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Article

Supplementary Cementitious Material from Epsom Salt Production Waste

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Abstract

In this study, the potential use of Epsom salt production waste as a supplementary cementitious material was investigated. This acidic waste was neutralized with lime milk and used to replace up to 25 wt.% of Portland cement. The following research methods were employed: XRD, XRF, SEM, DSC-TG, and isothermal calorimetry. The waste neutralization process was found to proceed consistently, producing a neutral material (pH = 7.5) composed of amorphous silicon compounds with a negligible impurity of crystalline antigorite. Consequently, this material exhibits very high pozzolanic activity. The neutralized Epsom salt production waste accelerates the early hydration of Portland cement and promotes an intense pozzolanic reaction. This new material is a highly effective supplementary cementitious material, capable of replacing up to 25 wt.% of Portland cement without reducing its strength class.

Keywords: Epsom salt; waste; pozzolana; Portland cement; hydration

1. Introduction

Ordinary Portland cement (OPC) production accounts for nearly 7% of global CO₂ emissions, primarily due to the decomposition of limestone during the formation of clinker and the combustion of fuels, as well as the extensive use of natural raw materials [1]. Waste utilization – be it supplementary cementitious materials (SCM), incorporated into raw feed, or co-processed in rotary kilns - offers a means to reduce clinker costs, lower the carbon intensity of cement production, and promote circular economy practices [2–4]. However, the practical implementation of these strategies remains limited by several factors, including fluctuations in waste composition, the presence of harmful impurities such as chlorides and heavy metals, and other related challenges [1,5].

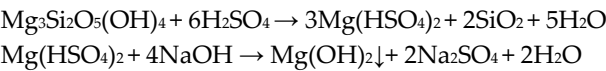
The main waste streams used include coal fly ash, ground granulated blast furnace slag, glass waste, municipal solid waste incineration ash, red mud, phosphogypsum and other industrial residues [2,3,6–8]. It is well established that many materials used as SCM enhance the mechanical strength and durability of Portland cement paste, as well as improve resistance to chloride ingress and sulfate attack, while reducing permeability [9–12]. These materials are employed not only for the beneficial properties they impart to the cement matrix, but also because large quantities of such waste are accumulated globally.

However, cement plants can use smaller quantities globally, but sufficient amounts of waste generated in local areas near the plants. This could be waste such as rice husk ash, spent catalytic cracking catalyst, construction and demolition waste, and other waste [13–15]. Additionally, small amounts of Portland cement can be replaced with synthetic materials from waste or inexpensive raw materials [16,17].

However, there are wastes from other industrial sectors that have been little studied. One of such waste is the Epsom salt production waste.

Epsom salt is usually produced by treating silicate minerals with a high concentration of magnesium, usually olivine [(Mg,Fe,Ni)₂SiO₄] or serpentinite [Mg₃Si₂O₅(OH)₄] with sulfuric acid

(H₂SO₄). The production of magnesium hydroxide from serpentinite is described by the following chemical reactions [18]:



This process produces a suspension with a high concentration of magnesium sulfate (MgSO₄) and contains solid residues, most of which are amorphous silica SiO₂ and other undissolved mineral fractions.

The production of MgSO₄ from magnesium silicates results in several primary waste streams, including a relatively Fe-rich sludge, a silica-rich residue and an acidic effluent. Upon neutralization with MgO or NaOH, Fe²⁺ is oxidized to Fe³⁺ and precipitated as Fe(OH)₃ or goethite (FeO(OH)) at pH 6-7. This sludge also contains traces of Ni, Cr, and other heavy metals. The undissolved silicate matrix, with its high silica content, is separated by filtration.

Resource utilization initiatives aim to convert waste into valuable products. Iron-rich sludge can be processed into magnetite (Fe₃O₄), which can be used in the production of iron and steel by magnetic separation, or as an adsorbent in wastewater treatment, effectively removing As, Cr and phosphates [19,20].

The situation with colloidal silica is somewhat different. First, colloidal silica complicates the filtration process and reduces the amount and purity of magnesium sulphate [18]. Second, due to its amorphous nature, silica adsorbs on the surface sulfuric acid and remains acidic, with a pH of 3-4. Due to this characteristic, this SiO₂-rich waste cannot be used directly in the cement or building materials industry. However, the various varieties of amorphous silica are a very valuable addition to Portland cement.

Sources of amorphous silica include natural materials such as volcanic ash, tripoli, opoka (natural pozzolans), industrial by-products such as silica fume, and synthetic compounds obtained from minerals such as olivine [21–23]. When incorporated as a supplementary cementitious material (SCM), amorphous silica exhibits pozzolanic behavior by reacting with calcium hydroxide produced during cement hydration, resulting in the formation of additional calcium silicate hydrate (C–S–H). This reaction enhances the mechanical strength and durability of concrete [24]. Therefore, the objective of this work is to determine the possibility of using silica-rich Epsom salt production waste as SCM and to investigate its influence on the characteristics of Portland cement.

2. Materials and Methods

Epsom salt production waste (ESW), collected during the production of Epsom salt, Portland cement CEM I 42.5 N (OPC), and CaO (purity 98.9 wt.%) was used. The chemical compositions of ESW and cement are presented in Table 1.

Table 1. Chemical composition of raw materials.

Component (wt.%)	ESW	OPC
SiO ₂	88.35	19.72
Al ₂ O ₃	0.10	4.93
Fe ₂ O ₃	2.47	3.25
CaO	0.48	61.19
MgO	2.64	3.93
K ₂ O	-	1.04
Na ₂ O	-	0.14
Cr ₂ O ₃	0.07	-
NiO	0.12	-
SO ₃	2.73	2.6
P ₂ O ₅	0.83	-
MnO	0.05	-
Other	2.16	3.2

XRD analysis data (Figure 1, (a)) show that ESW consists only of amorphous materials (hump at 15–40 2θ) with crystalline antigorite (PDF 04-015-5514) and magnetite (PDF 04-008-4511) impurities.

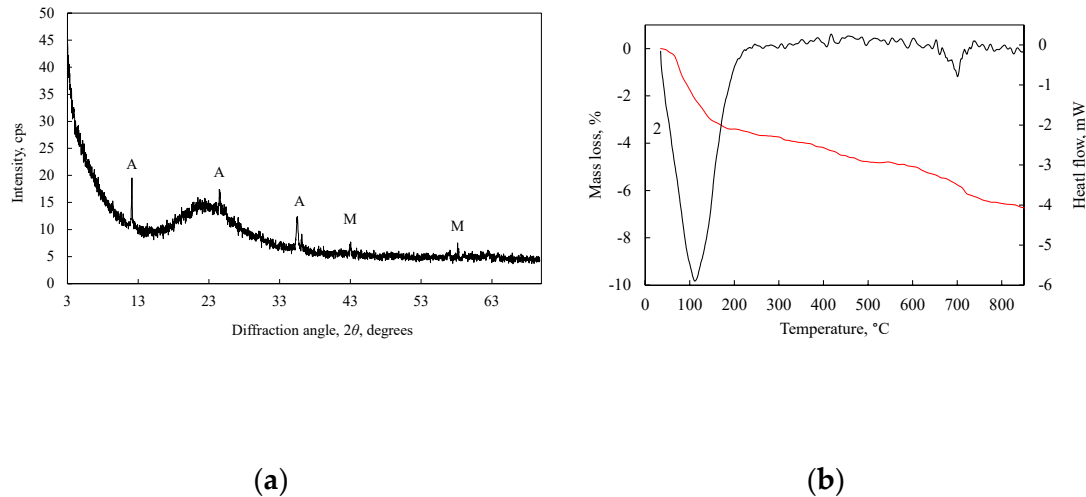


Figure 1. (a) – X-ray diffraction patterns of ESW, Indexes: A, antigorite ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$), M, magnetite (Fe_3O_4); (b) – Simultaneous thermal analysis (1 – TG, 2 – DSC) outcome of ESW.

In the DSC curve of the ESW sample (Figure 1(b)), an endothermic peak appears around 115 °C, corresponding to the release of physically adsorbed water. Another endothermic peak, observed near 700 °C, is associated with the thermal decomposition of antigorite [25]. The accompanying thermogravimetric (TG) analysis shows an overall weight loss of approximately 7.0 wt.%.

Because ESW has an irregular particle size distribution, it was milled to a specific surface area equal to 320 m^2/kg , with a mean particle diameter of 31.69 μm (Figure 2).

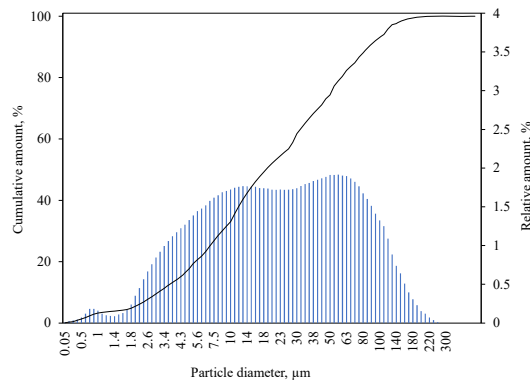


Figure 2. Particle size distribution of ESW.

SEM images show (Figure 3), that ESW consists of irregularly shaped sharp-edged particles. Meanwhile, the ground material also contains irregularly shaped but partially rounded particles.

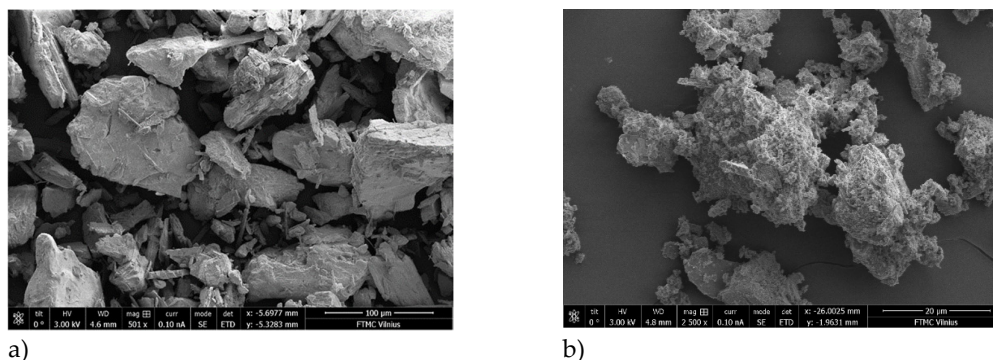


Figure 3. SEM images of ESW (a) – as received; (b) – additionally milled.

Neutralization of ESW. For the neutralization of acid rests, ESW was mixed with water in different water/solid material (W/S) proportions. Then the obtained waste pulp was transferred to a container with a 90 rpm propeller mixer. Later, lime milk is gradually dripped into this mixer. The process continues until the waste pulp is fully neutralized and the pulp pH = 7.5 is reached. The neutralized pulp is then filtered and dried in a dryer for 24 hours at 70 °C.

Lime milk preparation. Lime milk is prepared at a concentration of 3.5%, that is, 35 g. CaO was dissolved in a liter of deionized water.

Simultaneous thermal analysis (STA) was carried out using a PT1000 analyzer (Linseis, Germany) over a temperature range of 30–1000 °C, with a heating rate of 15 °C min⁻¹.

X-ray diffraction (XRD) measurements were conducted with a Bruker D8 Advance diffractometer employing Bragg–Brentano geometry, using a step size of 0.02° and a 2θ scan range between 3° and 70°.

The X-ray fluorescence (XRF) data were collected on a Bruker S8 Tiger WD spectrometer. The data were analysed using SPECTRAPlus QUANT EXPRESS software.

The materials' particle size distribution and specific surface area were analyzed using a CILAS 1090 LD particle analyzer, capable of measuring within the 0.04–500 μm range.

FEI Helios Nanolab 650 scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (Oxford Instruments, INCA 4.15 software) was used for surface investigation of the samples.

Properties of cement paste was ascertained according to EN 196-3 [26].

For instrumental analysis, a sand-free ordinary Portland cement (OPC) paste was produced. The samples were submerged in distilled water maintained at 20 ± 1 °C for curing durations of 2, 7 and 28 days. After each period, the specimens were ground, washed with isopropyl alcohol, dried at 45 °C for 12 hours, and preserved in airtight containers.

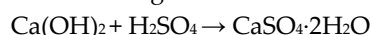
Samples (prisms 40 × 40 × 160 mm) for *compressive strength* analysis were formed according to EN 196-1 [27]. Cement-sand ratio in the samples was 1:3 and water-cement ratio 0.5:1.

Isothermal calorimetry (IC) was conducted with a TAM Air III calorimeter, with results expressed per gram of Portland cement.

3. Results

3.1. Neutralization of ESW

In this work, it was decided to neutralize the acidic ESW residues with lime milk. This method was chosen because sulfuric acid residues can react with calcium hydroxide, forming dihydrate gypsum according to the chemical reaction:



The dihydrate gypsum that can form during this reaction is a harmless material used in cement production as a setting time regulator. To evaluate the course of the neutralization process, 20g of

ESW was mixed with water to obtain three different W/S ratios – 1.25; 2.5 and 5. The results of the studies are presented in Figure 4

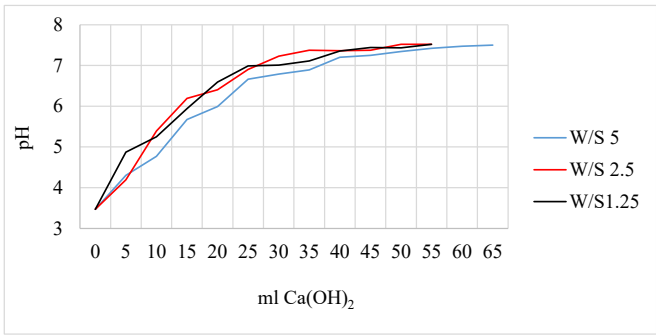


Figure 4. ESW neutralization curves.

The studies carried out show that the ESW neutralization process occurs consistently, and the proportions of water/solid material (W/S) have almost no effect on the neutralization. When the $\text{Ca}(\text{OH})_2$ content increases, the pH of the samples increases very similarly, but a higher W/S ratio leads to a slightly slower increase in pH and a longer duration of neutralization. When W/S is 1.25, neutralization occurs within 23 min; when W/S is 2.5 – 35 min, while W/S is 5 – 55 min.

The XRD analysis of neutralized ESW (NESW) samples (Figure 5) showed that the W/S ratio has no effect on the neutralization process, because the mineral composition of the samples is identical. Also, all XRD curves of NESW are very similar to the curve of raw ESW, except that the peaks of the magnetite disappear and the antigorite decreases slightly.

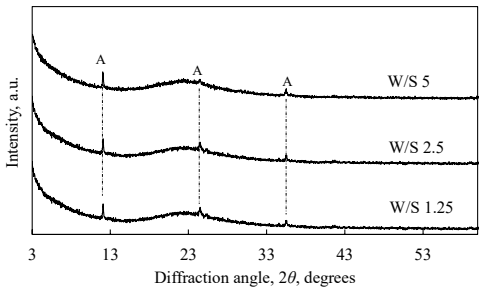
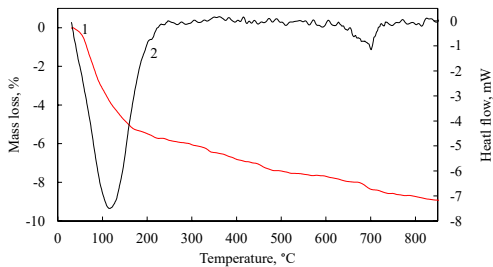
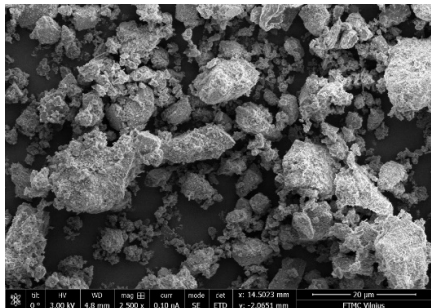


Figure 5. X-ray diffraction patterns of ESW after neutralization, Indexes: A, antigorite ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$).

Analogously, the STA curves and SEM images are similar, so only the NESW is given (Figure 6) with a W/S 2.5.



a)



b)

Figure 6. NESW, when W/S = 2.5 (a) – STA curves (1 – TG, 2 – DSC); (b) – SEM image.

As well as in case of raw ESW, two endothermic peaks are observed in the DSC curve (Figure 6, (a)) at 115 and 700 ° C that show the removal of adsorbed moisture and the decomposition of antigorite, respectively. The only difference is that during the antigorite decomposition, a slightly lower mass loss (0.62%) was recorded in the TG curve of the neutralized sample than in TG curve of the raw ESW (0.91%). Also, after neutralization the particle shape do not changes (Figure 6, (b)), because dominated irregularly shaped, partially rounded particles.

The chemical composition of NESW after neutralization with different W/S ratio are presented in Table 2

Table 2. chemical composition of NESW with different W/S ratio.

Component (wt.%)	ESW	NESW; W/S=1.25	NESW; W/S=2.5	NESW; W/S=5
SiO ₂	88.35	92.25	92.15	92.47
Al ₂ O ₃	0.10	0.12	0.13	0.13
Fe ₂ O ₃	2.47	0.86	0.93	0.90
CaO	0.48	0.56	0.55	0.58
MgO	2.64	1.52	1.51	1.51
Cr ₂ O ₃	0.07	0.07	0.06	0.06
NiO	0.12	0.06	-	0.08
SO ₃	2.73	0.84	0.76	0.76
P ₂ O ₅	0.83	0.56	0.55	0.52
Other	2.21	3.16	3.36	2.99

As expected, as the W/S ratio increases, a lower amount of SO₃ remains in the NESW material. However, this difference is negligible. The amount of P₂O₅ is also reduced, but the amount of P₂O₅ remaining in the neutralized material does not depend on the W/S ratio. The amounts of these two oxides are reduced because they are the most soluble components. The decrease in the content of MgO and Fe₂O₃ is related to the decrease in the amount of antigorite in NESW and the transition of Fe₂O₃ to the liquid phase, as shown in Figures 4 and 5. The decrease in the amount of both of these components is also independent of the W/S ratio.

In summarizing the results of the studies in this part, it can be stated that ESW can be neutralized with lime milk, and the water/solid material (W/S) ratio does not have a decisive influence on the neutralization process. During neutralization, acidic components are bound to neutral compounds. These compounds do not settle on the surface of ESW material, but pass into the liquid medium because they have not been identified in the NESW material by the XRD and STA methods. Due to the low concentration of newly formed compounds (gypsum and others) in the liquid medium, they do not crystallize, so this medium can be reused for neutralization, supplementing with lime milk to the required pH. Therefore, for economic and ecological reasons, NESW was chosen for the following studies, neutralized for 23 min when W/S was 1.25.

3.2. Influence of ESW on OPC Characteristics

The pozzolanic activity of NESW was determined. It is established that NESW is characterized by a very high pozzolanic activity. The pozzolanic activity of ground NESW reached 1085 mg CaO/g, which is similar to or higher than the activity value of metakaolinite, estimated by the same method [28].

Subsequently, the effect of NESW on the characteristics of cement paste was evaluated. Test samples were made by substituting 5–25 wt.% of OPC with the NESW additive. The influence of NESW on the normal consistency and setting time of the OPC paste is presented in Table 3.

Table 3. The effect of NESW on cement paste properties.

Component (wt.%)		Abbreviation	Normal consistency W/C (%)	Setting time (min)	
OPC	NESW			Initial	Final
100	-	OPC	0.27	84	126
95	5	S5	0.28	82	115
85	15	S15	0.30	80	105
75	25	S25	0.32	75	100

The addition of the NESW additive led to a slight increase in water demand to reach normal consistency in the cement paste. This effect is related to the absorption of part of the water for wetting of NESW particles. The shorter binding duration may also be related to the nature of NESW. Amorphous NESW particles have very strong adsorptive properties, and this process continues after the cement powder is mixed with water and the cement paste is formed. This way, part of the water required for cement hydration is consumed, and less water means faster stiffening, shortening initial and final sets.

The influence of NESW on early hydration of cement paste has been determined by isothermal calorimetry (IC) tests. The results of the test are shown in Figure 7.

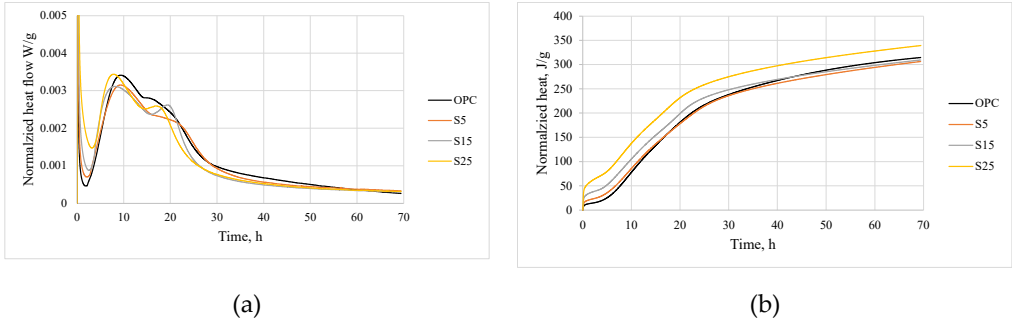


Figure 7. The IC tests results: (a) Heat flow; (b) Total heat.

Two peaks of heat evolution were observed on the calorimetric curve of the samples: the first concerned wetting a cement powder and the second related to hydration of the deeper layers of calcium silicates. On the second peak, a shoulder is visible that is associated with an aluminate hydration reaction [29]. In samples with additives, the induction period (Figure 7, (a)) lasts longer (2h 15min – 3h) than in the OPC sample (2h 10 min), but during this period, samples with additives release more heat (Figure 7, (b)). During the second exothermic reaction stage, the heat flow curve showed that the NESW-containing samples exhibited earlier heat release compared to the OPC sample. The sample with 15 wt.% NESW reached its maximum heat flow 8 hours 50 minutes after the hydration began, while the sample with 25 wt.% NESW reached its maximum at 8 hours 25 minutes. On the contrary, both the pure cement and the sample with 5 wt.% NESW achieved their maximum heat flow at 9 hours 12 minutes. Additionally, up to 20 h of hydration, all samples with the NESW additive emit more hydration heat than OPC. Therefore, it can be inferred that the addition of NESW enhanced the rate of early-stage cement hydration.

Figure 8 shows the compressive strength data of the OPC with different amounts of NESW. After 2 days of hardening, the highest compressive strength shows the OPC and S5 sample (~ 20 MPa). Although the S15 and S25 samples exhibited lower compressive strength values, all specimens achieved strengths above 10 MPa, satisfying the requirements of the EN 197-1:2011 standard [30]. After 28 days of curing, all samples with NESW additive exhibited a higher compressive strength (46.3–54.9 MPa) compared to the OPC sample (45.8 MPa). The S15 sample is characterized by a particularly high compressive strength, the compressive strength of which (54.9 MPa) corresponds

to an even higher strength class (52.5) than the class of OPC used (42.5). Thus, NESW is a very effective SCM, which can replace as much as 25 wt.% of Portland cement.

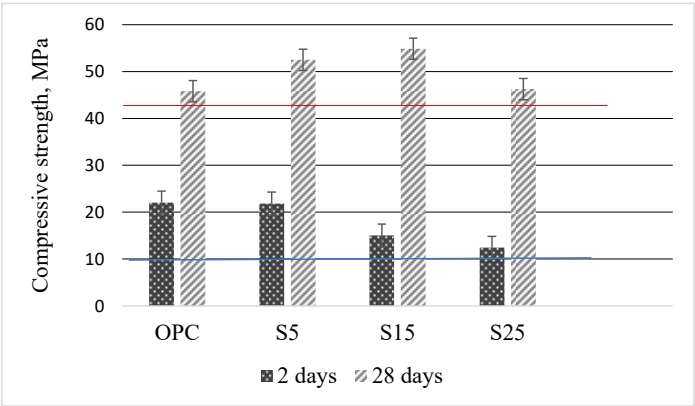
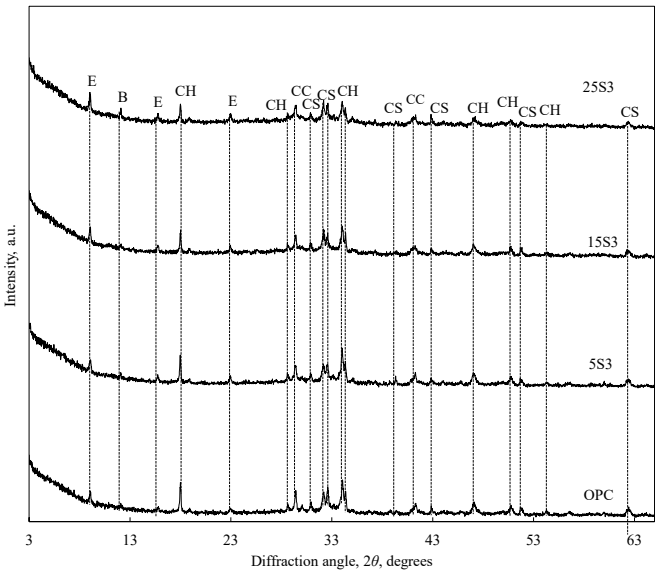


Figure 8. The compressive strength of the samples hardened for 2 and 28 days.

The XRD curves (Figure 9) indicate that no new crystalline phases were detected in the samples containing the NESW additive after 7 and 28 days of curing. In all samples, ordinary cement hydrates, ettringite ($\text{Ca}_6(\text{Al}(\text{OH})_6)_2(\text{SO}_4)_3 \cdot 26\text{H}_2\text{O}$) (PDF 41-1451) and portlandite ($\text{Ca}(\text{OH})_2$) (PDF 84-1271) was identified. Additionally, unhydrated calcium silicates (C_3S) (PDF 00-055-0739), brownmillerite C_4AF (PDF 00-030-0226) and calcite (CaCO_3) (PDF 5-586) were found in the samples. The observed differences are limited to variations in the relative intensities of the diffraction peaks corresponding to unhydrated phases and hydration products. At both hydration ages, the samples with the additive exhibit less pronounced portlandite and unhydrated calcium silicate peaks, compared to those in the reference OPC sample.



(a)

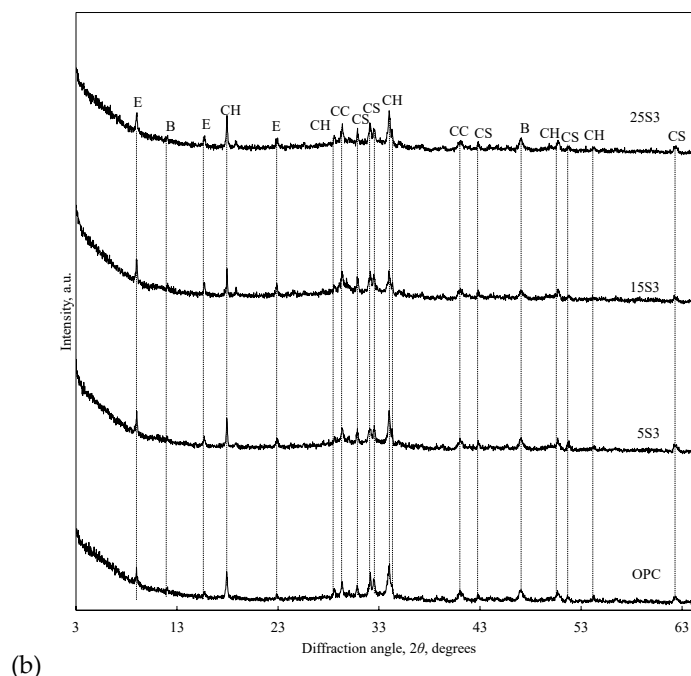


Figure 9. The XRD analysis curves of samples after (a) 7 days and (b) 28 days of hydration. Indexes: E – ettringite; CH – portlandite; B – brownmillerite; CS – calcium silicates, CC – calcite.

The DSC curves obtained for the samples after 7 and 28 days of ageing are presented in Figure 10, and the corresponding thermogravimetric (TG) analyses are shown in Figure 11. All DSC thermograms display three well-defined endothermic peaks located within the temperature intervals of 90–270 °C, approximately 450 °C, and 570–750 °C. The initial endothermic effect is associated with the dehydration of calcium silicate hydrates (C–S–H), calcium aluminate hydrates, and ettringite. The peak near 450 °C corresponds to portlandite dehydroxylation, while the high-temperature peak observed between 570 °C and 750 °C is attributed to the decomposition of carbonates generated by the carbonation of the samples [31].

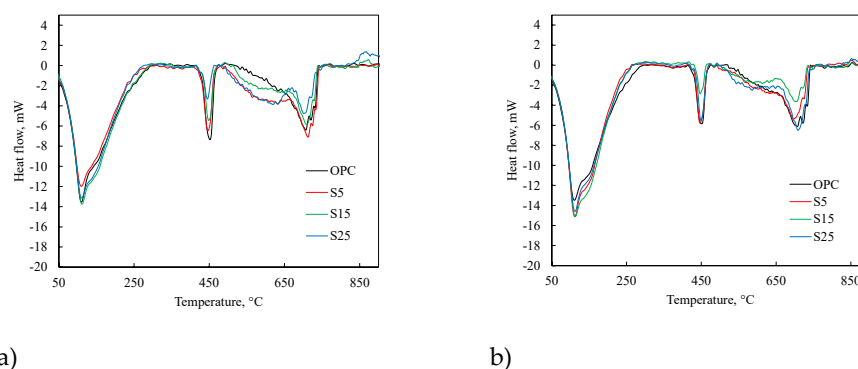


Figure 10. DSC analysis curves: (a) – 7; (b) – 28 days of curing.

All curves exhibit a generally similar shape; however, noticeable differences in their peak intensities are observed. For both curing durations, the intensity of the first endothermic peak, corresponding to the decomposition of the main cement hydration products, increases with the addition of NESW, indicating a direct relationship between additive content and peak magnitude. On the contrary, the endothermic peak associated with portlandite dehydroxylation is more pronounced in the OPC sample and in the S5 sample and progressively decreases as the proportion

of the additive increases. Meanwhile, the intensity of the calcite decomposition peaks does not show a clear trend.

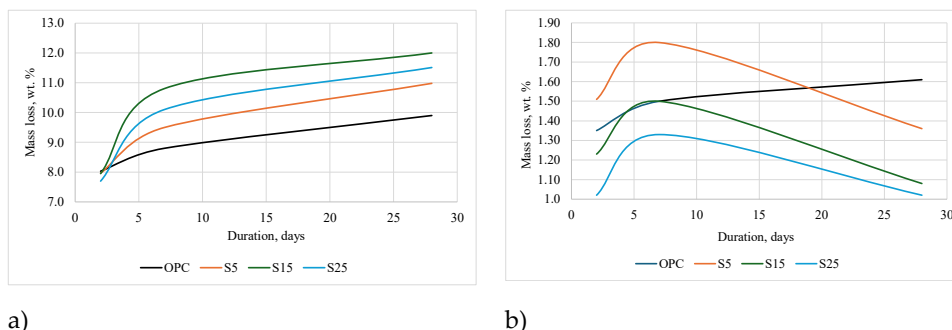


Figure 11. TG analysis curves: (a) – temperature range 90-270 ° C; (b) – ~450 ° C;.

The results of the thermogravimetric analysis (TG) are presented in Figure 11. After 2 days of hydration, the mass loss recorded within the 90-270 ° C range was quite similar for all samples. Meanwhile, in the portlandite decomposition region (~450 ° C), the greatest mass loss occurred in the S5 and OPC samples. Extending the hydration period to 7 days led to a consistent increase in mass loss within the 90–270 ° C range for all samples, but the slowest growth observed in the OPC sample. A similar trend was observed near 450 ° C. The increase in mass loss was more pronounced in mixtures containing the NESW additive than in the OPC reference. After 28 days of hydration, mass loss within the 90-270 ° C range (Figure 11a) increased further in all samples. It should be noted that the samples incorporating the additive exhibited significantly higher values (10.98–12.00%) than the pure Portland cement sample (9.9%). A distinct difference was observed near 450 ° C (Figure 11b), where portlandite decomposition occurred. At this stage, the mass loss associated with portlandite decomposition increased in OPC but decreased markedly in additive-containing samples, indicating the onset of pozzolanic reactions consuming portlandite.

The results of this section explain the results of the compressive strength of the tested samples. After two days of hardening, the amount of conventional cement hydrates (C-S-H, calcium aluminate hydrates, and ettringite) in all samples is similar. However, in OPC and S5, which had the highest compressive strength after two days of curing, a higher amount of portlandite formed was found. Meanwhile, after 28 days of curing, a higher amount of cement hydrates (mainly C-S-H, formed due to the pozzolanic reaction) was found in all samples with the NESW additive, determining the higher compressive strength of the samples.

4. Conclusions

- Epsom salt production waste consists of irregularly shaped sharp-edged amorphous materials with crystalline antigorite and magnetite impurities. It is an acidic material with a pH of 3.47.
- ESW can be neutralized with lime milk, and the water/solid material (W/S) ratio does not have a decisive influence on the neutralization process. After neutralization, the ESW becomes a non-acidic material with pH value 7.5 because during neutralization the acidic components are bound to neutral compounds that do not settle on the surface of the ESW material, but pass into the liquid medium.
- Neutralized Epsom salt production waste (NESW) characterized by a very high pozzolanic activity (1085 mg CaO/g). NESW results in a slight increase in water consumption to achieve normal consistency in cement pastes and a modest extension of the setting time of the Portland cement paste.

- The addition of NESW accelerates the initial hydration of Portland cement and induces a strong pozzolanic reaction, clearly observable after 28 days of hydration.
- NESW is a very effective supplementary cementitious material, which can replace as much as 25 wt.% of Portland cement without reducing the strength class of Portland cement.

Author Contributions: Conceptualization, R.K., A.Z and I.B.; methodology, R.K., A.Z and I.B.; software, A.Z and I.B.; validation, R.K., A.Z and I.B., formal analysis, R.K., A.Z; investigation, R.K., A.Z and I.B.; resources, R.K., A.Z and I.B.; data curation, R.K., A.Z and I.B.; writing—original draft preparation, R.K. and A.Z; writing—review and editing, R.K.; visualization R.K., A.Z and I.B.; supervision, R.K. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

The following abbreviations are used in this manuscript:

OPC	Ordinary Portland cement
SCM	Supplementary cementitious materials
C-S-H	Calcium silicate hydrates
ESW	Epsom salt production waste
DSC	Differential Scanning Calorimetry
TG	Thermogravimetric analysis
STA	Simultaneous thermal analysis
XRD	X-ray diffraction analysis
XRF	X-ray fluorescence analysis
SEM	Scanning electron microscope
IC	Isothermal calorimetry analysis
NESW	Neutralized Epsom salt production waste
W/S	water/solid material ratio
C ₃ S	Calcium silicates
C ₄ AF	Brownmillerite

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