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Chemical Resistance of Sustainable Concretes Modified with Waste Powder Containing Epoxy Resin, Calcite and Barite

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Abstract. The currently designed building materials should be in line with the principles of sustainable development, low-emission economy and circular economy. Guided by these criteria, waste powder containing epoxy resin, calcite and barite was included in the concrete mix. The waste material was used as an additional component of the concrete mix in the amount of 7.5% of the cement mass, without reducing the amount of binder. At the same time, the water/cement ratio was used at a level of 0.44 to 0.56. The obtained concretes were characterized by good workability and fluidity, despite the modifier used. Tests were carried out to assess the resistance of the obtained concretes to the effects of sulphates and chlorides. After 28 days of curing, the concrete samples were immersed in a 5% sodium sulphate solution and a 5% sodium chloride solution. After 48 days of exposure, a decrease in the compressive strength of the concrete samples modified with waste powder was noted in relation to the control concretes. In the case of samples exposed to sulphate salts, the introduction of 7.5% by weight of waste into the concrete composition was associated with a reduction in compressive strength by 8% or 9.9%, respectively, for a higher or lower water/cement ratio. The compressive strength of concrete immersed in sodium chloride solution was reduced by 9.9% for samples containing 7.5% waste at a water/cement ratio of 0.56 and by 5.4% when the water/cement ratio was 0.44. The mass of the samples was also monitored during the test. An increase in the mass of all samples, modified with waste powder and those without the modifier, was noted. The average mass changes for concretes immersed in sodium sulphate solution ranged from 0.16% to 0.31%, and for sodium chloride solution from 0.15% to 0.24% and were dependent on the water-cement ratio and the presence of waste in the concrete mix. The analysis of the durability of concretes showed no harmful effect of the modifier on the microstructure of the cement paste resulting from the action of corrosive reagents. The conducted FTIR, XRD, SEM tests did not confirm the formation of, among others, secondary ettringite, which could cause microstructural damage to the cement paste by generating expansive forces, especially in small pores due to the high crystallization pressure. The proposed method of modifying the composition of concrete is promising and can help reduce the problem related to waste management, but also help reduce greenhouse gas emissions.

Keywords: Sustainable concretes; circular economy; low carbon economy; waste powder; chemical resistance.

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INTRODUCTION

Among the current research trends in the field of construction, the topic of waste management methods stands out. Although landfilling, incineration, and composting are commonly used waste disposal technologies, it is still advisable to look for other methods of using waste, especially those that can be considered innovative, consistent with the idea of sustainable development. One of the possible solutions to the broadly understood problem of waste is to include it in the composition of the most popular building material, concrete. Such an approach is in line with the assumptions of a circular economy. The results of research described in the literature indicate that the modification of concrete by recycled materials can take place in various ways.

It is worth introducing recyclate instead of cement, which, apart from waste management, can contribute to reducing the carbon footprint. Positive effects of such substitution were noted, among others, by authors who introduced the following wastes instead of cement into the concrete mix: glass powder (WGP), coal gangue powder (CGP) and fly ash (FA) [1], barite, hematite, and lead powder [2], calcite-waste toner powder [3], processed bagasse ash and silica fume [4].

The inclusion of recyclates instead of aggregates in the composition of concrete can also be a way to reduce some of the problems resulting from the need to dispose of waste, and at the same time to limit the use of dwindling natural resources and often provides an opportunity to introduce environmentally friendly products to the market. This method of concrete modification was proposed in scientific works, in which the following waste materials were included among the modifiers: granite dust [5], hard-to-recycle cross-linked polyethylene (XLPE) waste derived from used electric cables [6], rubber powder [7], recycled aggregate concrete [8], polyethylene terephthalate (PET) plastic [9], waste air-cooled ferrochromium slag [10], waste marble [11], recycled glass powder [12].

Waste can also be an additive to concrete, as confirmed by research conducted by our team. With a properly designed concrete mix, the addition of waste powder containing epoxy resin, calcite and barite allows for obtaining concretes characterized by good workability and fluidity despite the modifier used. They are also characterized by high mechanical strength and relatively low water absorption.

When designing the composition of concrete, it is extremely important to take into account the conditions in which it will be used in order to ensure its appropriate durability. The basis for predicting the durability of concrete before it is approved for a given application is to determine its resistance to, among others, corrosive factors such as sulphate ions and chlorides [13, 14]. Sulphate anions (SO_4^{2-}) contained, for example, in water from industrial processes and sewage and in sea or underground water can penetrate concrete and react with hydrated calcium aluminates from cement hydration. Difficult to dissolve chemical compounds are formed, which is accompanied by an increase in the volume of reaction products and the occurrence of significant mechanical stresses, cracking and spalling, and consequently, loss of mechanical strength and destruction [14–17]. Corrosion caused by chlorides contained, among others, in sea water or mine water is equally unfavourable for concrete. Concrete is destroyed by the reaction of chloride ions with the products of cement hydration, and additionally the corrosion processes of the reinforcing steel are significantly accelerated.

Taking into account the issues of ecology and durability of the designed materials, this article describes the results of tests on concretes modified with waste powder (WP) from the paint industry, exposed to direct action of corrosive media in the form of 5% solutions of sodium sulphate and sodium chloride. The results of physico-mechanical tests combined with analyses of the chemical composition and microstructure of the obtained composites indicate that the inclusion of waste powder in the composition of concretes is possible and allows to obtain products attractive in the context of future construction applications.

MATERIALS AND METHODS

Raw Materials

Basic ingredients of the concrete mix:

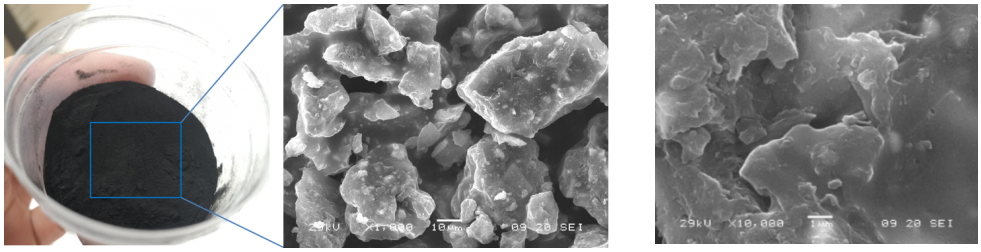
• Portland cement CEM I 42.5 R, produced by “Ozarow S.A.”, Ozarow, Poland, comply with the requirements of [18]. Mineral composition of cement clinker: C3S—68.0%, C2S—10.3%, C3A—7.7%, C4AF—7.2%;

• Quartz sand of 0–2 mm fraction, produced by “Kruszgeo S.A.”, Mrowla, Poland, according to [19];

• Superplasticizer (SP) MasterGlenium ACE 430, highly effective liquefaction admixture based on polycarboxylate ether technology, produced by “Master Builders Solution”, Myslenice, Poland, according to [20];

• Tap water that meets the requirements PN-EN 1008:2004 [21].

Waste powder (WP) from the paint industry was used to modify the concrete. The morphological SEM analysis shows a predominance of equidimensional and angular-shaped particles (fig. 1). Furthermore, EDS mapping indicates the presence of elements Ba, Ca and S, agreeing with the XRD and XRF results (fig. 2).



(a) (b)

FIGURE 1. Waste powder used in research a) general view, b) waste grain morphology with a lamellar structure.

XRD analysis was performed for the waste, the results of which are presented in Figure 2. The qualitative XRD analysis shows peaks associated with barite, bastnasite and calcite, as well as an amorphous halo between 12° and 24° (2θ). The qualitative XRF composition is: Ce, Ca, S, Ba, Pr, Cu, Sr, Mn, Si, and Co.

The detailed composition of the concrete mix is given in Table 1. Four types of compositions were prepared, differing in the water/cement ratio, with or without waste.

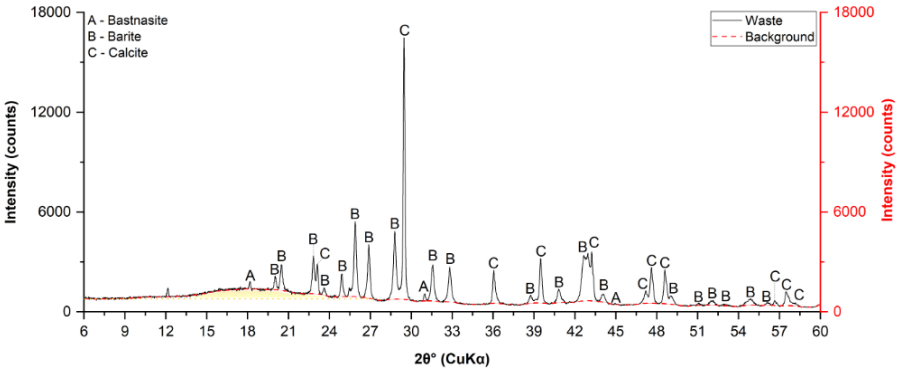


FIGURE 2. Results of XRD analysis of waste.

TABLE 1. The concrete mix composition for 1m^3.

Specimens	Cement	Fine aggregate	Coarse aggregate	Plasticizer	Waste powder	Water	w/c
	kg	kg	kg	% c.m.	kg	dm^3	-
10 3S	368	623.2	1400.6	0.6	-	163.2	0.44
11 2S		587.0	1319.3	-	-	204.8	0.56
5 3S		609.1	1368.9	0.5	27.6	163.2	0.44
6 2S		573.0	1287.7	-	27.6	204.8	0.56

Methods

For **thermogravimetry (TG)** and **differential thermal analysis (DTA)**, **X-ray diffraction (XRD)**, and **Fourier Transform Infrared Spectroscopy (FTIR)** analyses, the concrete samples were crushed and manually milled in a mortar, then sieved using standardized 53-micrometers sieve. For **scanning electron microscopy (SEM)** analysis, the concrete samples were crushed and mounted on carbon tape attached to a metallic stub, followed by gold coating.

For **FTIR** analysis a Nicolet iS5 spectrometer (Thermo Scientific) was used in the range of 4000 to 400 cm^{-1}, with measurements performed on compressed tablets produced by mixing the sample of interest with KBr.

The **TG/DTA** analysis was carried out using a Mettler Toledo TGA/DSC 1 Stare System thermogravimetric analyzer, under standardized measurement conditions with a temperature range of 30°C to 980°C, a heating rate of 10°C/min, and a N2 gas flow of 30 mL/min. In this context, the concrete samples were analyzed in closed alumina crucible, while the waste sample was analyzed in open alumina crucible.

For **XRD** analysis, a Bruker D2 Phaser diffractometer was used for mineralogical prospecting, operating at 10 mA and 30 kV. The concrete samples and waste sample were analyzed under standardized measurement conditions using a back-loading preparation method, with a scanning range of $5° \leq 2\theta \leq 60°$, with a step size of 0.01819°, and 1.25 s per step. Qualitative phase identification was conducted based on reference patterns from the Crystallography Open Database (COD) [22, 23], using the X'Pert HighScore Plus software.

The **SEM** testing was performed using the Tescan Vega 3 scanning electron microscope on the waste powder and the fractured surfaces of the concrete samples. An accelerating voltage of 20 kV and a variable working distance were applied for this purpose. Microstructure analysis was also performed on concrete samples mechanically separated using scanning electron microscopy. The JSM–5500 LV microscope was used for this study.

The concrete mixture was formed in the appropriate moulds according to [24]. Immediately after dismolding, the samples were placed in a climate chamber with a temperature of 20 ± 2°C and a humidity of 95 ± 5%, where they were cured until the appropriate study time according to [25].

The **compressive strength** at the age of 28 and 90 days was performed on a FormTest PRÜFSYSTEME press, type ALPHA 3-3000S, according to [26]. For the test, six 10x10x10 cm samples were made for each concrete mixture.

The **mass change** was monitored as one of the indicators of concrete degradation. The mass of the sample before (m_1) and after (m_2) exposure to solutions containing sulphate or chloride ions was measured on an electronic balance with an accuracy of 0.01 g. Before immersion in corrosive solutions, the samples were cured in water for 28 days, and the mass of the samples in the saturated state was measured as the initial mass (m_1). After immersion in the sulphate or chloride solution for a specified time, the samples were dried and their mass (m_2) was determined. The study was carried out on three samples for each concrete mix.

RESULTS AND DISCUSSION

Results of TG/DTA and FTIR Analyses of the Residue

The results of the TG/DTA analyses are presented in Figure 3.

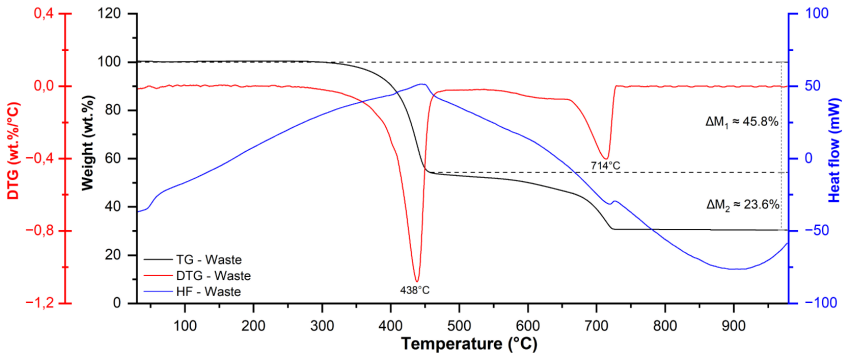


FIGURE 3. TG/DTA analysis results of waste.

Regarding the TG/DTA analysis, two main mass loss events can be observed. The first one shows a mass loss of approximately $\Delta M_1 \approx 45.8\%$, occurring between 300°C and 475°C, with a DTG peak at 438°C and an exotermic peak on the DTA curve. Considering the origin of the waste and based on the previous results in the literature regarding thermal behavior of resins [27, 28], it is possible to suggest that the exothermic peak on the DTA curve indicates an ongoing chemical reaction, probably the cross-linking reaction of the epoxy resin, while this mass loss is affected from the thermal degradation of epoxy resin present in the waste. The second mass loss event, which presents $\Delta M_2 \approx$ 23.6%, occurred between 480°C and 740°C, with a DTG peak at 714°C and a corresponding endothermic peak observed on the DTA curve. Considering the presence of calcite ($CaCO_3$) identified by XRD in this study, as well as characteristics bands associated with carbonate groups indicate by FTIR, this peak can be predominantly attributed to

the decomposition reaction of calcium carbonate [29]. By the other hand, the presence of barite ($BaSO_4$) was observed by XRD and the presence of barium atoms were confirmed by SEM/EDS. In this sense, the thermal analysis of barites by Gondim et al. [30] shows that the barites can loss mass mainly between 400 °C and 600°C. About this, probably, the loss of mass from the barite is also influencing the thermal behavior in both interval of temperature.

Summary of FTIR analysis results of waste are presented in Table 2.

TABLE 2. Summary of FTIR analysis results of waste.

Peak wavenumber identified	Possible phase	Assignment	Wavenumber reference	Reference
cm^{-1}			cm^{-1}	
3440	Water or epoxy resin	O–H	3440	[31]
2962 2923 2869		C–H of CH_2 and CH aromatic and aliphatic	2965–2873	
1608	Epoxy resin	C=C of aromatic ring	1608	[32]
1508		C–C of aromatic	1509	
1427	Calcite	CO_3^{2-}	1420–1490	[33]
1242	Epoxy resin	Ether bonds in epoxide groups	1246	[34]
1180	Barite	SO_4^{2-}	1180	[35]
1107	Barite	SO_4^{2-}	1080–1130	[36]
1084	Barite	SO_4^{2-}	1090	[35]
953	Epoxy resin	C–O–C of oxirane group (asymmetric vibration)	950–810	[32]
876	Calcite	CO_3^{2-}	870 880	[36] [37]
829	Epoxy resin	C–O–C of oxirane group (symmetric vibration)	831	[32]
710	Calcite	CO_3^{2-}	714 712	[33] [37]
632	Barite	SO_4^{2-}	610–638	[36]
609	Barite			
467	-	Si–O	460	[38]

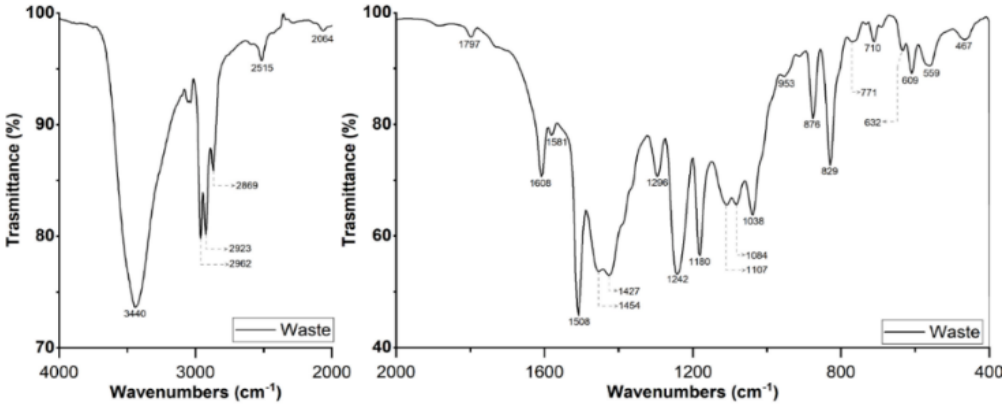


FIGURE 4. FTIR analysis results of waste.

Regarding the FTIR analysis of the waste, the spectrum exhibited different absorption peaks (fig. 4). Based on the characteristic bands in the FTIR spectrum, it can be concluded that the waste powder used contains epoxy resin, calcite and barite, which was also confirmed by TG/DTA tests.

Compressive Strength

The compressive strength test results after 28 days curing of concrete are presented in Table 3. A deterioration in compressive strength was observed due to the introduction of waste powder into the concrete mix in the amount of 7.5% of the cement mass. The greatest decrease in strength was obtained in the case of the highest water-cement ratio of 0.56; a decrease of 16%. Concrete modified with waste powder with a water-cement ratio of 0.44 showed a decrease in compressive strength of only 2%. The concrete compressive strength test was also conducted after 90 days, including 62 days of exposure to corrosive solutions. Durability tests in corrosive solutions showed a decrease in the strength of concretes modified with waste powder compared to unmodified concrete. The decrease in compressive strength ranged from 5.4% to 10%. According to the obtained results, it is concluded that a waste powder had no significant effect on the durability of concrete.

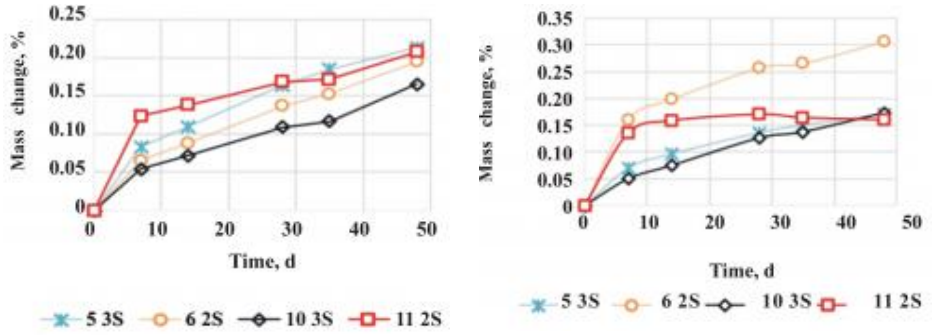
TABLE 3. Results of the compressive strength test of concrete.

Specimens	w/c	Waste powder	Compressive strength after 28 days	Standard error of the mean	Compressive strength after 90 days[a]	Standard error of the mean	Compressive strength after 90 days[b]	Standard error of the mean
	-	% c.m.	MPa	MPa	MPa	MPa	MPa	MPa
10 3S	0.44	-	68.8	1.60	86.2	0.98	83.8	0.57
11 2S	0.56	-	49.3	0.66	56.3	2.35	54.6	2.62
5 3S	0.44	7.5	67.4	2.03	77.7	0.74	79.3	0.48
6 2S	0.56	7.5	41.4	1.17	51.8	1.44	49.1	1.67

[a] samples exposed to 5% sodium sulphate solution, [b] samples exposed to 5% sodium chloride solution

Mass Change

In the assumed period of exposure, a slight increase in the mass of all tested concrete samples was noted (fig. 5), which is consistent with the results described by other authors [18]. In the case of exposure in a 5% sodium chloride solution, the lowest values of mass change were obtained for the composition without waste, with a low w/c ratio (10 3S). After 48 days of immersion, the average mass change in this case was 0.17%, while the remaining concrete samples were characterized by an average mass change of 0.19% to 0.21%. The average mass change of samples containing waste and high w/c, immersed in 5% sodium sulphate, was the highest and amounted to 0.31%. While maintaining a low w/c value, there is no major difference in the average mass change of samples containing waste powder and unmodified ones. For these concretes, the tested parameter assumed values equal to approx. 0.17%. For the mass change, the values of the coefficients of variation were calculated, which in most cases do not exceed 20%, so the data are characterized by small variability, most values are close to the mean. Only two results are characterized by strong variability, greater than 45%. In this case, the data are very scattered and show large variability in relation to the mean.



(a) (b)

FIGURE 5. Change in mass of samples exposed to a) 5% NaCl; b) 5% Na_2SO_3.

FTIR, TG, XRD of the Concrete

The FTIR analysis results of concrete are given in Table 4.

TABLE 4. Summary of FTIR analysis results for concrete.

Peak wavenumber identified	Possible phase	Assignment	Wavenumber reference	Reference
cm^{-1}			cm^{-1}	
3637	$Ca(OH)_2$	O–H	3640	[31]
3444–3429	Water	O–H	3440	
2970	Water	H–O–H	2970	[39]
1632	Water	H–O–H	1640	[40]
1435–1427	Calcite	CO_3^{2-}	1420–1490	[41]
			1480–1430	[41]
1084	Calcite	CO_3^{2-}	1080	[41]
876	Calcite	CO_3^{2-}	875	[41]
795	Quartz	Si–O	~800	[42]
513	-	Si–O	525	[41]
462	-	Si–O	460	

Figure 6 shows the FTIR spectra obtained in the range of 4000–400 cm^{-1}. The FTIR analysis qualitatively confirms the presence of bands associated with typical chemical bonds found in common phases of the cementitious composite. It was possible to identify characteristic bands associated with common phases related to the hydration of Portland cement. No specific changes related to the introduction of the baryte-rich residue were confirmed, neither by band shifts nor by the appearance of new bands.

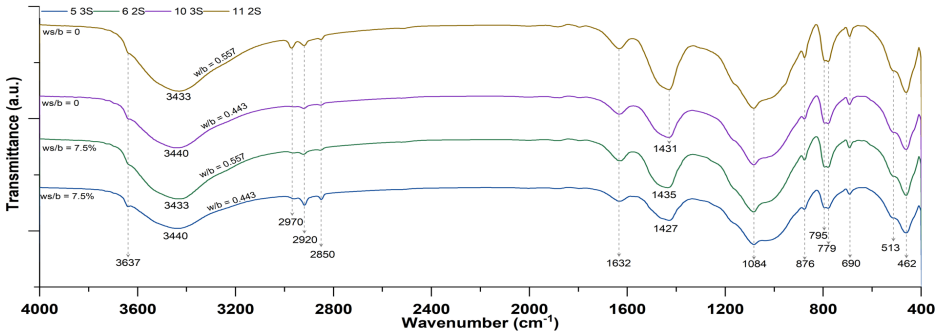
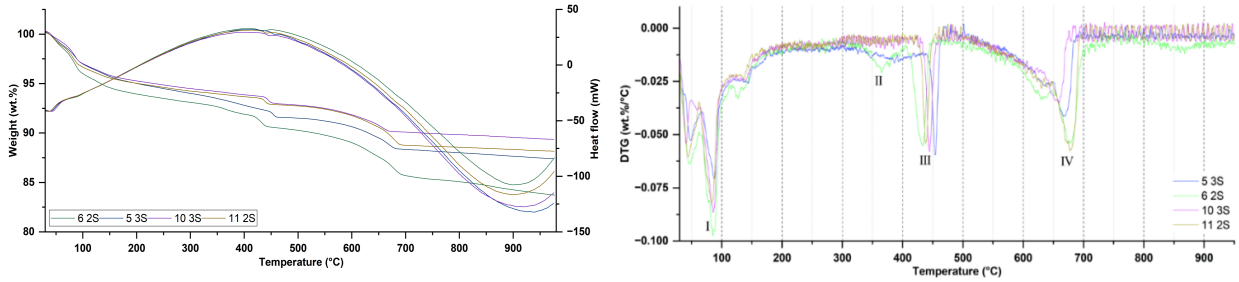


FIGURE 6. FTIR analysis results of concrete.

Figure 7-8 shows the TG analyses of the concretes.



(a) (b)

FIGURE 7. Results of analyses performed for concrete samples a) TG, b) DTG

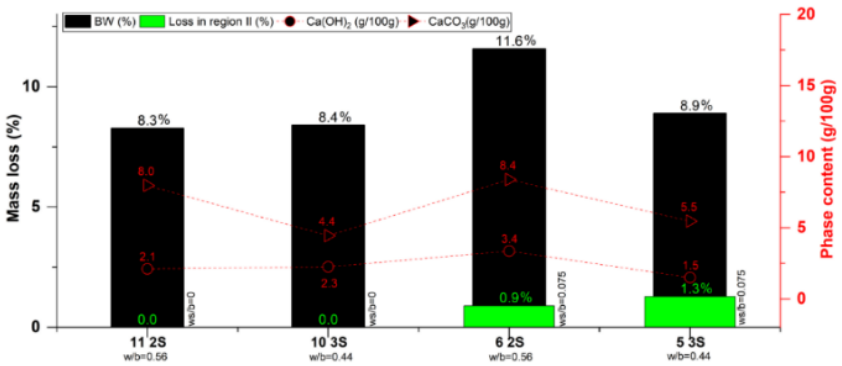


FIGURE 8. Mass loss and phase content obtained for individual concrete samples in TG/DTG tests.

The TG curves were plotted as temperature (X) versus percentage mass change (Y). First and second derivatives (DTG) were calculated with respect to temperature, followed by smoothing using the Savitzky-Golay method. For Region II, the mass loss was determined by directly subtracting the residual mass values. For Regions III and IV, linear fits were applied to obtain tangent lines, followed by linear extrapolation and slope calculation aligned with the DTG peaks. In Regions III and IV, standard mass balance calculations were performed for $Ca(OH)_2$ and $CaCO_3$, respectively, along with normalization by the remaining mass at 600°C and 980°C, respectively. For the combined water content associated with other phases, it was subtracted the calculated contents in Regions II, III, and IV, as well as the final mass, from the initial 100% mass (without balance or normalization). The samples containing residue exhibited a slightly altered TG curve, due to the presence of two characteristic mass loss regions that overlap with those of $Ca(OH)_2$ and $CaCO_3$ - typical phases of hydrated cement - which likely influenced the overall curve profile. A subtle peak was identified exclusively in the residue-containing samples, designated as Region II (300–400 °C). It was also observed that samples with higher water content and residue in their composition (6 2S) exhibited more pronounced DTG peaks in the 60–100 °C and 100–150 °C ranges. It is important to acknowledge the limitations introduced by the incorporation of a multiphase residue and the influence of an aggressive environment, which may promote the formation of new phases within the matrix. It is worth continuing the research taking into account a longer exposure time.

Figure 9 presents the XRD analysis of the concrete samples.

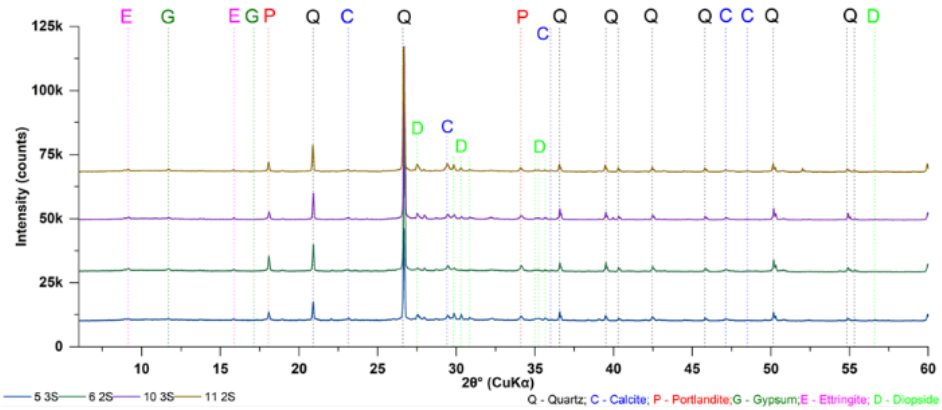


FIGURE 9. XRD analysis of the concrete samples.

Typical phases associated with the hydration of Portland cement, such as ettringite and portlandite, were identified. Peaks related to quartz and diopside were also observed, likely originating from the aggregates used in concrete production. In the case of calcite, its presence may be attributed both to the carbonation process (caused by exposure to environmental CO_2) and to the waste material employed, which showed characteristic calcite peaks in the initial analysis. In addition, two characteristic low-intensity peaks of gypsum were identified. Given that the material had already undergone hydration for several days, it is likely that these peaks are associated with the reaction in which portlandite reacts in the Na_2SO_4 presence, leading to the gypsum formation.

CONCLUSION

The tests showed that the use of waste powder as a modifier of concrete composition does not significantly affect the strength parameters of concrete, especially at a low water-cement ratio (strength drop after 28 days by only 2% with the share of waste powder in the amount of 7.5% of the cement mass) and does not cause deterioration of the durability of these concretes assessed on the basis of mechanical tests and mass change as well as FTIR, TG, XRD analyses.

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