



Article The Advantages on Using GGBS and ACBFS Aggregate to Obtain an Ecological Road Concrete

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Abstract: This work aims to show the advantages of using GGBS (Ground Granulated Blast Furnace Slag) and ACBFS aggregate (Air-Cooled Blast Furnace Slag) on the tensile strength and durability properties of infrastructure concrete at the reference age of 28 days. Three concrete mixes were prepared: the first one was a control sample; the second one had 15% GGBS (instead of Portland cement) and 25% ACBFS (instead of natural sand); and the third had 15% GGBS (instead of Portland cement) and 50% ACBFS (instead of natural sand). The studies on mortars focused on the ratio of compressive strength (CS) in correlation with the specific surface area (obtained by the Blain method). The microstructure of the prepared mortars was examined at the age of 28 days by X-ray diffraction, SEM electron microscopy with an energy-dispersive EDX spectrometer, and NMR nuclear magnetic resonance relaxometry. The results of the tests carried out afterwards on the concretes containing slag (15% GGBS and 25% or 50% ACBFS) showed values that met high-quality criteria for exfoliation $(S_{56} < 0.1 \text{ kg/m}^2)$, carbonation, and gelling G100 (with a loss of resistance to compression $\eta < 25\%$). The slag concretes showed a degree of gelation of G100 (with a loss of compressive strength below 25%), low volume losses below 18,000 mm^3 /5000 mm^2 (corresponding to wear class 4, grade I), and moderate penetration of chlorine ions (according to the RCPT test). All of these allow the concrete with slag (GGBS/ACBFS) to be recommended as an ecological road concrete. Our study proved that a high-class road concrete of BcR 5.0 can be obtained, with tensile strengths of a minimum 5 MPa at 28 days (the higher road concrete class in Romania, according to national standards).

Keywords: blast furnace slag; hydration activity index; flexural tensile strength; exfoliated mass; penetrability of chlorine ions; wear resistance; frost-thaw resistance

1. Introduction

In less than one century, concrete has become the most widely used construction material in the world [1], which is why large amounts of natural resources are consumed in its production. As a measure to ensure the ecological balance, it is necessary to implement the use of various unconventional and renewable resources [2,3]. An important characteristic of cementitious materials is their ability to accommodate large amounts of waste by replacing some or all of the cement and/or natural aggregate. Extensive research work has been carried out to evaluate the suitability of various industrial by-products as supplementary cementitious materials (SCM) and their influence on the rheology and physical and mechanical properties of mortars and concretes [4–10]. In the steel industry, millions of tons



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of slag are a by-product that has to be neutralized for environmental reasons. The slag obtained is classified according to the origin of the minerals and the solidification process. Slag that cools slowly to atmospheric temperature is called air-cooled blast furnace slag (ACBFS), and slag that is instantaneously cooled under pressurized water jets is called granulated slag; the latter is ground into a powder that turns into ground granulated blast furnace slag. A crystalline character is observed in ACBFS slag by slow cooling in the air [11], while little or no crystallization occurs in slag rapidly cooled in water, where the glass content predominates [12,13].

The durability and mechanical properties of cement composites with GGBS largely depend on the hydraulic activity of the slag. The properties of the slag powder and the hydraulic activity are mainly affected by the glass content, chemical composition, and fineness of the slag powder. The vitreous content and chemical composition are passively determined by the ironmaking process, but the activation performance of GGBS can be changed by the fineness of the slag powder. The fineness of GGBS is usually expressed in terms of specific surface area and particle size distribution. Different levels of fineness can be achieved by grinding cementitious materials using different methods and grinding times [14–16]. By increasing the fineness of GGBS, the cementitious properties of cement containing steel slag are significantly improved [17]. The use of GGBS obtained by wet milling results in improved mechanical strength, reduced porosity, and low Ca(OH)₂ content in concrete composites [18]. One of the important uses for GGBS is in concrete structures in marine environments as it has been shown to reduce the penetration of chlorides into concrete. Other positive effects of GGBS in concrete are protection against sulfate attacks and the alkali–silica reaction.

More stringent requirements apply to pavement concretes used on roads, platforms, and/or airfields than those used in hydraulic or civil engineering [19-21]. Atmospheric factors caused by temperature fluctuations and the influence of traffic caused by dynamic loads, as well as the presence of chemical deicing agents, contribute to the demanding operating conditions of pavement concrete [21,22]. The pollutant transport processes through concrete are different and can occur through absorption, when the movement of liquids is due to the capillary forces generated in the capillary pores. Transport occurs by permeability when the fluid movement is due to the action of pressure and by diffusion when the fluid movement is due to a concentration gradient [23]. Finally, the penetration of aggressive substances from the operating environment into the component impairs the durability of road concrete [24]. Due to the limitations related to the percentage of cement substitution, for example, the Swedish standard SS 13 70 03 [25] allows the addition of GGBS up to 25% of the CEM I cement for exposure class XF4, a class which is specific to road concrete. According to the Indian standard IS: 383, the replacement of natural sand with blast furnace slag is allowed up to a limit of 50% for unreinforced concrete [11], and the ACI 213R guidance [26] recommends the range of 20%-60% for the replacement of natural sand with ACBFS. The substitution percentage limitation is based on previous research, which indicated that ACBFS aggregate could be a potential internal source of sulfates released into the pore solution. These sulfates contribute to the filling of air voids in the concrete with ettringite. The filling process reduces the space for water to expand as it freezes, making the concrete microstructure more susceptible to freeze-thaw damage [27]. On the other hand, the study in [28] highlighted that the porous and rough ACBFS aggregate increased the contact area with the concrete matrix; thus, the concave holes and micropores on the surface of the slag aggregate could be filled with mortar and hydrated cement paste. This process can increase the interlocking and mechanical bond between the aggregate and the concrete matrix. ACBFS slag is used as a fine and a coarse aggregate in concrete [11,29,30]. The study in [31] reported that replacing coarse aggregate with up to 40% ACBFS and cement with 10% fly ash increased the concrete's compressive and flexural strength. The study in [32] showed that road concrete with coarse aggregate replacement by ACBFS and fly ash cement developed a 6% higher compressive strength, while the flexural strength decreased by 15% compared to the reference.

Coarse aggregate road concrete ACBFS showed a decrease in wear resistance in a study [33]. Due to the low resistance to the crushing and fragmentation of coarse aggregate in the slag, it is recommended to use them only in the road infrastructure base layer [12]. Regarding the use of ACBFS as a fine aggregate, the study in [34] highlighted better properties in geopolymer mortars than in Portland cement mortar. The influence of the replacement degree of up to 100% natural sand by ACBFS in the concrete composition showed no detrimental effects on the properties studied, such as workability and mechanical strength, or on durability, which recorded a reduction in porosity, water absorption, chloride permeability, etc., at the age of 90 days [11].

Most of the research has examined the individual effects of GGBS replacing cement and ACBFS aggregate replacing sand in concrete. The research results [35–38] show that GGBS does not significantly improve compressive strength up to 28 days but that it does significantly improve it after 56 and 90 days as the pozzolanic reaction of GGBS gradually continues. Moreover, the studies in [38,39] show that by using GGBS with a low reactivity index in mortar mixtures at older ages (90 and 360 days) lower porosity and lower water absorption were achieved. The 0–10 micron particles of the GGBS particle distribution were strongly correlated with the hydration activity index at different curing ages [40].The study in [41] highlights a reduction in the shrinkage of concrete at the age of 150 days when granulated slag was used with a reduced hydration activity index.

This study focuses on the durability of rigid pavements and the environmental benefits of using possible industrial waste as raw material in the mass of road concrete, while ensuring the transition to a circular economy. At least two benefits emerge from this research; the first is the reduction in the energy consumption needed for the additional grinding of slag powder, and the second is the conservation of natural resources. First, the effect of GGBS fineness on compressive strengths was investigated on two mortars with 50% GGBS with specific Blaine surface areas of 360 m²/kg and 330 m²/kg. The ratio of compressive strengths between slag mortars and standard mortar was recorded at 7 and 28 days. A detailed study of the microstructure of the mortars was carried out using X-ray diffraction, SEM electron microscopy with an EDX energy dispersive spectrometer, and NMR nuclear magnetic resonance relaxometry.

To further explore the blast furnace slag benefits, two pavement concrete compositions were studied; in these, the cement was replaced with 15% GGBS with a low Blaine specific surface area (330 m²/kg) equivalent to that of Portland cement, and the natural sand was replaced with 25% and 50% ACBFS. The tensile strength and durability properties were tested, including freeze–thaw resistance with and without deicing agent, abrasion resistance, resistance to carbonation, and chlorine ion permeability by the RCPT rapid test. The analysis of the results sought to establish the optimal percentages of using blast furnace slag in the composition of road concrete with performances equivalent to those of the reference concrete at the age of 28 days.

2. Materials and Methods

2.1. Materials

Portland cement-CEM I 42.5R, supplied by Romcim, CRH Romania Company, Romania, presents technical properties with values falling within the limits recommended by SR EN 197-1 [42]. The granulated blast furnace slag purchased from Steelworks Galati has a glass content of 95% and an oxide composition as given in Table 1, and the physicomechanical properties have been declared according to SR EN 15167-1 [43]. The superunitary ratio (1.07) of calcium oxide to silicon indicates efficient hydraulic activity, and the 70% concentration of oxides of silicon, calcium, aluminum, magnesium, and iron accumulated in GGBS indicates good pozzolanic activity according to standard D 6868 [38,44,45].

Table 1. Oxide analysis of granulated and ground GGBS slag.

(GGBS)	SiO ₂	Al_2O_3	MnO	MgO	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	Other
(%)	38.10	9.50	0.23	8.10	40.80	0.56	0.30	0.68	1.73

Air-cooled blast furnace slag aggregate ACBFS, crushed to 0/4 mm size, was purchased from Galati Steelworks. The quality assurance of the slag aggregate (0/4) mm; natural sand NA quality (0/4) mm; and gravel (4/8) mm was controlled according to SR EN 12620 [46]. The geometric and physical–mechanical properties of the crushed quarry chippings of grade (16/25) mm correspond to the requirements of SR 667 [47] and SR EN 13043 [48] for grade (8/16) mm. Figure 1a–e present the granulometric composition of the used aggregate, determined in accordance with SR EN 933-1 [49]. For the ACBFS, the slag aggregates are given in Figure 1f, photographic images before and after crushing.



Figure 1. Granulometric composition for aggregate: (**a**) natural sand 0/4 mm; (**b**) gravel 4/8 mm; (**c**) crushed stone 8/16 mm; (**d**) crushed stone 16/25 mm; (**e**) ACBFS 0/4 mm; (**f**) ACBFS slag during the air-cooling process and after crushing (Source: Galati Steel Works and the Technical University of Cluj-Napoca Laboratory).

The additives used were purchased from Master Builders Solutions Romania group, Romania, MasterGlenium SKY 527 superplasticizer additive, and Master Air 9060 air entrainer additive, with characteristics following SR EN 934-2 [50]. The water was taken from the Cluj-Napoca supply system, and the properties corresponded to SR EN 1008 [51].

2.1.1. Preliminary Mortar Mixtures

To assess the compressive strength, preliminary tests were carried out on the mortars with different specific GGBS surfaces. Then, the ratio of the compressive strength (CS) between the mortars with GGBS and the standard mortar was determined to show the influence of the specific surface area on the mortars; these experiments were similar to those of the study in [52]. The recipe for standard mortar M I with 100% Portland cement and two mortar recipes with 50% GGBS M II/360 and M III/330 and different specific surface areas of 360 m²/kg and 330 m²/kg were created in the experiment. The prepared mortars were stored in a polyethylene bag at a relative humidity of (90 ± 5) % for 7 days and then in a humidified air box at a temperature of (20 ± 2) °C and a relative humidity of (65 ± 5) %. As curing conditions, water according to SR EN 196-1 [53] or a wet environment according to SR EN 1015-11 [54] can be optionally applied [55]. For this experiment, we chose the method of curing the samples in a wet environment, which is less favorable for compressive strength development and closer to the in situ conditions. The water to binder ratio was 0.5, and the quantities of materials used to make the mortars are listed in Table 2. The prismatic specimens of $40 \times 40 \times 160$ mm were tested according to SR EN 1015-11 [54].

Table 2. The composition of mortars.

Materials	MI	M II/360	M III/330
Specific binder surface (SS)	331 m ² /kg	360 m ² /kg	330 m ² /kg
Portland cement, (g)	450 ± 2	225 ± 1	225 ± 1
(GGBS < 63 μm), (g)	-	225 ± 1	225 ± 1
$(NA_0/4 mm), (g)$	1350 ± 5	1350 ± 5	1350 ± 5
Water, (ml)	225	225	225

2.1.2. Concrete Mixtures

In the run-up to this work, the optimal proportions of the blast furnace slag material substitution were analyzed, and the material proportions were selected based on the various preliminary recipes in the study in [56]. We then proceeded with three paving mixes, one reference and two that were constantly incorporated with 15% GGBS blast furnace slag and 25% or 50% ACBFS slag aggregate crushed to a (0/4) mm size.

When preparing the concrete, the aggregate was saturated with the SSD dry surface. The initial moisture state of aggregate in concrete containing recycled materials has a major impact on the fresh and hardened state of the concrete. To avoid changes in the effective water–cement ratio and prevent the transfer of water from the mix to the porous aggregate, water balancing is generally suggested [57]. The quantities of material (kg/m³) for the concrete production are given in Table 3.

The road concrete design parameters were established for resistance class BcR 5.0, which corresponds to very heavy traffic, with casting in the sliding formwork system, according to the requirements of NE 014 [58] and SR EN 206 [59]. The test results for the concrete mix are shown in Table 4. It can be seen that the results were within the limits established by the applicable standards and that the compressive strengths in the preliminary recipes reached the minimum value of 50 MPa from the seventh day which are found in the previous work, [56].

Materials (Kg/m ³)	S 0/0 Reference Concrete	S 15/25 (15% GGBS_25% ACBFS)	S 15/50 (15% GGBS_50% ACBFS)
Cement ©	370	314.50	314.50
(GGBS < 63 μm)	-	55.50	55.50
Binder (b)	370	370	370
Water (w)	151.68	154.51	155.92
w/b	0.41	0.418	0.421
(ACBFS_0/4 mm)	-	163.44	326.90
Natural sand (AN_0/4 mm)	654.70	490.33	326.90
Coarse aggregate (AC_4/25 mm)	1215.88	1214.16	1214.18
Total aggregate	1870.58	1867.94	1867.97
Admixtures (SP-SKY 527)	5.55	6.18	6.29
Admixtures (MA 9060)	0.74	0.74	0.81

Table 3. Concrete composition kg/m^3 .

Table 4. Design parameters of road concrete composites in accordance with NE 014 [58] and SR EN 206 [59].

Parameter Design	Min. Dosage Cement	(w/l)	Compaction (%)	Density (kg/m²)	Occluded Air (%)	fcm 28 days MPa	fcfm 28 days MPa
NE 014 and SR EN 206	360 kg/m ³	max. 0.45	$1.15 \div 1.35$	2390 ± 30	$5.0 \div 6.5$	min. 50	min. 5.5
Results	370 kg/m^3	$0.41 \div 0.421$	$1.26 \div 1.33$	$2392 \div 2394$	$5.1 \div 6.4$	-	-

2.2. Method

2.2.1. Influence of GGBS on the Ratio of Compressive Strength of Mortars at 7 and 28 Days The CS ratio of mortars was calculated with Equation (1):

CS ratio =
$$(fc_{GGBS}/fc_C) \times 100, (\%),$$
 (1)

In the above relationship, (fc $_{GGBS}$) is the mean compressive strength (MPa) of the mortar made from 50:50 slag and cement, and (fc $_{\rm C}$) is the mean compressive strength (MPa) of the mortar made from cement, (Figure 2a,b). Table 5 shows the minimum hydration activity index values determined according to the specifications SR EN 15167:1 [43] and ASTM C989 [60], correlated with the resistance classes for GGBS.



Figure 2. (a) Preservation of mortar specimens; (b) compressive strength test on mortars; (c) mortar samples prepared for the NMR relaxometry measurements.

SR EN	15167:1	ASTM C989 (Class 80)		ASTN (Clas	ASTM C989 (Class 100)		ASTM C989 (Class 120)	
7 days	28 days	7 days	28 days	7 days	28 days	7 days	28 days	
45	70	-	75	75	95	95	115	

Table 5. The minimum value of the hydration activity index at 7 and 28 days of slag GGBS.

2.2.2. Characterization of the Microstructure of Standard Mortars and Mortars with GGBS

Phase composition analysis by X-ray diffraction (XRD) and microstructure analysis by electron microscopy coupled with energy dispersive spectroscopy (SEM–EDX) were performed on the fragments resulting from the compression test on the standard mortar (M I) and the mortar with 50% GGBS (M II/360, M III/330) at the ages of 7 and 28 days.

X-ray Diffraction on Mortars at 7 and 28 Days

XRD patterns were recorded on the powder samples using a D8 Advance diffractometer (Bruker, Karlsruhe, Germany) with CuK α 1 radiation (λ = 1.5418 Å), operating at 40 kV and 35 mA, at room temperature. The semi-quantitative evaluation was performed following the reference intensity ratio (RIR) method [61].

SEM Measurements on Mortars at 7 and 28 Days

SEM–EDX analysis was performed at room temperature using a scanning electron microscope (VEGAS 3 SBU, Tescan, Brno-Kohoutovice, Czech Republic) with a Quantax EDX XFlash (Bruker, Karlsruhe, Germany) detector. The ~4 mm² samples were mounted with carbon tape on an SEM stub.

2.2.3. NMR Relaxometry Measurements Made on Mortars, at 28 Days

From the intact prismatic samples prepared with standard mortar and mortar with 50% GGBS, a cylindrical sample with a length of 20 mm and a diameter of 9.5 mm was taken at the age of 28 days. The sample was used to extract the relative distribution of the pore sizes by the NMR relaxometry technique (Figure 2c). After oven drying, the samples were placed and sealed in 10 mm diameter glass tubes, and NMR measurements were performed to reveal the intra-C-S-H pores. To better highlight the inter-C-S-H and capillary pores, a second set of NMR measurements were performed on cyclohexane-saturated samples [62]. The NMR measurements were performed with a MinispecMQ20 low-field device (Bruker, Germany) using the CPMG technique (see Ref. [57] for a description). Before each measurement, the samples were brought to the thermal equilibrium at a temperature of 35 °C. A total of 2000 spin echoes were recorded in each experiment, and the time between two echoes was kept at 0.1 ms to mitigate the effects of internal gradients on the relaxation measurements. The relaxation time distributions were extracted from the CPMG series using a numerical Laplace inversion of the recorded data [63,64].

2.2.4. Road Concrete Mixes with Embedded Slag (GGBS and ACBFS), Tested at 28 Days

In this study, hardened concrete, mechanical strengths (flexural and compression), wear resistance, freeze–thaw resistance, permeable void content, corrosion resistance by chlorine ion penetration, and carbonation were evaluated. For each mixture, three $150 \times 150 \times 600$ mm prismatic samples were cast and kept in air for 24 h and then kept in water at (20 ± 2) °C until the reference age of 28 days. Subsequently, flexural tests were carried out according to SR EN 12390-5 [65], (Figure 3a,b). To determine the corrosion resistance by carbonation according to SR CR 12793, a 1% phenolphthalein solution was sprayed onto the freshly cleaved fragments [66] (Figure 3c). From the remaining prisms, 18 cubes with a side length of 150 mm were cut; with these, the compressive strengths were evaluated according to SR EN 12390-3 [67], and the loss of compressive strength between the water-preserved control samples and the samples tested in 100 freeze–thaw cycles was evaluated according to SR 3518 [68] (Figure 3d). For each mixture, 9 slabs with a cross-

section of 150×150 mm and a thickness of 50 mm were cut from the remaining fragments to estimate the mass loss by exfoliation in the presence of thawing agents (3% NaCl) after 56 freeze–thaw cycles according to SR assessment CEN/TS 12390-9 [69] (Figure 3e), and 9 cubes with a side length of 71 mm were used to assess the volume loss through wear according to SR EN 1338 [70] (Figure 3f,g). The permeable void content was evaluated in 71 mm cubes according to ASTM C 642 [71] (Figure 3h).



Figure 3. (a) Prisms $150 \times 150 \times 600$ mm; (b) bending tensile strength test; (c) prism fragments sprayed with phenolphthalein solution; (d) cubes of 150 mm sides maintained in the thermostatic chamber for 100 freeze-thaw cycles; (e) samples prepared to assess loss of mass through exfoliation; (f) abrasive material; (g) Böhme abrasive disc wear test; (h) weighing of samples with hydrostatic balance.

The concrete quality in terms of frost–thaw resistance was assessed against the criteria presented in Table 6, and the conditions for wear resistance are given in Table 7.

Table 6. Acceptance criteria for the frost resistance of concrete SS 13 72 44 [72,73], NE012-1 [74], and	t
SR 3518 [68].	

Frost–Thaw Resistance	Criteria SS 13 72 44	Criteria NE 012-1	Criteria SR 3518
Very good	$m_{56} < 0.10 \text{ kg/m}^2$		
Good	$m_{56} < 0.20 \text{ kg/m}^2$	$m_{56} < 0.50 \text{ kg}/\text{m}^2$	-
(High)	or $m_{56} < 0.50 \text{ kg/m}^2$ and $m_{56}/m_{28} < 2$	-	
	or $m_{112} < 0.50 \text{ kg/m}^2$		
Acceptable	$m_{56} < 1.00 \text{ kg/m}^2$ and $m_{56}/m_{28} < 2$	$m = \epsilon 1.0 \log/m^2$	
(Moderate)	or $m_{112} < 1.00 \text{ kg/m}^2$	$m_{56} < 1.0 \text{ kg/m}^{-1}$	-
Unacceptable	$m_{56} \geq 1.00 \ kg/m^2$ and $m_{56}/m_{28} \geq 2$	$m_{\rm ex} < 2.0 {\rm kg} {\rm m}^2$	
(Low)	or $m_{112} \ge 1.00 \text{ kg/m}^2$	$m_{56} < 2.0 \text{ kg/m}$	-
Degree of gelation G100	-	-	Loss of compressive strength $(\eta) < 25\%$

Table 7. Wear resistance classes SR EN 1338 [70].

Class	1	3	4
Mark Criteria	F No measured performance	H $\leq 20,000 \text{ mm}^3/5000 \text{ mm}^2$	I $\leq 18,000 \text{ mm}^3/5000 \text{ mm}^2$

To assess chlorine ion penetration, cylindrical specimens 100 mm in diameter and approximately 200 mm long were cast, from which 50 mm thick strips were cut for the RPCT test according to ASTM C 1202 [75]. The samples, protected on the side surface with epoxy material and covered with boiled and chilled water, were kept in the vacuum saturation apparatus with a vacuum pump and manometer for 20 h until fully saturated (Figure 4a). Immediately after completion of the RCPT test (Figure 4b), the chlorine ion migration coefficient was then determined on the same samples using the NT BUILD 492 colorimetric method [76]. A silver nitrate solution (AgNO₃) was sprayed at 0.1 mol/dm³ (0.1 M) onto axially split samples. After 15 min, a light gray precipitate of silver chloride formed on the surface of the sample on the part of the face where chlorine ions were present. The chlorine penetration depth (X_d) for each sample was obtained from the average of seven measurements taken at different positions across the sample width. Then, the migration coefficients of the chlorine ions (D) were calculated using the Nernst–Planck Equation (2) [76,77]:

$$D = \frac{0.0239(273+T)L}{(V-2)t} \left(x_d - 0.0238 \sqrt{\frac{(273+T)Lx_d}{V-2}} \right)$$
(2)

under:

D—Migration coefficient in non-equilibrium state ($\times 10^{-12} \text{ m}^2/\text{s}$);

V—Applied voltage (V);

T—Mean value between initial and final temperature in the anolytic solution, NaCl (°C); L—Sample thickness (mm);

X_d—Average chlorine penetration depth (mm).



Figure 4. (a) Vacuum apparatus and pressure gauge; (b) RCPT rapid test.

The correlation between the past electric charge Q and chlorine ion penetrability is shown in Table 8:

Table 8. Interpretation of results according to ASTM C 1202 [75].

Past electrical Charge (Coulomb)	High	Moderate	Low	Very Low
Penetrability of chlorine ions	>4000	2000-4000	1000-2000	100-1000

3. Results and Discussion

3.1. Mortar Mixes with GGBS

3.1.1. Influence of GGBS on the Ratio of Compressive Strength of Mortars at 7 and 28 Days

The mean values of the three specimens of the standard mortar M I and the slag mortars M II/360 and M III/330 that were tested for compressive strength and the CS ratios for the mortars are shown in Table 9.

Table 9. Compressive strength fc determined for mortars (MPa) and CS ratio (%).

Age	fc_MI	fc_M II/360	fc_M III/330	CS Ratio-MII/360	CS Ratio-MIII/330
7 days	34.40	22.16	20.34	64	59
28 days	43.62	40.26	35.14	92	81

The compressive strengths of the standard mortar and the test mortars are shown in Figure 5a. It can be seen that the compressive strength of the mortar samples increased with age; this was due to the continued duration of the hydration process and the reactions of the cement with the water in the pores and capillaries of the system [78]. The hydration reactions led to the formation of new hydration products, of which C-S-H occupied $50\% \div 60\%$ of the volume of cement paste after 28 days and was the main product contributing to the development of mechanical strength [79]. The CS ratio of the standard and GGBS mortars with the two specific surface areas at 7 and 28 days are shown in Figure 5b. The results recorded for the CS ratio in the two test mortars with GGB, as associated with the requirements specified in SR EN 15167:1 [43], classified the GGBS slag powder as being in resistance class 80 according to ASTM C989 [60]. With the specific GGBS area increase from 330 to 360 m^2/kg , the CS ratio increased by 8.47% after 7 days and by 13.58% after 28 days. The larger the specific surface area, the stronger the reaction capacity and the faster the increase in the hydration rate [14,40,80], which increased the strength of M II/360 slag mortar more compared to M III/330. The linear equations in Figure 4b show that the CS ratio could reach 100 for the specific area of $382 \text{ m}^2/\text{kg}$ at the age of 28 days and the specific area of $576 \text{ m}^2/\text{kg}$ at the age of 7 days under the conditions of the linear increase in the specific surface area, according to the results of the study in [40].



Figure 5. (a) Compressive strengths of mortars; (b) evolution of CS ratio by specific surface area of (GGBS) at the ages of 7 and 28 days.

3.1.2. XRD Measurements with X-ray Diffractometer Made on Mortars at 7 and 28 Days

The XRD patterns of the mortar samples at the ages of 7 and 28 days are presented in Figure 6.



Figure 6. (**A**) Diffraction patterns of M I; (**B**) Diffraction patterns of M II; Diffraction patterns of (a) M I, (b) M II/360, (c) M III/330 mortar samples at 7 days and (d) M I, (e) M II/360, (f) M III/330 mortar samples at 28 days.

The semi-quantitative analysis of the crystalline phases identified in the investigated samples is presented in Table 10. At the age of 7 days, quartz SiO₂ was the main phase observed; this was followed by albite NaAlSi₃O₈, portlandite Ca(OH)₂, ettringite Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O, calcium silicate hydrate CaH₂O₄Si, and orthoclase K(AlSi₃O₃) (Figure 5). At the age of 28 days, the diffraction peaks of portlandite and ettringite, which are characteristic of early cement hydration [81], were no longer observed.

Table 10. Phase fraction (%) identified by RIR method of mortar samples investigated at the ages of 7 and 28 days.

Mortar Mixture	Ν	ΙN	MI	I/360	MI	II/360
	7 Days	28 Days	7 Days	28 Days	7 Days	28 Days
Quartz	+++	+++	+++	+++	+++	+++
Albite (Ab)	++	++	++	++	++	++
Orthoclase	+	+	+	+	+	+
Calcium-silicate (C-S-H)	+	+	+	+	+	+
Portlandite (CH)	+		+		+	
Ettringite (C-A-S-H)	+		+		+	

+++ major phases (>20%), ++ minor phases (5%–10%), + phases in traces (<5%).

The crystallinity degree of the investigated samples at the ages of 7 and 28 days is presented in Figure 7. At the age of 7 days, the highest value was obtained for MI. Replacing the cement with 50% GGBS led to more disorder in the early-stage structure of the mortars. At the age of 28 days, the degree of crystallinity of MI decreased, probably due to the change in structure leading to the disappearance of the portlandite and ettringite crystals. For the mortars with the addition of GGBS, the degree of crystallinity increased. A possible explanation for this behavior could be the higher proportion of calcium silicate hydrates found in the concrete with GGBS [82] than in the concrete made with Portland cement only. Moreover, the higher degree of crystallinity in slag mortar M II/360 could be attributed to the increased pozzolanic activity of the GGBS particles with higher SS [83–85].



Figure 7. Degree of crystallinity of mortar samples at the ages of 7 days and 28 days.

3.1.3. SEM Measurements with Scanning Electron Microscopy and EDX with Energy Dispersive X-ray Spectroscopy Performed on Mortars Aged 7 and 28 Days

The pore size and morphology of the mortar samples M I, M II/360, and M III/330 at 7 and 28 days were studied by SEM and are shown in Figure 8. As can be seen, their surfaces were irregular and inhomogeneous, with pore sizes larger than capillary pores, which was characteristics of air holes (>a few μ m) [86,87].

Table 11 shows the sizes of the pores measured as well as the pore spacing on the samples studied at 7 and 28 days.



Figure 8. Cont.



(e) M II/360-28 days, 100 μm





[able 11. Pore measurements of morta	r samples M I, M II/360, and M III/330
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Sample	Pore Identification Code	Pore Radius (µm)	Distance Code Qi(Ci – Ci + n)	Distance (µm)	Pore Identification Code	Pore Radius (µm)	Distance Code Qi(Ci – Ci + n)	Distance (µm)
		7 D	ays		28 Da	ys		
MI	C1	5.90	Q1(C1 - C2)	24.01	C1	11.52	Q1(C1 - C2)	37.57
	C2	4.53			C2	6.28		
	C3	2.52			C3	10.96		
	C4	2.65	Q2(C3 - C4)	14.73	C4	8.61		
	C5	4.97			C5	8.51		
	C6	5.13	Q4(C5 - C6)	34.92	C6	5.49		
	C7	3.03			C7	3.42		
	C8	2.26	Q3(C7 - C8)	21.28	C8	3.20	Q2(C5 - C8)	45.33

Sample	Pore Identification Code	Pore Radius (µm)	Distance Code Qi(Ci – Ci + n)	Distance (µm)	Pore Identification Code	Pore Radius (µm)	Distance Code Qi(Ci – Ci + n)	Distance (µm)
		7 D	ays			28 Da		
M II/360	C1	7.02			C1	13.24		
	C2	3.83	Q1(C1 - C2)	21.81	C2	8.65	Q1(C2 - C3)	35.51
	C3	3.61	Q2(C3 - C4)	32.60	C3	11.39	Q2(C1 - C3)	124.04
	C4	4.19						
	C5	6.57						
	C6	2.41	Q4(C6 - C7)	15.24				
	C7	4.07						
	C8	2.09	Q3(C8 - C9)	11.26				
	C9	2.35						
M III/330	C1	8.75	Q1	10.70	C1	4.30	Q1(C1 - C4)	19.84
	C2	15.24	Q4(C2 - C5)	187.62	C2	3.02		
	C3	6.48	$Q_3(C_3 - C_4)$	28.96	C3	2.57	Q2(C3 - C4)	103.50
	C4	5.57	- · /		C4	2.05	/	
	C5	6.06	Q2(C1 - C5)	187.62	C5	2.41		

Table 11. Cont.

Larger air holes at smaller intervals indicate a more porous surface, and conversely, small pore sizes spaced at larger intervals indicate a more compact surface [69,70].

At 7 days, the standard mortar sample M I (Figure 8a) had the most compact surface, with air holes (from 2.26 to 5.90 μ m) at intervals (from 14.73 to 34.92 μ m). In comparison, the slag sample M II/360 (Figure 8b) had larger air holes (from 2.09 to 7.02 μ m) which were identified at smaller intervals (from 11.26 to 32.60 μ m). Sample M III/330 (Figure 8c) was characterized by the largest pore ranges (from 10.70 to 187.62 μ m) and sizes (r ranges from 5.57 to 15.24 μ m). The different morphology of the slag mortar surfaces at the early age of 7 days is explained by the slower hydration reaction and lower heat release rate in GGBS than in Portland cement [13]. The SEM results were in correlation with the compressive strengths; an increase in porosity in the M II/360 and M III/330 samples caused a decrease in the compressive strengths [88].

At 28 days, the M III/330 slag mortar sample (Figure 8f) showed the most compact surface with smaller air holes (from 2.05 to 4.30 μ m) at larger spacings (from 19.84 to 103.50 μ m). In comparison, for the sample standard mortar sample M I (Figure 8d), the air holes were larger (r ranges from 3.20 to 11.52 μ m), and the distances at the intervals were smaller (from 37.57 to 45.33 μ m). For the slag sample M II/360, (Figure 8e) we observed air holes at larger spacings (from 35.51 to 124.04 μ m) and pore sizes (from 8.65 to 13.24 μ m) close to the standard sample M I. The surface morphology of the M II/360 sample was consistent with the slightly lower compressive strength compared to the M I, which was justified by the fineness of the GGBS. But the denser surface in sample M III/330 contrasted with the compressive strength, which decreased the most compared to the standard mortar. This behavior may be due to the filling of the free pores and the ability of GGBS to reduce the pore volume of the matrix [89] or to the inhomogeneity of the mortar.

The EDX images are presented in Figures 9 and 10, and the results for the concentration of the identified elements are given in Table 12.



Figure 9. Surface mapping of (**a**) M I, (**b**) M II/360, (**c**) M III/330 mortar samples at 7 days, and (**d**) M I, (**e**) M II/360, (**f**) M III/330 mortar samples at 28 days.



Figure 10. Cont.



Figure 10. Cont.



Figure 10. Surface mapping of (**a**) M I, (**b**) M II/360, (**c**) M III/330 mortar samples at 7 days, and (**d**) M I, (**e**) M II/360, (**f**) M III/330 mortar samples at 28 days; (**g**) M I, (**h**) M II/360, (**i**) M III/330 mortar samples at 28 days.

Table 12. Element concentrations (%) in M I, M II/360, M III/330 obtained by mapping the mortar sample surface.

Mix	0		Ca		Si		Al		Mg		Ca/Si	
	7 Days	28 Days										
MI	51.69	49.51	38.87	33.47	6.90	14.60	1.15	2.42	1.39	-	5.63	2.29
M II/360	53.44	41.49	28.00	27.04	14.63	15.39	2.7	3.87	1.23	1.15	1.92	1.85
M III/330	53.63	46.96	28.44	32.63	14.69	14.66	3.24	3.25	-	1.47	1.94	2.23

In the three samples analyzed, the predominant element, along with oxygen, was calcium, followed by silicon. The calcium-rich gel formation mechanisms imparted strength to the cementitious materials [90]. Hydrated calcium silicate C-S-H contributed significantly to the macro-properties of the concrete, such as strength and durability [91].

In the slag mortars, these high calcium concentrations can be attributed to the formation of C-S-H calcium silicate hydrate gel in addition to the hydraulic and pozzolanic reactivity of GGBS [89]. In addition, Al and Mg compounds were observed in samples M II/360 and M III/330 since the GGBS contained higher amounts of aluminum and magnesium than the Portland cement [92,93].

Different variations in the Ca/Si ratio characterized the composition of the gels C-S-H [94]:

- For standard mortar M I with 100% cement, the value of 5.63 at 7 days old indicated the presence of most of the calcium-based structure, and the value of 2.29 at 28 days old indicated the presence of different forms of C-S-H there. As C-S-H gels are

amorphous [94], the EDX result was in accordance with the XRD data obtained for the crystallinity degree.

 For M II/360 and M III/330 with 50% GGBS, the values obtained in the range (1.85–2.23) at 7 and 28 days indicated the presence of different forms of C-S-H in the slag mortar samples [91,92].

The EDX measurements correlated with the 28-day SEM measurements, showing a more compact surface in the MIII/330 sample with 50% GGBS with smaller air holes and larger spacings compared to the standard mortar.

3.1.4. NMR Relaxometry Data at 28 Days

The evolution of the CPMG echo series in the empty mortar samples and cyclohexanesaturated samples at 28 days is shown in Figures 10 and 11.



Figure 11. CPMG echo series for cyclohexane-saturated bare mortar samples M I, M II/360, M III/330 at 28 days (note: S1 = M I; S2 = M II/360; S3 = M II/330).

A clearer interpretation of these results comes from using the inverse Laplace transform to reveal the distribution of the relaxation times extracted from the CPMG series [63,64], as shown in Figure 11. The distribution of the relaxation times in the bare mortar samples after oven drying and in the cyclohexane-saturated samples obtained by the inverse Laplace numerical analysis is shown in Figure 12.



Figure 12. (a) Distribution of relaxation times in empty samples; (b) distribution of relaxation times in cyclohexane-saturated samples at 28 days (note: S1 = M I; S2 = M II/360; S3 = M II/330).

The values for the transverse relaxation time T_2 plotted on the horizontal axis are proportional to the relative pore size, and the values plotted on the vertical axis represent the probability density that such relaxation times exist in the sample (here, a.u. stands for arbitrary units). The probable density indicates the probability of a certain relaxation time in the measured sample. In Figure 12, three peaks can be distinguished, the position of which can be assigned to the three types of pores: intra-gel pores C-S-H (Peack 1) with dimensions up to 2 nm; inter-gel pores C-S-H (Peack 2) with dimensions up to 10 nm; and capillary pores (Peack 3) with dimensions between 50 nm and 1 μ m [62].

The peak area is proportional to the amount of water or cyclohexane that is absorbed into the pores. The highest amount of water in the gel pores penetrating C-S-H (Peack 1, Figure 12a) was recorded in the slag mortar samples M II/360 and M III/330. But the gel pores in the cementitious materials had a protective effect due to their small size; the water in the pores of the gel did not freeze during the operation and was characterized by minimal permeability to liquids and gases. The C-S-H inter-gel pores, (Peack 2, Figure 12b) showed a similar relaxation time distribution in the three measured samples. The macro-capillaries with a radius in the range of $0.1 \div 10 \,\mu\text{m}$ were filled with water only in direct contact with water but had the ability of capillary condensation of moisture, making the cementitious materials hygroscopic. The main pores that caused damage to the structure of the cement stone were capillary pores [21,90].

The surface area of the Peack 3 in Figure 12b, corresponding to the capillary pores saturated with cyclohexane, shows that the test samples M II/360 and M III/330 with 50% GGBS absorbed the least amount of cyclohexane, and the sample of standard MI absorbed the greatest amount. These results indicated a lower capillary porosity compared to the standard mortar MI. The region of the transverse relaxation time distribution T_2 (on the Ox axis) moved slightly to the right at higher values (100 ms) for samples M II/360 and M III/330 compared to sample M I, which had a larger radius pore but a greater decreasing probability density below 0.01. In addition, it was observed that the maximum intensity (Peack 3) in sample M III/330 was slightly shifted to the left at the lower values compared to sample M II/360 which had smaller radius pores; the NMR results were consistent with those obtained by SEM–EDX.

3.2. Road Concrete Mixes with Embedded Slag (GGBS and ACBFS), Tested at 28 Days

3.2.1. Mechanical, Mechanical Wear, Freeze–Thaw Resistance, and Permeable Pore Content

The flexural tensile strength evolution at 28 days, the compressive strengths of the blanks and samples maintained in the thermostatic chamber, and the loss of compressive strength after 100 freeze–thaw cycles are shown in Figure 13. It was observed in all mixtures that the value of the tensile strength by bending in (Figure 13a) was above the limit of 5.5 MPa, which allowed the classification of the road concrete as class BcR 5.0, corresponding to very heavy traffic according to NE 014 [58]. However, the tensile strengths decreased slightly with the increasing dosage of ACBFS aggregate, by up to 4.1% for composite S15/25 and 10% for S15/50 compared to the reference mixture S 0/0. Regarding compressive strength, the S15/50 composite performed better than the S15/25 composite although it was up to 7.58% lower than the S0/0 reference mixture, (Figure 13b). It was also observed that the two slag mixtures had lower compressive strength losses at 100 freeze–thaw cycles than the reference composite S0/0 (Figure 13c), with values below the limit of 25% set in SR 3518 [68].



Figure 13. (**a**) The tensile strengths at 28 days; (**b**) the compressive strength of the reference samples and the compressive strength at 100 freeze–thaw cycles, (**c**) loss of compressive strength.

The total exfoliated mass S at 56 freeze–thaw cycles, the volume loss from attrition, and the permeable void content gave the results shown in Figure 14a. It shows an 8.9% increase in exfoliated cumulative mass in the S15/50; Figure 14b shows a wear volume loss of 5.5% in the S15/50 composite compared to the reference S0/0 composite. For the slag mixtures, however, the total value of the exfoliated mass S56 was <0.1 kg/m², which corresponds to a very good exfoliation resistance according to SS 13 72 44 [72,73] and falls into the same wear resistance class as the reference concrete (class 4, marking I, with values $\leq 18,000 \text{ mm}^3/5000 \text{ mm}^2$) [70]. In Figure 14c, the permeable pore content of slag compositions shows values above the reference composite S0/0.



Figure 14. (**a**) Total mass exfoliated after 56 freeze–thaw cycles; (**b**) volume loss after mechanical wear test; (**c**) permeable pore content.

Figure 14 shows the relationship between the permeable void content and compressive strength, the total mass exfoliated after 56 freeze–thaw cycles, and the volume loss through abrasion and tensile strength. It was found that a second-order polynomial relationship developed between these features with a very good correlation coefficient (R-value), with decreasing permeable void content, flexural strengths, and freeze–thaw resistance and a wear resistance increase (Figure 15a–c). The porosity increase justified the reduction in performance parameters since more porous concrete usually results in a lower degree of hydration and lower compressive and tensile strengths [24].



Figure 15. Relationship between pore permeable content and (**a**) flexural tensile strengths; (**b**) the total mass exfoliated after 56 freeze–thaw cycles; (**c**) loss of volume from wear.

3.2.2. Corrosion Resistance from Chlorides and Carbonation

The passing electric charges recorded values between 2273 and 3650 Coulombs for all the mixtures made (Figure 16a), which placed them in the moderate class of chlorine ion permeation, in accordance with ASTM C 1202 [75]. A discrepancy in RCPT values was observed with the S15/50 blend, which increased by 61%, while the S15/25 blend increased by only 16%. This observation needs to be investigated in a future study.



Figure 16. (a) Electric charge Q recorded at 6 h; (b) penetration depth Xd and migration coefficient Dx $(10^{-12} \text{ m}^2/\text{s})$ of chlorine ions; (c) the bonding relationship between the migration coefficient of chlorine ions and the content of permeable pores.

It can be seen that as the degree of substitution by ACBFS aggregate increases, the penetration depth of the chlorine ions and the migration coefficient calculated with the Nernst–Planck equation increased (Figure 16b). Due to the higher water absorption of the ACBFS aggregate, the water requirement to maintain specific workability increases at higher levels of sand substitution [11]. A higher amount of water led to an increase in the porosity of the S15/50 composite and the highest migration coefficient of chlorine ions compared to the reference mixture. The two features developed a directly proportional relationship, as shown in Figures 16c and 17, with images of the chlorine ion migration front.



Figure 17. (a) Chlorine ion migration front for sample S 0/0; (b) for sample S 15/25; (c) for sample S 15/50.

3.2.3. Corrosion Resistance from Carbonation

Both the reference samples (Figure 18a) and the slag samples (Figure 18b,c) were unaffected by corrosion by carbonation at the age of 28 days. The color of the indicator solution (red-purple) on the contour areas of the surfaces was kept identical to the interior of the test specimens. The diffusion of carbon dioxide from the cement matrix was impeded by the increased compactness of the concrete cement stone [95,96].



Figure 18. Carbonation progress one hour after spraying phenolphthalein solution: (**a**) sample S0/0; (**b**) sample S15/25; (**c**) per sample S15/50.

The reported results represented the average of the three samples tested for each mixture with the CoV coefficient of variation (%), as shown in Table 13.

Mix Coefficient of Variation	S0/0 CoV (%)	S15/25 CoV (%)	S15/50 CoV (%)
The tensile strength—fcf (MPa)	12.4	7.3	1.7
Reference compressive strength—fc (MPa)	5.0	9.0	1.0
Compressive strength at 100 freeze-thaw—fc (MPa)	11	8.0	5.0
The exfoliated mass at 56 freeze-thaw cycles (kg/m^2)	8	7.8	12.2
Loss of volume from wear (mm^3)	5.5	3.6	7.7
Permeable pore content (%)	11.3	3.3	11.2
Passing charge at 6 h (Q)	1.1	1.5	0.8
Penetration depth Xd (mm)	1.9	8.6	0.9
Migration coefficient of chlorine ions (D× $(10^{-12} \text{ m}^2/\text{s}))$	2.1	9.3	1.0

Table 13. Coefficient of CoV variation (%) of physic mechanical characteristics.

Note that the ratio of the standard deviation to the mean value obtained was below the accepted limit of 15% [97], with a reasonable quality in the range of 0.8% to 12%.

4. Conclusions

In this work, the influence of blast furnace slag (GGBS and ACBFS) on the physicomechanical properties at 28 days was studied in three road concrete mixes, as follows: S0/0, a blank sample (exclusively Portland cement), and two other mixes S15/25 and S15/50, in which the Portland cement was replaced by 15% GGBS and the natural sand by 25% ACBFS and 50% ACBFS.

First of all, before making concrete, studies were made on mortars. This method was very useful in finding the best direction for the study of concrete with different percentages of slag (GGBS/ACBFS).

The main results of the physico-mechanical and durability properties of the road concretes and mortars showed the following:

The report on the compressive strengths classified the GGBS powder as resistance class 80, relative to the criteria established by ASTM C989.

At 7 days, the XRD measurements showed the highest level of crystallinity in the standard mortar M I.

After 28 days, mortar M II/360 showed a level of crystallinity above the level of the standard mortar.

At 7 days, the SEM measurements indicated the most compact surface for the standard MI mortar.

At 28 days, the slag mortar M III/330 showed a compact surface. The surface morphology of the M II/360 was similar to that of the standard sample; results were also confirmed by the compressive strength and degree of crystallinity determined by XRD.

The EDX results complemented the SEM assessment and showed the presence of C-S-H in all the samples analyzed.

At 28 days, in M III/330, the Ca/Si ratio value was the closest to the standard M I.

The NMR results showed that the slag mortars M II/360 and M III/330 had the largest proportion of C-S-H intra-pores, which had a protective effect against the freeze–thaw strength due to their small size.

Due to the evolution of the CS ratio and a similar microstructure in both the M II/360 and M III/330 mortars, it was decided to use GGBS slag with a low specific surface, to create an ecological pavement concrete.

The physical and mechanical properties and the durability values of slag concrete S15/25 and S15/50 were:

Above the limit of 5.5 MPa, which allowed the classification of the road concrete as class BcR 5.0, corresponding to very heavy traffic according to NE 014 [58].

The tensile strengths decreased slightly with the increasing dosage of ACBFS aggregate, by up to 4.1% for composite S15/25 and 10% for S 15/50 compared to the reference mixture S0/0.

In terms of compressive strength, the S15/50 composite performed better than the S15/25 composite, although it was up to 7.58% lower than the S0/0 reference mixture.

In general, the results were 10% lower, and they were up to 16% in the RCPT test. However, they were at the same level as the quality criteria of the reference concrete. The low reactivity of GGBS and the increased porosity of ACBFS explain the reduction in resistances: mechanical, freeze–thaw resistance, exfoliation, wear, and chlorine ion penetration.

The pavement mixes with 15% GGBS and 25% or 50% ACBFS met the criteria for the assessment of the concrete quality as being very good in terms of exfoliation (S56 is <0.1 kg/m²) and carbonation, which corresponds to a G100 degree of gelation (with losses η < 25%), full placement into wear resistance class 4, brand I (volume loss values \leq 18,000 mm³/5000 mm²), and a moderate level of chlorine ion penetration.

This research shows that blast furnace slag containing 15% GGBS and 25% or 50% ACBFS is an effective substitute for cement and natural sand in road concrete for all traffic classes.

Furthermore, from an environmental and carbon footprint perspective, the sustainability of GGBS with a low specific surface area and an ACBFS fine aggregate is evident.

Although the current focus is on mechanical strengths at 28 days, as a reference for determining the strength class of concrete further investigations aim to investigate the influence of blast furnace slag on road concrete composition at the age of 90 days since the pozzolanic activity of GGBS slag lasts longer than that of Portland cement.

The authors are currently working on publishing results on the quality and sustainability of ecological BcR in terms of strength and exposure class.

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