez, E.B. da Costa, S. Da Dalt, J.J. de Oliveira Andrade, Valorization of water treatment sludge waste by application as supplementary cementitious material. Constr. Build. Mat. **223**, 939-950 (2019). https://doi.org/10.1016/j.conbuildmat.2019.07.333
6. W. Al-Kutti, A.B.M. Saiful Islam, M. Nasir, Potential use of date palm ash in cement-based materials. J. King Saud University – Eng. Sci. **31(1)**, 26-31 (2019). https://doi.org/10.1016/j.jksues.2017.01.004
7. S.A. Miller, V.M. John, S.A. Pacca, A. Horvath, Carbon dioxide reduction potential in the global cement industry by 2050. Cement and Concrete Research **114**, 115-125 (2018). https://doi.org/10.1016/j.cemconres.2017.08.026
8. E. Gartner, Industrially interesting approaches to “low-CO2” cements. Cement and Concrete Research **34**, 1489-1498 (2004). https://doi.org/10.1016/j.cemconres.2004.01.021
9. W.-H. Chen, B.-J. Lin, Y.-Y. Lin, Y.-S. Chu, A.T. Ubando, P.L. Show, H.C. Ong, J’-S. Chang, S.-H. Ho, A.B. Culaba, A. Pétrissans, M. Pétrissans, Progress in biomass torrefaction: Principles, applications and challenges. Progress in Energy and Combustion Science **82**, 100887 (2021). https://doi.org/10.1016/j.pecs.2020.100887
10. P.S.L. Souza, D.C.C. Dal Molin, Viability of using calcined clays, from industrial by products, as pozzolans of high reactivity. Cement and Concrete Research **35**, 1993-1998 (2005). https://doi.org/10.1016/j.cemconres.2005.04.012

11. A. Alujas, R. Fernández, R. Quintana, K.L. Scrivener, F. Martirena, Pozzolanic reactivity of low grade kaolinic clays: Influence of calcination temperature and impact of calcination products on OPC hydration. Applied Clay Science **108**, 94-101, (2015). https://doi.org/10.1016/j.clay.2015.01.028
12. INN, "NCh 1515:1979 Mecánica de suelo – Determinación de la humedad", (1979).
13. INN, "NCh 2281/3: 1995 Aditivos para hormigón – Método de ensayo – Parte 3: Determinación del contenido de cenizas", (1995).
14. INN, "NCh 2281/1: 1995 Aditivos para hormigón – Método de ensayo – Parte 1: Determinación de la densidad", (1995).
15. A. Dalinaidu, B.B. Das, D.N. Singh, Methodology for rapid determination of pozzolanic activity of materials. J. ASTM Int. **4(6)**, 1-11 (2007). https://doi.org/10.1520/JAI100343
16. INN "NCh 148: 1968 Terminología, clasificación y especificaciones generales". (1968)
17. INN "NCh 158: 2019 Ensayo de Compresión", (2019)
18. INN "NCh 158: 1967 Cementos – Ensayo de flexión y compresión de morteros de cementos", (1967).
19. INN "NCh 2257/1: 1996 Morteros – Determinación de la consistencia – Parte 1: Método del extendido en la mesa de sacudidas", (1996).