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Properties and Uses of Biochars Incorporated into Mortars

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Abstract: The construction industry is responsible for a large amount of CO_2 emissions and an intensive energy consumption. Cement production is the third largest source of anthropogenic CO_2 emissions and is responsible for about 1.8 Gt of CO_2 emissions into the atmosphere. The use of waste materials to replace a fraction of cement in the mortar makes it more economically and ecologically friendly. In this work, the main objective was to test incorporations of biochar produced at temperatures of 300, 350, and 400 °C, as a partial replacement for cement in the production of mortar. The materials used for the tests were residual lignocellulosic biomass (WBL) and electrical cable insulation waste (WIEC) mixed in a ratio of 1:1. The biochars produced were crushed and sieved after production to reduce the particles. A sample of biochar was used and tested under these conditions and another sample was washed in water and dried before being incorporated; all tests were carried out with a 5% replacement. Waste recovery tests were also carried out without thermochemical treatment. The specimens were studied for compressive strength and water absorption by immersion. All tests were replicated and were analyzed and compared with a control mixture with no incorporation of biochar in the mixture. It was possible to observe that the tests with the incorporation of biochars at 400 °C showed better results, with only a 24% reduction in resistance to compression.

Keywords: waste valorization; biochar; concrete; WEEE; e-waste

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1. Introduction

Cement is considered one of the most manufactured products in the world, being composed of a combination of water and different mineral aggregates to form cement-based materials, such as mortar. After water, cement is the second most consumed substance in the world, as it is one of the materials that make up a considerable proportion of the building industry [1].

With the development of industries and cities, Portland cement has become one of the products of greatest interest since it is used in the construction of roads, bridges, buildings, and various infrastructures. There was a need to increase production 34 times in the last 65 years; on the other hand, in the same period, the population increased less than 3 times [2,3]. Carbon dioxide (CO_2) emissions are one of the main concerns of the civil construction industry; this concern is related to the intensive cement production process, thus causing an increase in energy needs and an increase in emissions [4].

The increase in demand for this material causes a greater consumption of natural resources and, consequently, an increase in greenhouse gas emissions. In 2020, the cement industry produced 0.59 tons CO_2/ton of cement, with production increasing by 1.8% between 2015–2020. The current scenario contrasts with the need for a 3% reduction by 2030 to follow the Zero Net Emissions Scenario by 2050 [5].

The search for sustainable development and the need to reduce emissions forces the cement industry to seek alternatives for the development of an application that uses more ecological materials in the production of cement [1,6,7].

As a way to improve the material efficiency and sustainability of Portland cement, we have (1) the use of alternative fuels to increase energy efficiency for the clinker process and production [8]; (2) the use of solid waste as an alternative to natural sources for the synthesis of clinker [9]; and (3) the partial replacement of Portland cement with supplementary commercial materials [10]. The partial replacement of Portland cement reduces production and therefore emissions without affecting the performance of the material.

The use of efficient reinforcing materials for concrete in the partial replacement of cement must also take into account environmental sustainability to obtain these materials; in this sense, the use of biochar (carbon material derived from waste) [11,12] may be an interesting alternative. Among the advantages of using this material, it is worth mentioning the low cost of production [13], the possibility of a wider use [14], highly adjustable properties [15,16], and the composition of free atoms. Regarding the mechanical, thermal, and acoustic properties, promising results were reported for the use of biochars [17–19].

Biochar was recently identified as one of the materials that can be considered multi-functional, as they have the potential for use in carbon sequestration and the reduction in greenhouse gases [20]. Biochar has properties that improve performance characteristics through cement incorporation, such as low bulk density, and low thermal conductivity, and the porous nature of the material presents different advantages for building materials [21]. The low density allows the production of lighter concrete because it can be used to displace the largest volumetric fraction occupied by denser materials such as Portland cement.

The low thermal conductivity and pores present in biochar material both serve to increase thermal insulation, resembling common insulating materials [22,23]. Biochar also creates voids and networks of pores interconnected to the concrete, forming a porous structure, and making the material's sound absorption greater [24]. Studies using biochar to reduce the amount of cement in concrete are limited. Choi, Yun, and Lee studied the behavior of mortar using biochar in partial replacement and found that a 5% incorporation of wood biochar improved compressive strength, and with higher amounts of incorporation, this strength decreased [25].

Restuccia and Ferro studied agro-food residues (coffee powder and hazelnut husk) pyrolyzed in different proportions of weight to incorporate in substitution of cement; the incorporations were 0.5, 0.8, and 1% of the weight and the results showed that in small amounts, with both materials, the mechanical properties of the cement were improved. The use of biochars increased the flexural and compressive strength as well as the fracture energy, with a more tortuous crack path that increased the final fracture surface [26].

Akhtar and Sarmah carried out tests with biochar derived from poultry litter, rice husk, and sludge from a pulp and paper mill to replace the percentage of cement, up to 1%. The results showed that the incorporation of 0.1% biochar from the sludge resulted in a compressive strength similar to the control specimen. Biochar from poultry litter and rice husk in the same proportion has already been shown to improve flexural strength by 20% compared to the control specimen. Based on the results, they concluded that the incorporation of biochar replacing cement improved the properties of concrete [17].

The structure that composes mortar made with geopolymer can be improved with the use of nanoparticles due to their ability to uniformly disperse the binder mass [27]. Nanoparticles are used in combination with other traditional materials to modify and improve properties [28]. Geopolymer materials have advantageous characteristics such as higher compressive strength, good fire resistance, low creep and shrinkage, better acid resistance, and durability properties [29].

Currently, alternatives are being studied for the manufacture of halloysite-based geopolymers filled with beeswax microparticles. Filling with beeswax has been demonstrated using techniques such as thermal analysis, spectroscopy, microscopy, and contact angle experiments. They concluded that only a large amount of beeswax was reliable in the flexural characteristics and water absorption capacities [28].

Another study was carried out incorporating biochar produced with rice husk and incorporated in various proportions in mortar. Tests of compressive strength, bending, water loss, permeability, and resistance to sulfate attack were carried out to investigate the mechanical and durability characteristics of the mortar. The results indicated that the incorporation of 4% biochar improves compressive strength by 2.32% and flexural strength by 23.52% [30].

This article aims to study the incorporation of biochar produced from electrical cable insulation waste (WIEC) and lignocellulosic biomass waste (WLB) [12], for the production of mortar. The biochars were incorporated to replace 5% of Portland cement, and compressive strength and water absorption tests were carried out to investigate the mechanical characteristics of the specimens and the influence of the incorporation of carbonaceous residues in the mortar.

2. Materials and Methods

2.1. Raw Materials for Carbonization

The materials used are part of the process for energy recovery; however, the volume of WIEC waste produced makes it an interesting product for use in the replacement of binders in mortar.

The WIEC was provided by a company that recycles electrical cables and uses noble metals. Waste is mainly composed of PP, PE, and PVC. The WLB was composed mainly of pine and was supplied by a biomass waste management company; both wastes are from companies located in Portugal.

2.2. Carbonization Experiments

The carbonization experiments were carried out based on the tests used previously [12], using temperatures according to Nobre et al. [31]. The mixture proportions used in the tests were 50% WIEC and 50% WLB. Briefly, 15 kg of the mixture was placed in lidded clay pots and heated at a heating rate of 10 °C/min to carbonization temperatures (300, 350, and 400 °C) for 2 h. For the carbonization tests, an electric furnace was used (Fornoceramic KS 72L, Leiria, Portugal) which had a capacity of between 12 and 20 kg per test depending on the properties of the raw materials to be used. The oven has a temperature and heating rate control panel and a capacity of 72 L in the carbonization zone. After the carbonization process, the biochars were ground and sieved to a particle diameter of less than 425 µm, using an Octagon digital sieve.

2.3. Biochar Washing for the Removal of Water-Soluble Compounds

After the screening process, the biochars were washed in heated water to remove soluble compounds and their improvement was evaluated [32,33]. For the washing, the biochars were placed in glass containers under heating and agitation plates with a ratio of 100 g/200 mL. The water was heated to a temperature of 95 ± 5 °C and the biochars were stirred for 30 min. After the process, the biochars were allowed to cool to room temperature, filtered, and dried in an oven (Holelab, Lapugnoy, França) at 105 °C for 24 h [33].

2.4. Characterization of Biochars

The biochars were characterized using elemental analysis, thermogravimetric analysis, and X-ray fluorescence analysis.

The elemental analysis of biochars was determined using a Thermo Fisher Scientific Flash 2000 CHNS-O analyzer (Waltham, MA, USA); the assays were performed in triplicate and the values shown are averages. The elemental composition influences the stoichiometry composed mainly of carbon, hydrogen, oxygen, and sulfur, as well as the level of disorder and the macrostructure [34].

Thermogravimetric analysis (TGA) was used to determine moisture, volatile fractions, fixed carbon, and ash [30]. Tests were performed in triplicate with sample weights between 3.5 and 4.5 mg. A PerkinElmer STA 6000 thermogravimetric analyzer (Waltham, MA, USA) was used, using a heating rate of 20 °C/min in an oxidative atmosphere without the addition of inert gas, and in a temperature range of 30 to 995 °C.

X-ray fluorescence (XRF) analyses were performed to monitor the mineral composition of the biochar used. Assays were performed in triplicate using the Thermo Scientific Niton XL 3T GoldD+ analyzer (Waltham, MA, USA).

2.5. Row Materials for Cement Mortar

River sand with a granulometry of between 2 and 5 mm, water, and Portland cement binder were used for mortars. Cement type CEM I 42.5 R was used as a binder, consisting of more than 95% clinker, and its characteristics are shown in Table 1. This cement is suitable for various applications such as industrial mortars, reinforced and prefabricated concrete, grout, fiber cement, underground works, geotechnical works, and particularly for environments that induce carbonation.

Table 1. Characteristics of CEM I 42.5 R cement.

Characteristics	Property	Specific Value
Chemicals	Loss on fire (P.F.)	≤5.0%
	Insoluble residue (I.R.)	≤5.0%
	Sulfates (SO_3)	≤4.0%
	Chlorides (Cl^-)	≤0.10%
Physics	Start of prey	(min) ≥60
	Expandability	(mm) ≤10
Mechanics	Compressive strength	
	2 days	20 MPa
	28 days	42.5 MPa

Source: CIMPOR [35].

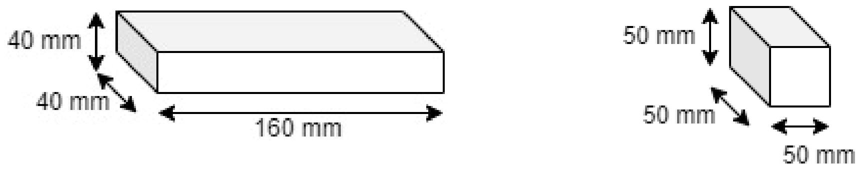
2.6. Experimental Procedure

To evaluate the incorporation of biochars in the specimens, 10 sets of tests were performed. These tests are described in Table 2. For the incorporation tests, a control mortar (CTL) was used, which determined the amount of water for the other specimens. The tests included the partial replacement of sand with WIEC residues without treatment and the mixture of WIEC with WBL (GW-50) that was used for the process of obtaining the biochars.

Table 2. Proportion of mixtures from mortar preparation to test pieces.

Reference	Description	Cement (g)	Sand (g)	Water (g)	Waste (g)	Biochar (g)	Ratio W/B
CTL	Control mortar	2000	6000	1100	0	0	0.55
GW-50	Mortar with 10% incorporation in place of sand	2000	5400	1100	600	0	0.55
WIEC		2000	5400	1100	600	0	0.55
B300	Mortar with 5% biochar incorporation in place of cement	1900	6000	1100	0	100	0.58
B350		1900	6000	1100	0	100	0.58
B400		1900	6000	1100	0	100	0.58
B300-L	Mortar with the incorporation of 5% of washed biochars in place of cement	1900	6000	1100	0	100	0.58
B350-L		1900	6000	1100	0	100	0.58
B400-L		1900	6000	1100	0	100	0.58

For the preparation of the mixtures, the solid materials were mixed in a tank to ensure uniformity, the water was added and mixed until a smooth and homogeneous mass was obtained using a Fort CP 230 vibrator (Barcelona, Espanhã). The mortar was then placed in molds (Figure 1) covered by polyethylene sheets for 24 h at room temperature. After demolding, the specimens were transferred to water tanks where they remained submerged for curing. The tests were carried out with the specimens cured at 7, 14, 28, and 56 days, and all tests were performed in triplicate. The device used for mechanical tests of resistance to compression was manufactured by the company TECNOTEST from Modena, Italy, product number P 4327TC, year of manufacture 2008.



(**a**) (**b**)

Figure 1. (**a**) Specimen dimensions for compression tests; (**b**) Specimen dimensions for water absorption tests by immersion.

The W/C ratio is normally between 0.4 and 0.7; a mortar with a 0.3 ratio will be very dry and difficult to work with, while a mortar with a 0.8 ratio will make the mixture weaker. Excess water not only influences the workability of the mortar but mixing it with excess water leads to increased shrinkage after drying and causes more cracking. The permeability of the mortar to gases, mainly CO_2 that forms carbonic acid and calcium hydroxide and increases corrosivity, strongly depends on the permeability of the mortar, which increases with the increase in the water/binder ratio (W/B) and the degree of hydration of the cement [36,37].

2.7. Compression Tests

The compression test has, as its main objective, the evaluation of the resistance of the specimens to compressive actions. For the compression tests, the two halves obtained in the bending test were used and the test was also carried out using Tecno-test press compression equipment. The test consists of placing one of the halves of the specimens on the lower plate of the press and applying a gradual force continuously until it breaks. The tests were carried out in accordance with EN 1015-11 (1999) [38] and calculated according to Equation (1).

$$f_c = \frac{F}{A_c} \quad (1)$$

where f_c corresponds to compressive strength (MPa), F is the maximum load at failure, and A_c is the cross-sectional area of the specimen to which the compressive force was applied (mm^2).

2.8. Immersion Water Absorption Tests

To determine the amount of water absorbed through the immersion water absorption test, an adaptation was made to the normative documentation LNEC E 394-1993 specification [39]. To carry out these tests, three specimens were used for each mortar–dough mixture, with dimensions as shown in Figure 1b.

To carry out the water absorption measurements through immersion, the specimens were placed in water for 28 days. To carry out the analyses, it was necessary to obtain the hydrostatic mass ($m2$), which corresponded to the weight of the specimens in the water, and the mass of the saturation state ($m1$), corresponding to the weight of the specimens after being removed from the water. After weighing the cubes, they were placed in an oven at 105 ± 5 °C until a constant mass was obtained, which corresponded to the weight of the dry specimen ($m3$). To calculate the water absorption by immersion, the formula below was used:

$$Ai = \frac{m1 - m3}{m1 - m2} \times 100 \quad (2)$$

3. Results and Discussion

3.1. Raw Material Characterization

The elemental analysis results for the materials incorporated in the specimens are presented in Table 3. The elemental analyses were carried out in triplicate and the results presented are the average of the values. The moisture and ash values were calculated from tests in a thermogravimetric analyzer. The oxygen contents of the samples were calculated by the difference between the total mass and the elements with the ash.

Table 3. Elementary composition of biochars as percentages.

Parameters	WLB	WIEC	B300	B350	B400	B300-L	B350-L	B400-L
Moisture (wt.% a.r.)	7.73	0.79	8.83	6.65	4.87	6.22	6.17	3.69
Ash (wt.% d.b)	23.55	33.20	43.26	39.1	33.85	33.4	33.6	34
Elemental composition (wt.% d.b.)								
C	37.52	52.3	40.77	42.64	43.95	42.38	43.06	43.14
H	7.9	2.5	4.02	3.74	2.83	2.81	3.33	3.76
N	2.29	0.2	5.08	4.24	4.01	12.9	11.38	10.41
S	<d.l.	<d.l.	<d.l.	<d.l.	<d.l.	<d.l.	<d.l.	<d.l.
O	47.02	42.7	6.87	10.28	15.36	8.51	8.63	8.69
Atomic composition (% at.)								
C	36.58	52.19	67.15	66.01	63.73	61.07	61.78	61.88
H	15.3	4.95	13.15	11.49	8.15	8.04	9.49	10.71
N	2.23	0.21	8.37	6.57	5.82	18.61	16.34	14.94
S	0	0	0	0	0	0	0	0
O	45.89	42.65	11.33	15.93	22.3	12.28	12.39	12.47

Biochar is a material derived from the recovery of solids and has a high amount of carbon [40]. The conditions under which they are produced influence the stoichiometric parameters, level of disorder, and macrostructure [34]. It is possible to observe that when the biochar production temperature is higher and when the condensable inorganic compounds that were on the surface of the biochar are washed away, the amount of carbon present in the biochar is higher. In addition, it is possible to observe that the carbon content of the biochars is inversely proportional to the ash content, i.e., the greater the amount of ash, the smaller the amount of carbon; a greater presence of ash hinders the formation of aromatic carbon [41].

Figure 2 shows the main inorganic compounds present in the biochars. The results were obtained through XRF analysis and the chemical elements are presented as typical oxides and presented in higher percentages.

3.2. Mechanical Compression Properties

The compressive strength tests for the test specimens in the hardened and cured state were carried out using three specimens of each mortar mixture. Figures 3–5 show the results of the averages of the compression tests. The graphs were divided for better visualization and comparison of the used biochars. Figure 3 corresponds to the compression tests for the specimens where biochars replaced part of the sand with residues without any type of thermochemical treatment (GW-50 and WIEC). Figures 4 and 5 correspond to the incorporation of biochars replacing cement and washed biochars, respectively, and the color scale presented relates to the strength in MPa. The graphs are presented according to the maximum strength applied (f_c) in MPa, the mortar mixtures, and the curing times.

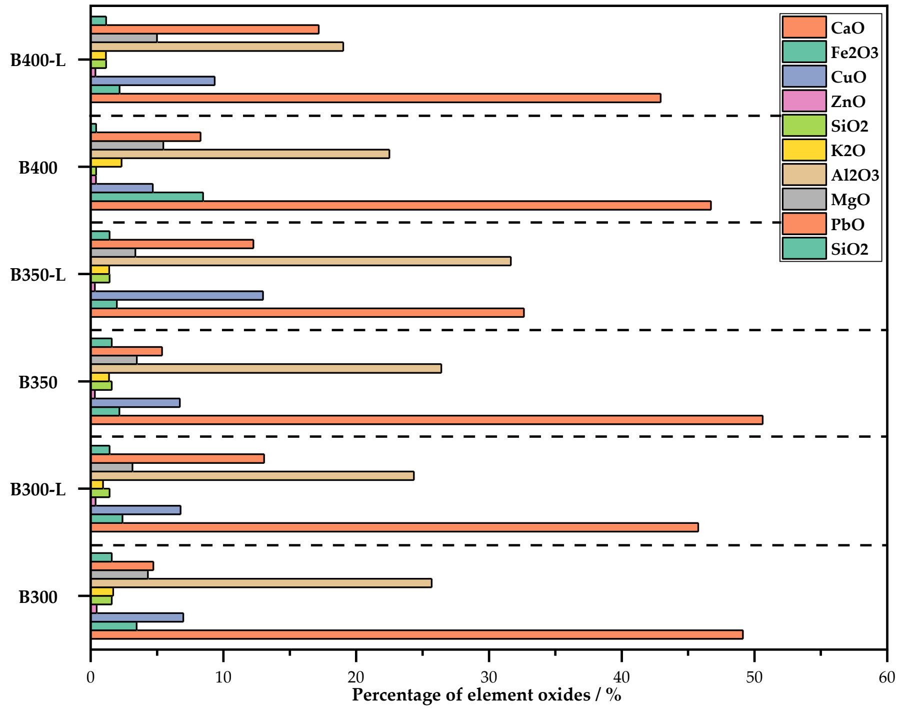


Figure 2. Composition of biochar ash elements expressed in oxides.

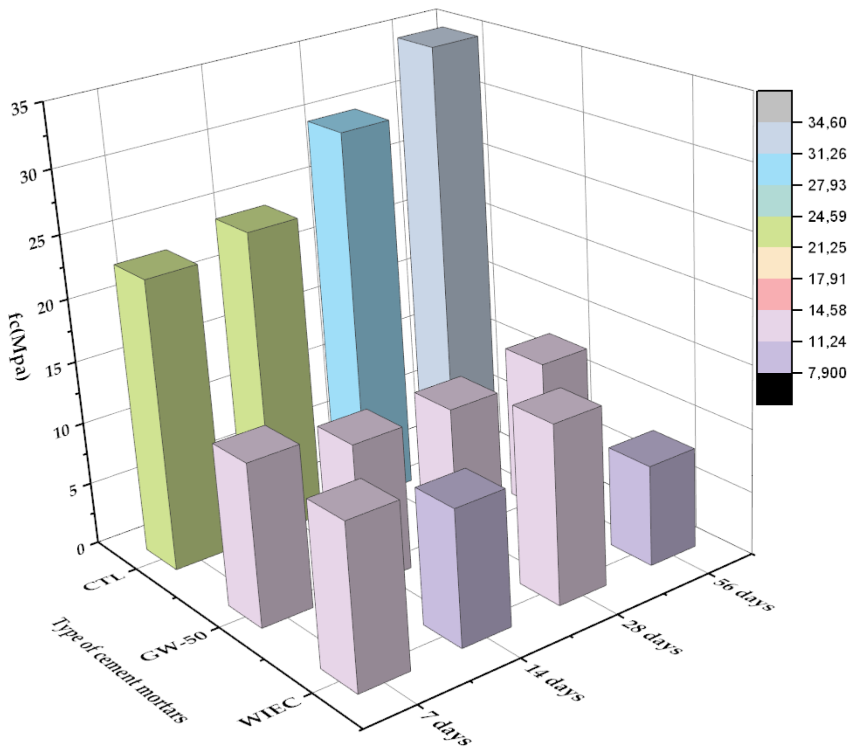


Figure 3. Compression test results for specimens with the partial replacement of sand with the GW-50 mixture and WIEC.

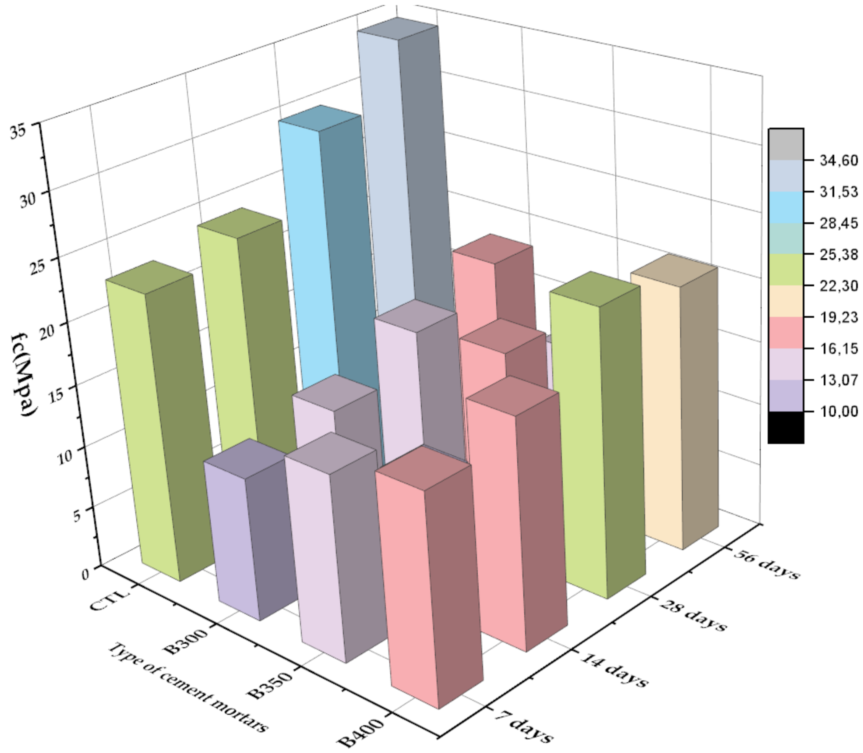


Figure 4. Compression test results for specimens with a partial replacement of cement with biochars produced at temperatures of 300, 350, and 400 °C.

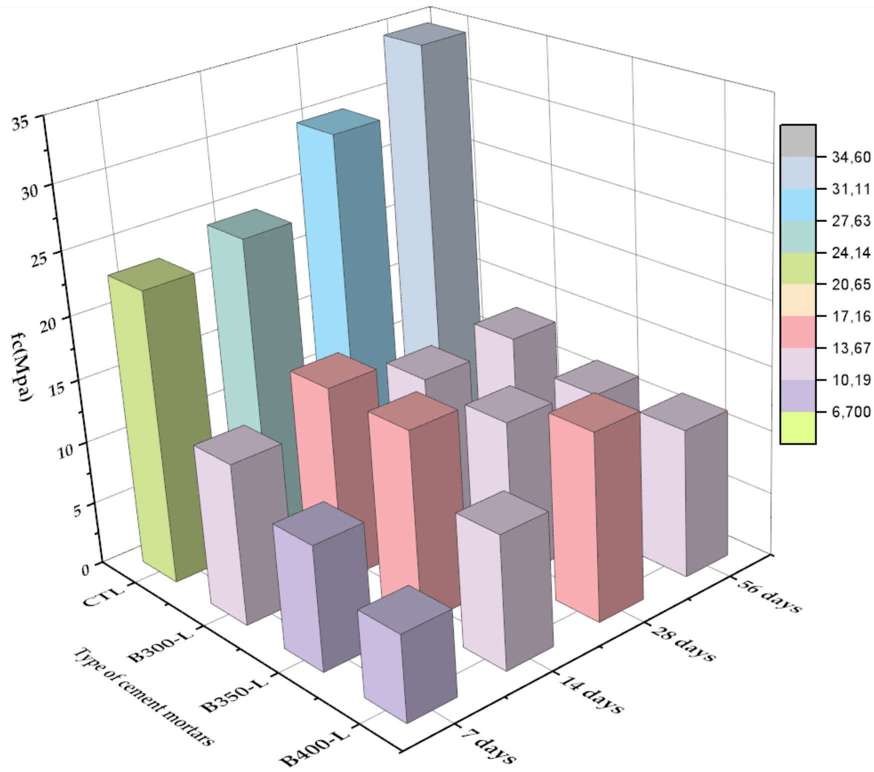


Figure 5. Compression test results for specimens with a partial replacement of cement with biochars produced at temperatures of 300, 350, and 400 °C after washing with water.

The control specimens, CTL, presented better results in the compression tests compared with the mixtures incorporating residues, demonstrating that the incorporation of residues reduces the capacity of standard resistance.

Regarding the tests with the partial replacement of cement that is presented in Figures 5 and 6, it is possible to observe that the incorporation of biochars reduces the compressive strength compared to the standard control at the beginning of the process. The higher carbon content causes larger pores to be added, which act as a weak link and tend to increase cracks during load applications [42]. Although the amount of carbon present in the biochar reduces the flexural strength, the increased incorporation causes the biochar density to increase, thus providing an increase in flexural strength [43].

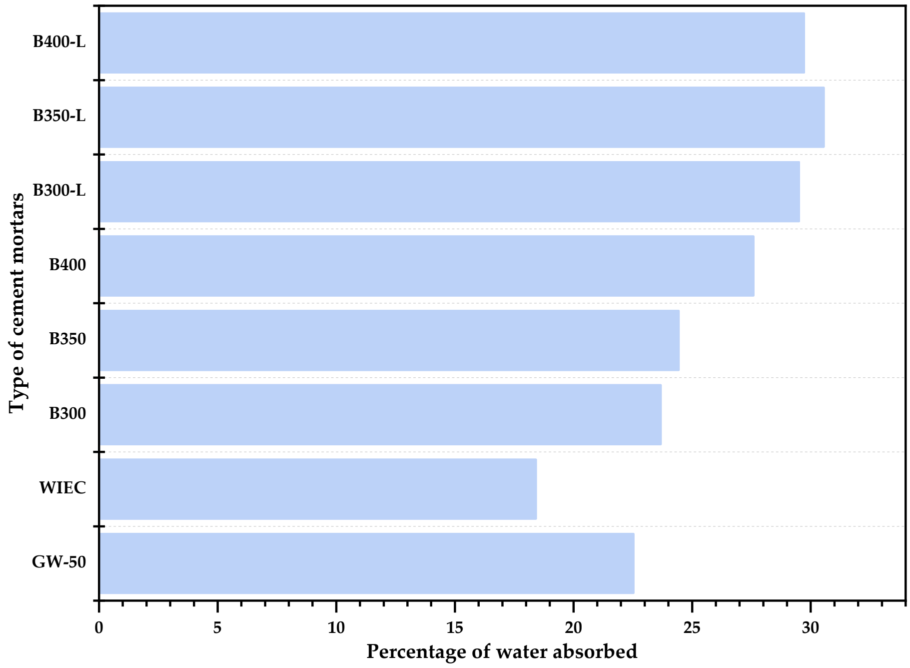


Figure 6. Water absorption by immersion for the different mixture specimens.

In the compression tests, it is possible to observe that the B400 mixture presented better resistance, despite still being lower than the CTL-2. The aggregates present in the composition of the biochars provide an increase in the resistance of the mixtures, which can be attributed to the microfilling effect and the porous structure that absorbs part of the kneading water, which is then released during the internal curing process that accelerates the hydration [17,44]. The compressive strength results are in agreement with other studies with the addition of biochars derived from waste. In general, when cement is replaced by a filler in mortars, the strength can be influenced due to the dilution and packing effects, contributing to a re duction [45]. Mrade and Chehab determined that part of the water used in the mortar mixture is removed from the biochar particles and, when released, cures the mortar from the inside to the outside, increasing compressive strength [46].

3.3. Water Absorption by Immersion

The results relating to the immersion water absorption tests are shown in Figure 6. It is possible to observe that specimens from mixtures with biochar have a greater tendency to absorb water when compared with current mortars, whose percentage of absorbed water is generally less than 10% [47,48]. Biochars, when produced at low temperatures, may contain more aliphatic compounds in the pores, which causes an increase in hydrophobicity [49,50].

The incorporation of aggregates from recycled materials in self-compacting concrete reduces the strength of the material, the resistance to chloride permeability, and the electrical resistivity, also increasing porosity, which leads to greater water absorption [51].

Although the presence of macropores in the microstructure of biochar particles can result in a reduction in water absorption and retention, they do not form a continuous capillary network in the mortar. The increase in empty pores in the mortar results in greater water absorption [20]. When biochars are washed, by removing compounds that are on the surface of the biochar, the surface area of the biochar becomes greater, making it lighter and more porous, thus being more likely to absorb water since the compounds that grant hydrophobicity to biochars have been removed.

Environmental damage related to the porous structure can affect mortar deterioration, caused by liquid transport in these pores. Water is one of the biggest problem factors related to deterioration because harmful components are easily carried by water into the mortar [52].

4. Conclusions

The use of biochar as a partial substitute for the binder in materials for civil construction shows interesting results. The use of biochar as part of the binder can improve the physical and mechanical properties of mortars. The use of biochar influences important parameters in the pore structure of mortars, such as porosity and water absorption, which significantly impact strength and durability. Thus, the present study was possible to conclude the following:

1. With regard to compressive strength and the incorporation of biochars, unwashed biochars showed better results than those that were subjected to the washing process; however, in both situations, there is a loss of compressive strength at 28 days, which for biochar produced at 400 °C, this was a loss of around 24% for the unwashed biochars and around 50% for the washed biochars.
2. Mortars produced with biochar present higher percentages of water absorption than control (current) mortars; this effect is amplified in the case of washed biochar.
3. Water absorption is proportional to the increase in biochar production temperature; however, with washed biochars, this difference is relatively smaller, varying by 1% between the three biochars.

The use of biochar in cementitious materials is of great importance for the reduction in emissions and in creating a more sustainable material. Many studies are being developed with different types of materials incorporated in mortars and concrete. For electrical cable insulation waste, it is necessary to carry out further tests to optimize the incorporated quantity.

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

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