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Improvement of biomass bottom ash properties by carbonation and pulverisation methods for application in cement-based materials

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ABSTRACT: This study evaluated the viability of biomass bottom ash (BBA) as a supplementary cementitious material (SCM) and sand substitute in cement-based materials. Physical and chemical characterization of BBA from eucalyptus forestry biomass and BBA from olive industry residues was performed. BBA underwent processing through grinding for SCM application and carbonation for sand substitution. Mortar mixtures were prepared with 25% ground BBA replacement for cement and 25% and 50% BBA replacement for sand, both carbonated and non-carbonated. Mechanical behaviour tested through flexure and compressive strength and water absorption at 28 days were assessed. Pulverised BBA enhance compressive strength of mortar compared to a conventional CEM II type cement and Carbonated BBA mortar mixtures improved 28-day compressive strength by 35-55% compared to non-carbonated BBA. BBA from eucalyptus forestry biomass, with pozzolanic activity, exhibited superior strength. The findings support the technical feasibility of BBA in cementitious materials, contingent on adequate processing.

KEY WORDS: Biomass bottom ash; Carbonation; Pulverization; Cement-based materials; Mechanical behaviour.

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RESUMEN: *Mejora de propiedades de cenizas de fondo de biomasa mediante métodos de carbonatación y pulverización para su aplicación en materiales base cemento.* Este estudio evaluó la viabilidad de usar cenizas de fondo de biomasa (BBA) como material cementante suplementario (SCM) y sustituto de arena en materiales base cemento. Se realizó una caracterización física y química de las BBA procedentes de biomasa forestal de eucalipto y de residuos de la industria del olivo. Las BBA se sometieron a un proceso de pulverización para su aplicación como SCM y a un proceso de carbonatación para su uso como sustituto de arena. Se prepararon mezclas de mortero con un 25% de BBA pulverizada como reemplazo de cemento y con un 25% y 50% de BBA como sustituto de arena, tanto carbonatada como no carbonatada. Se evaluó el comportamiento mecánico mediante ensayos de resistencia a la flexión y compresión simple, y se determinó la absorción de agua a los 28 días. La BBA pulverizada mejoró la resistencia a compresión del mortero en comparación con un cemento tipo CEM II convencional y las mezclas de mortero con BBA carbonatada mejoraron la resistencia a compresión a 28 días en un 35-55% en comparación con la BBA no carbonatada. La BBA de biomasa forestal de eucalipto, con actividad puzolánica, exhibió un desempeño mecánico mejor. Los resultados respaldan la viabilidad técnica de la BBA en materiales cementantes, condicionada a un procesamiento adecuado.

PALABRAS CLAVE: Cenizas de fondo de biomasa; Carbonatación; Pulverización; Materiales base cemento; Comportamiento mecánico.

1. INTRODUCTION

The prevailing modes of consumption and production exhibit a high degree of dependence on the exploitation of natural resources. This has resulted in a global escalation in the total volume of materials utilised, with projections indicating a further increase of 60% by the year 2060 compared to baseline levels established in 2020 (1). These practices trigger a cascading series of environmental harms throughout the entire life cycle of a product or service (2).

The traditional linear approach that extracts resources transforming them into products and discarding them as waste burdens the environment (3). The circular economy has emerged as an alternative that envisions a closed-loop system where discarded materials are reimagined as valuable resources emphasising reuse and recovery (3–5).

The circular economy offers a significant advantage through its ability to reclaim valuable materials from waste streams (6). A critical concept underpinning the circular economy is the designation of "end-of-waste" status (7). This status applies to materials that have fulfilled their initial product function but retain the potential to be reintroduced into the system (8).

In consonance with the burgeoning shift towards a circular economy, waste management policies are undergoing a substantial transformation. This transformation entails a departure from conventional landfill-based practices, prioritising instead the development of innovative solutions that utilise waste to produce renewable energy and recycled materials (9). Notably, the European Union actively promotes waste-to-energy processes as a means of generating renewable energy sources (10). Incineration, the predominant technology within the Waste-to-Energy sector, presents a potential avenue for mitigating dependence on fossil fuels and fostering a more sustainable approach to waste management (11–13).

The utilisation of biomass for energy generation offers a compelling advantage over traditional fossil fuels due to its inherent renewability, ensuring a dependable supply chain (14). Furthermore, the incorporation of waste materials into the energy production process aligns with the principles of the "cradle-to-cradle" design philosophy, which emphasises the continuous reuse of materials and promotes the development of a circular economy (15). This strategy is increasingly viewed as a potential pathway to achieve a confluence of enhanced energy efficiency and diminished waste landfilling (16).

The term biomass signifies a comprehensive category that incorporates a vast array of organic materials. These materials originate from biological processes and encompass a heterogeneous collection of residual by-products derived from diverse sources (17). Biomass for energy production comes in many forms. Agricultural residues like straw and stalks, leftover from farming activities, are a major source. Forests contribute wood waste from felling and maintenance, while animal manure and used bedding fall under livestock biomass. Fisheries and industries like food processing also generate usable waste. Municipal solid waste can be broken down into organic materials and wastewater, both potential sources. Even energy crops are included in the biomass category (18, 19).

Within the European Union's biomass sector, Spain is ranked as the fifth-largest producer of agricultural biomass particularly olive trees (20). Andalusia boasts a whopping 60% of the country's olive groves. Córdoba holds 22.47% of the olive grove area, while Jaén boasts an impressive 37.37% (21). This fact serves as a prime example of the potential inherent in utilising agricultural by-products as a sustainable source of energy and materials for diverse applications.

Biomass is emerging as a powerful force in the European Union's transition to cleaner electricity generation. In 2022, it achieved an impressive 11.2% share of the EU's total renewable energy production (22). Spain ranked thirteenth in Europe in 2022 for reliance on renewable electricity generation. Notably, renewables still contributed a significant 44% to the nation's total electricity that year. In Andalusia, a 2022 report found biomass specifically accounted for around 39% of primary energy consumption from renewable sources (22).

The combustion of biomass generates a burgeoning waste stream in the form of ash. Landfilling, the prevailing method for disposing of this ash, carries significant economic and environmental burdens due to its potential to generate harmful leachates and airborne particulates (16). Identifying methods for the utilisation of these ash residues is essential to guaranteeing the long-term sustainability and economic viability of Waste-to-Energy facilities (23).

Biomass combustion generates two main types of ash: fly ash and bottom ash. Fly ash, captured by filters in the exhaust stream (18), consists of exceptionally fine particles (mostly under 75 micrometres) (24) originating from both biomass and potentially coal combustion. Bottom ash, accumulating at the furnace bottom, is a sandy mix of unburnt and non-combustible materials like stones and soil (18). While fly ash originates primarily from the combustion of finer biomass particles in the flue gas, bottom ash forms from larger biomass pieces that undergo incomplete combustion in the furnace (25). Biomass bottom ash is rich in calcium and silicon (typically CaO and SiO_2) (26), elements commonly found in binding materials like cement. This chemical composition suggests that it could be a viable alternative to construction materials.

Biomass bottom ash (BBA) holds promise as a sustainable construction material. Studies show it can replace virgin materials in road embankments and cement-treated road layers due to its cementing properties (26, 27). BBA may also improve the stability of challenging soils (28). Additionally, research suggests BBA can be incorporated into concrete mixes as a partial substitute for cement and sand in non-structural applications, without compromising strength (29–31). Ongoing research explores its use in mortars, with pre-treatment methods like crushing showing promise (25).

One promising avenue for BBA utilisation explores its potential for carbon sequestration. Carbon sequestration refers to the capture and long-term storage of atmospheric carbon dioxide (CO_2). In the context of construction materials, carbonation presents a desirable process for CO_2 capture and storage. This process involves the reaction of CO_2 with a material, often leading to the formation of stable carbonate minerals. Two primary approaches to carbonation in construction materials exist: static and dynamic (32–34).

Carbon capture potential within the built environment hinges on a complex interplay between material properties and environmental conditions. Material characteristics like chemical composition, particularly a high content of calcium (Ca) and magnesium (Mg) for promoting stable carbonate formation, influence CO_2 uptake. Porosity and specific surface area, which are intricately linked, provide more reaction sites for CO_2 molecules, further enhancing capture capacity. Manufacturing processes can be optimised to manipulate these parameters for superior CO_2 capture. Environmental factors like humidity, crucial for carbonate formation, CO_2 concentration, the driving force for capture, and moderate temperature ranges all contribute to a material's ability to sequester and retain CO_2 (35).

Studies show incorporating biomass bottom ash (BBA) in concrete increases its carbonation rate, especially when partially replacing cement with BBA (36). This suggests BBA concrete could capture CO_2 and improve building energy efficiency. Separate research explored how carbonation affects concrete strength. Submerging carbonated samples in water after treatment resulted in significant strength gains (up to 45%) compared to standard curing (37). Similarly, pre-carbonated samples with water misting showed improved strength (32). These findings suggest carbonation, particularly followed by water curing, may improve the pore structure within concrete, potentially explaining the observed strength increase (38).

This research investigates using olive (BBA-OL) and eucalyptus biomass bottom ash (BBA-EU) as a partial replacement for sand and cement in mortars. This approach aims to promote sustainability by reducing reliance on virgin resources, lowering cement's CO_2 footprint, and diverting BBA from landfills. Additionally, the research will explore how carbonation treatment affects BBA properties and the resulting mortar's performance. This project seeks to contribute to sustainable construction practices by fostering a circular economy within the building industry.

2. MATERIALS AND METHODS

2.1. Standard natural sand

CEN Standard Sand is a specially designed material used in laboratory tests to measure the compressive strength of mortar. Its key feature is a tightly controlled grain size distribution, ranging from 0.08 to 2.00 millimetres. To ensure consistency, the sand is limited to a maximum moisture content of 0.2% and packaged in bags weighing 1350 grams, with a tolerance of +/- 5 grams. Table 1 show particle size distribution of sand according to EN 196-1.

2.2. Cement

Two types of cements were used, Portland cement type I of medium-high strength 42.5 MPa at 28 days with high initial strength and Portland cement with limestone type CEM II/B-L of medium strength 32.5 MPa at 28 days with normal initial strength. The main properties are shown in Table 2.

2.3. Biomass botton ash

Biomass bottom ash is the solid residue remaining after the combustion of biomass in a boiler or incinerator. This type of ash is found at the bottom of the combustion device and generally consists of the non-combustible materials that were not volatilised

Table 1. Particle size distribution of the CEN Reference sand (SNS).

Square mesh size (mm)	2.00	1.60	1.00	0.50	0.16	0.08
Cumulative sieve residue (%)	0	7±5	33±5	67±5	87±5	99±1

Table 2. Characteristics of cement.

	CEM I 42.5 R		CEM II/B-L 32.5 N	
Cement Characteristics	Standard	Usual	Standard	Usual
Clinker (%)	95 - 100	95	65 – 79	≥ 65
Limestone (L) (%)	0 – 5	5	21 – 35	≤ 35
Fly Ash (V) (%)	-	-	-	-
Steel Slag (S) (%)	-	-	-	-
Setting retarder, "gypsum" (%)	-	5	-	-
Minorities (%)	-	-	0 – 5	≤ 5
Sulphur trioxide (SO_3) (%)	≤ 3.5	2.9	≤ 3.5	≤ 3.5
Chlorides (Cl) (%)	0.10 max.	0.01	≤ 0.1	≤ 0.10
Loss on ignition (%)	≤ 5.0	2.9	-	-
Insoluble residue (%)	≤ 5.0	0.7	-	-

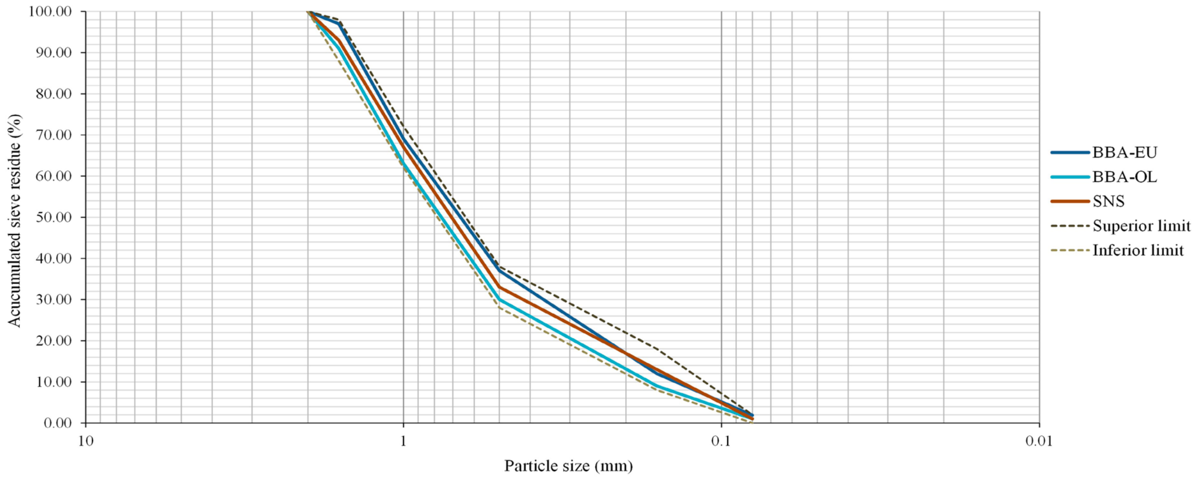


Figure 1. Particle size distribution of BBA-OL, BBA-EU and SNS.

during the burning process. The composition of bottom ash can vary depending on the type of biomass used and the combustion conditions, but generally contains minerals such as silica, aluminium oxides, calcium, iron and other trace elements. Two types of biomass bottom ash have been studied, one mainly from eucalyptus (BBA-EU) and the other mainly from olive trees (BBA-OL). The main properties of the raw material are shown below.

For this investigation a maximum particle size of 2 mm was taken, typically (0 – 2) mm.

As illustrated in Figure 1, both BBA-EU and BBA-OL exhibit continuous granulometry in the (0-2) mm range with a particle size distribution similar to that of SNS. Furthermore, BBA-EU has a greater proportion of fine aggregates. For instance, 70% of BBA-EU particles pass through a 1 mm sieve, compared to 65% for BBA-OL.

Table 3 summarises the physical and chemical properties of BBA-OL and BBA-EU. The carbonated versions of both BBA materials display a significant change in both density and water absorption compared to their non-carbonated counterparts. The carbonation process leads to a substantial increase in density (17% for BBA-OL and 14.7% for BBA-EU) and a drastic decrease in water absorption (89% reduction for BBA-OL and 97% reduction for BBA-EU). This transformation is particularly noteworthy for mortar mix design, as the higher density and lower absorption of carbonated BBA could significantly influence the amount of mixing water required to achieve optimal workability.

The major components, silicon (Si) and calcium (Ca), are present in significant quantities (over 20%), which are typical of good binder materials and contribute to the necessary reactions. However, the

Table 3. Chemical properties of BBA-OL and BBA-EU.

Properties		BBA-OL	BBA-EU
Density (SSD) kg/m3	not-carbonated	1.96	2.04
	carbonated	2.3	2.34
Water Absorption (%)	not-carbonated	12	10
	carbonated	1.3	0.3
Major components (%)	SiO_2	26.18	51.42
	CaO	28.19	9.67
	K_2O	14.45	8.49
	MgO	3.06	1.69
	Fe_2O_3	8.30	6.27
	Al_2O_3	16.27	18.16
	Na_2O	1.20	1.19
	TiO_2	0.47	0.55
Water-Soluble Sulphate			
(SO3)		0.173	0.07
(SO4)		0.207	0.09
Acid-Soluble Sulphate			
(SO3)		0.401	0.07
(SO4)		0.481	0.08
Organic Matter (%)		1.49	2.03
Chloride (%)		0.275	0.11
Loss on Ignition (%)		0.118	0.095

presence of potassium (K) raises concerns about alkali-aggregate reaction (AAR), which can cause expansion due to gel formation upon water absorption (39). The impact of this will be further investigated in the dimensional stability tests.

The levels of water-soluble sulphate and acid-soluble sulphate were below the detection limit. The organic matter content likely results from incomplete combustion, which depends on the power plant's efficiency (40).

2.4. Processed materials

2.4.1. Carbonated biomass bottom ash

Both biomass bottom ashes were subjected to static carbonation processes. Static carbonation consists of introducing the material into CO_2 chamber in which both the amount of CO_2 and the temperature and humidity are monitored. The material is deposited on trays inside the chamber without being stirred.

The carbonation process consisted of keeping the biomass bottom ash for 6 days in an airtight chamber with a CO_2 concentration of 15%, at a temperature of 21ºC and a RH of 65%. The material samples were weighed under oven-drying conditions before and after having undergone the carbonation process. Olive BBA showed a mass increase of 1.5% and eucalyptus BBA showed a mass increase of 0.5%.

Carbonation of eucalyptus and olive BBA showed the same trend in terms of density and water absorption, reducing water absorption to 11% and 3% of the water absorbed by uncarbonated BBAs and increasing density from 1.96kg/m^3 to 2.3kg/m^3 and from 2.04kg/m^3 to 2.34kg/m^3 for olive and eucalyptus BBA respectively. The increase in density and the reduction in water absorption when carbonating these materials are in line with other previous studies in which the density of carbonated materials increased by up to 4% and water absorption was reduced by 26% (41).

2.4.2 Pulverised biomass bottom ash

In order to obtain physical properties similar to cement, the biomass bottom ash from olive and eucalyptus were subjected to a process of crushing, sieving and grinding. The bottom ashes are more reactive by obtaining larger specific surface areas (31, 42)

Given the high friability ratio of these materials, the micronisation process to obtain BBA powder is not very costly in terms of energy.

First, the material was pre-crushed using a jaw crusher, then the material was fed into an aggregate abrasion testing machine, called Micro-Deval, with a ball charge of three different sizes and the BBA sample was subjected to 6000 revolutions per minute. 0.5kg of material and 5kg of abrasive load were introduced.. After this milling process, they were sieved through the 0.125mm sieve to obtain a powder with physical properties similar to cement. In figure 2 and in table 4 it can be seen how the grinding and sieving process makes the powder from BBA have characteristics similar to cement. The micronisation yields were 89.6% for olive BBA and 93.4% for eucalyptus BBA. In addition, to determine the pozzolanic activity of the BBA, the calcium quantity concentration through the Frattini test is presented in Figure 3.

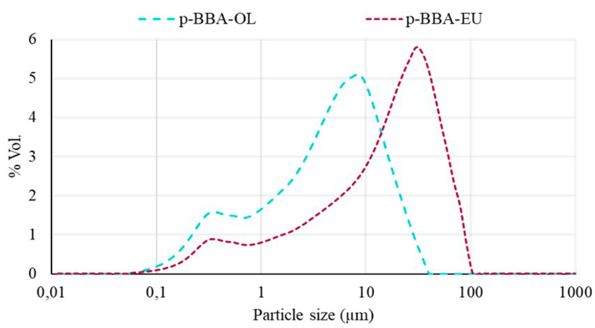


Figure 2. Laser granulometry measurements of the particle size distribution.

The particle size distribution and specific surface area of hydration hardening materials is an important quality. There is a direct relationship between particle size distribution, specific surface area and the physical phenomena that occur during the setting time (43). Other studies determine specific surface areas 2820cm^2/g and 6600cm^2/g for a particle size distribution that establishes that 8.85% and 30.5% of the particles are retained by the 10µm sieve. The mortar mixtures in this study determine that the higher mechanical strength is provided by the powder with higher specific surface area and smaller particle size, although it is not a definitive condition, since the chemical composition of these micronised materials, with reactive elements such as calcium or silicon, is a determining factor in their hardening capacity (44, 45).

The density of the powders from BBA is somewhat lower than that of conventional cement, in line with previous studies (46, 47, 48). The chemical composition of the ashes showed a high content of SiO_2, CaO

Table 4. Recycled powders physico-chemical characterization.

Properties	p-BBA-OL	p-BBA-EU
Grain size distribution, R_{30} (µm) (%)	0.32	26.06
Real Density (kg/m^3)	2589	2328
Blaine fineness (cm^2/g)	5070	2660
Organic matter content (%)	2.42	2.06
Acid-soluble sulphate (% SO_3)	0.131	0.049
Chloride content (%)	1.191	0.322
Main components XRF (%)		
SiO_2	33.09	63.82
CaO	40.41	12.07
K_2O	7.92	4.18
MgO	5.74	2.61
Fe_2O_3	2.80	5.96
Al_2O_3	5.29	8.81
Na_2O	0.38	1.33
P_2O_5	4.22	1.02
SO_3	0.15	0.20

and Al_2O_3 so that hydration reactions are expected to be strong. Other works presenting XRFs of conventional cements and alternative cements with recycled additions have CaO, SiO_2 and Al_2O_3 sum of 89.4% or 83.17%, while the pulverised BBA samples of this research determined this sum as 78.79% for p-BBA-OL and 84.7% for p-BBA-EU (49). A variation in the chemical composition of the original materials and the materials subjected to the pulverisation process was observed (table 3 and table 4). The screening of less friable particles that have not been pulverised in the milling process leads to the pulverised materials having a different composition to the original materials. The presence of SiO_2 and CaO was increased and the K_2O content was reduced, so that the hydration reactions are increased without significantly affecting the solidification rate. With regard to the presence of alkalis, which can be negative for the durability of cement-based materials, the amount of K_2O is between 8 and 4so there is no concern that this element is present at the amounts determined on p-BBA-OL and p-BBA-EU (50).

The results of the Frattini test showed that the p-BBA-EU is in the pozzolanic region in agreement with other previous studies in which bottom ash powder of organic origin were analysed (51). Other studies comparing organic waste used as biomass found that the calcination temperature can cause the ash to be in the pozzolanic or non-pozzolanic region. Calcination temperatures between 700ºC and 800ºC cause

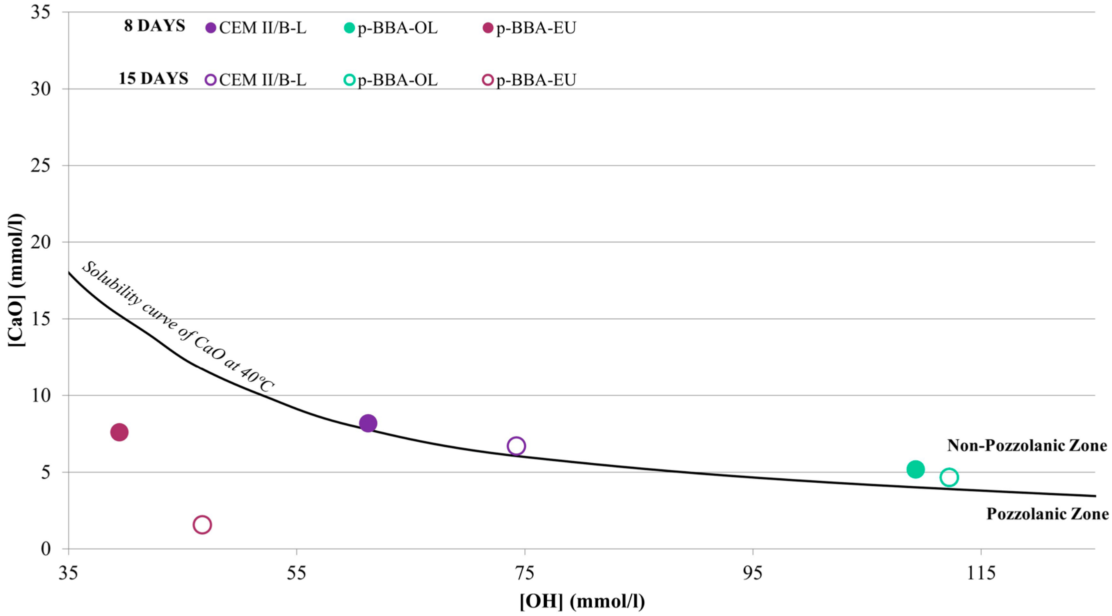


Figure 3. 8 days and 15 days pozzolanicity test.

the biomass bottom ash to be located in the pozzolanic region (52). Other studies determine that the crystallinity of the minerals presented in the samples is indirectly proportional to the pozzolanic activity that the material may present. (53). Pozzolanic oxides (SiO_2 +Al_2O_3 +Fe_2O_3) cause biomass ash to be located in pozzolanic or non-pozzolanic zones. p-BBA-EU has a sum of these three elements of 78.59% and p-BBA-OL presents a sum of 41.18%, being decisive for the area in which they are located, in accordance with previous studies that studied forest biomass ash (54, 55).

2.5. Experimental scheme and dosages

In this study, two industrial by-products (BBA-OL and BBA-EU) were applied as partial substitutes for cement and sand in the manufacture of mortars.

First, material processing was carried out, consisting of material carbonation (BBA-OL-CA and BBA-EU-CA) and material micronisation (p-BBA-OL and p-BBA-EU). With each of the materials obtained, the manufacture of cement mortars was carried out with partial replacement of sand by carbonated and non-carbonated biomass bottom ash and partial replacement of cement by micronised biomass bottom ash.

The mortars were manufactured in accordance with EN 196-1. Standard CEN-NORMSAD (SNS) sand was used in all of them, and two different cements were used, CEM II B-L 32.5N for the control mortar and for the mortars in which a partial sand substitution was applied and CEM I 42.5 R in which a partial cement substitution was applied. The reason why two different cements were used was because the addition of 25% biomass bottom ash was intended to obtain a cement with a clinker content similar to CEM II B-L. For the substitution of sand by BBA, the density of the material was taken into account, and the substitution was carried out in volume. The dosage of the manufactured mortars is shown in Table 5.

In the manufacture of each of the mixtures, it was considered necessary to obtain a workability similar to that of the reference mortar. In this study, no superplasticiser additive was used, so, to achieve this parameter, the amount of mixing water was increased. The amount of mixing water was increased by adding a quantity of water added to achieve the workability of reference mortar (workability water).

The flowability value of the fresh mortar was determined by measuring the extension after a standardised shaking process. The workability of the reference mortar was 170 mm. In order to obtain a workability that did not differ by more than 10% from the value obtained for the reference mortar, the amount of water to be added to each mortar mixture manufactured was evaluated (table 5).

For the manufacture of each of the mortars, the saturation water of each aggregate was added (water absorption), it was observed that it was necessary to use more absorption water in the mortars manufactured

Table 5. Cement mortar dosage expressed in grams of material used and workability (mm).

Mixes	Dosages Serie (g)											Workability (mm)
	SNS	CEM II/B-L 32.5 N	CEM I 42.5 R	p-BBA-OL	p-BBA-EU	BBA-OL	BBA-EU	BBA-OL-CA	BBA-EU-CA	Water (+ workability water)	Water absorption	
CEM II/B-L 32.5 N	1350	450	-	-	-	-	-	-		225	-	170
25p-BBA-OL	1350	-	337.5	112.5	-	-	-	-	-	225	-	171.5
25p-BBA-EU	1350	-	337.5	-	112.5	-	-	-	-	225	-	167.5
25BBA-OL	1012.5	450	-	-	-	251.5	-	-	-	225	29.8	161.5
50BBA-OL	675	450	-	-	-	502.9	-	-	-	225	59.6	172.5
25BBA-EU	1012.5	450	-	-	-	-	270	-	-	225	27.1	170.5
50BBA-EU	675	450	-	-	-	-	540	-	-	225	54.3	167.5
25BBA-OL-CA	1012.5	450	-	-	-			303.7		225+67.5	3.61	173.5
50BBA-OL-CA	675	450	-	-	-			607.5		225+135	7.22	175
25BBA-EU-CA	1012.5	450	-	-	-				309.4	225+20	0.54	169
50BBA-EU-CA	675	450	-	-	-				618.9	225+40	1.07	169.5

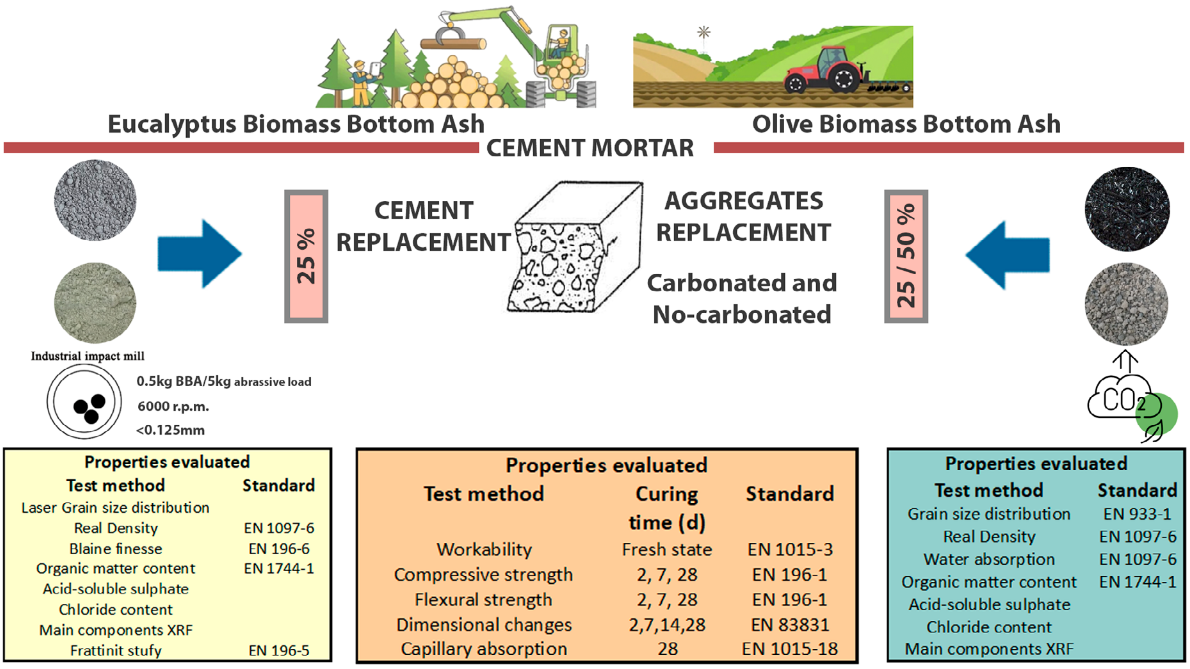


Figure 4. Experimental scheme.

with non-carbonated BBA. However, the carbonated ashes required more additional water to achieve a workability like the control. This added water may influence the mechanical properties subsequently obtained.

Figure 4 shows a scheme of the research development carried out, indicating the methodology applied to each material (processed and unprocessed) and the evaluated properties of the mortars manufactured with each standard used.

3. RESULTS AND DISCUSSION

3.1. Mechanical behaviour: Compressive and flexure strength (EN 196-1)

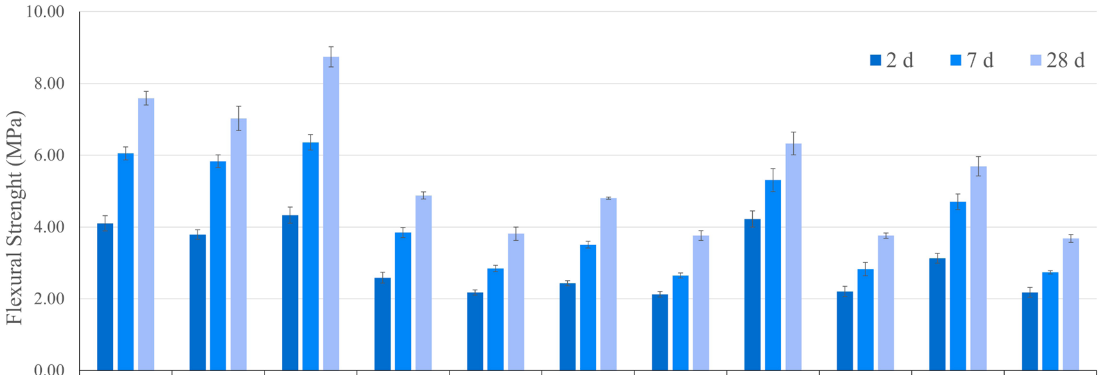
The mechanical behaviour of the mortar mixtures described in previous sections was evaluated through the determination of compressive strength and flexural strength at curing ages of 2, 7, and 28 days. Two specimens were tested for flexural strength determination and four for simple compressive strength determination.

Figure 5 shows the results obtained for the flexural strength test, and Figure 6 shows the results for compressive strength. Additionally, Figure 7 illustrates the effect of carbonation in biomass bottom ash on compressive strength values at 2, 7 and 28 days.

According to Figure 5, the 28-day flexural strength for series 1 mortar mixtures was 7.03 MPa for the p-BBA-OL mortar mixture and 8.74 MPa for the p-BBA-EU mortar mixture. Compared to the reference mortar (7.59 MPa), a slight decrease of 7.42% was observed for the p-BBA-OL mortar mixture and an increase of 15.15% for the p-BBA-EU mortar mixture.

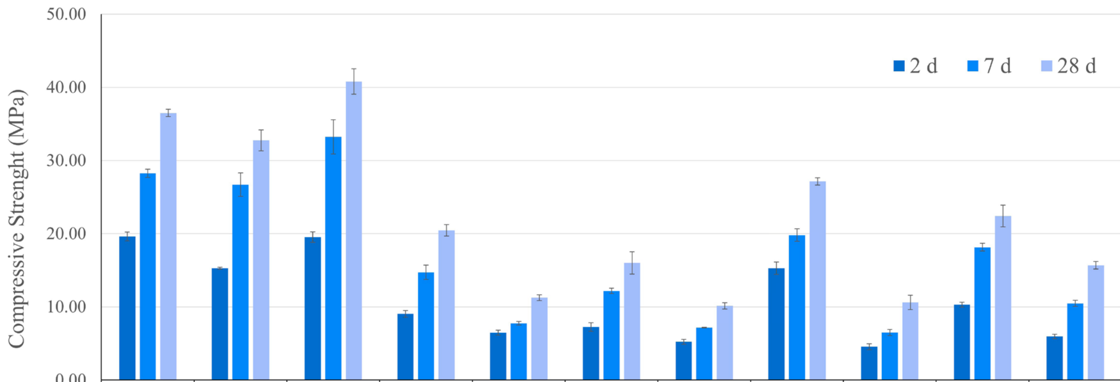
Regarding the 28-day simple compressive strength values obtained, as shown in Figure X2, series 1 mortars exhibited values of 32.76 MPa and 40.80 MPa for the p-BBA-OL and p-BBA-EU mortar mixtures, respectively. Compared to the reference mortar (36.50MPa), a decrease of 10.26% was observed for the p-BBA-OL mortar mixture and an increase of 11.79% for the p-BBA-EU mortar mixture.

Both mortar mixtures with a 25% replacement of cement by BBA showed positive values, with mechanical strengths comparable to conventional cement. However, the different mechanical behaviour for each type of BBA applied may be attributed to their composition. BBA-OL ashes presented high-



	Ref	Serie 1 - OPC remplacement		Serie 2 - Sand remplacement				Serie 3 - Sand remplacement carbonated			
	CEM II B-L 32.5 N	25p-BBA-OL	25p-BBA-EU	25BBA-OL	50BBA-OL	25BBA-EU	50BBA-EU	25BBA-OL-CA	50BBA-OL-CA	25BBA-EU-CA	50BBA-EU-CA
2 d	4.10	3.79	4.33	2.59	2.18	2.43	2.13	4.22	2.21	3.13	2.18
7 d	6.05	5.83	6.36	3.85	2.84	3.51	2.65	5.31	2.83	4.71	2.74
28 d	7.59	7.03	8.74	4.88	3.81	4.81	3.76	6.33	3.76	5.69	3.68

Figure 5. Flexure strength results.



	Ref	Serie 1 - OPC remplacement		Serie 2 - Sand remplacement				Serie 3 - Sand remplacement carbonated			
	CEM II B-L 32.5 N	25p-BBA-OL	25p-BBA-EU	25BBA-OL	50BBA-OL	25BBA-EU	50BBA-EU	25BBA-OL-CA	50BBA-OL-CA	25BBA-EU-CA	50BBA-EU-CA
2 d	19.62	15.28	19.53	9.04	6.46	7.25	5.22	15.29	4.56	10.29	5.95
7 d	28.25	26.71	33.23	14.71	7.76	12.18	7.16	19.80	6.50	18.15	10.48
28 d	36.50	32.76	40.80	20.46	11.26	16.00	10.13	27.15	10.62	22.42	15.69

Figure 6. Compressive strength results.

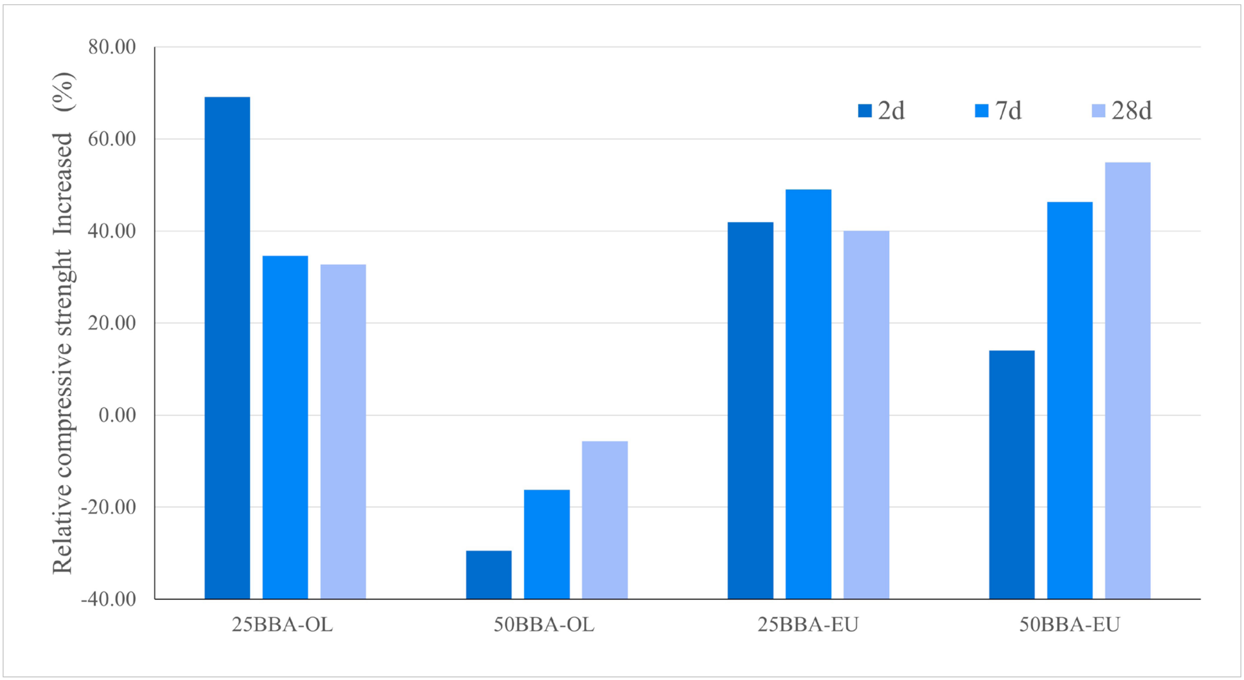


Figure 7. Effect of carbonated biomass bottom ash on compressive strength at 28 days.

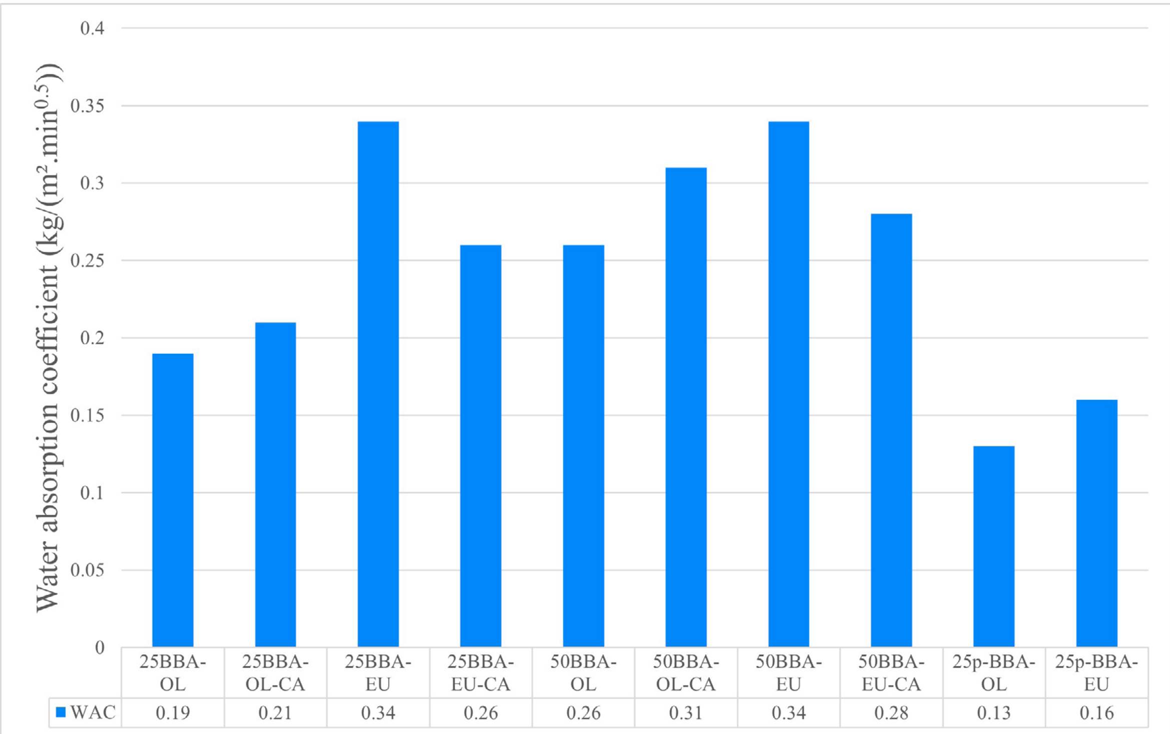


Figure 8. Water Absorption Coefficient.

er calcium and potassium values, whereas BBA-EU mixtures had higher Si content. The composition of BBA-OL indicated inert behaviour, similar to limestone filler, while the composition of BBA-EU suggested pozzolanic behaviour (56).

Analysing the data shown in Figures 5 and 6 for series 2, a change in behaviour compared to series 1 was observed. The 28-day flexural strength values range from 3.76 MPa (50BBA-EU) to 4.88 MPa (25BBA-OL), reducing the strength compared to the reference mortar by approximately 35% for mixtures with 25% replacement and 50% for mixtures with 50% replacement.

Compressive strength values ranged from 10.13MPa (50BBA-EU) to 20.46 MPa (25BBA-OL), with strength reductions of 40-50% for 25% replacement and 70% for 50% replacement.

This behaviour has been described in the literature by other authors and is attributed to the lower density of BBA, which increases the porosity of mortars, thereby reducing their mechanical strength. Additionally, the high-water absorption of BBA (10-12%) necessitated the addition of extra water during the mortar fabrication process, altering the water/cement ratio and reducing strength (57).

Comparing the values obtained for both types of BBA, mortars made with BBA-OL showed higher flexural and compressive strength values than those made with BBA-EU. This may be due to the granulometry of the BBA used as sand substitutes. BBA-EU contained a higher amount of material with a granulometry below 125 microns compared to BBA-OL. Rosales et al. (2023) (50) demonstrated that mortars with sand substitutions by BBA exhibited higher mechanical strengths when the fraction below 125 microns was removed.

Regarding the values obtained for series 3 in the flexural and compressive strength tests, shown in Figures 5 and 6, a similar behaviour trend to series 2 was observed, with strength reductions compared to the control. Analysing the 28-day flexural strength values, they ranged from 3.68 MPa (50BBA-EU-CA) to 6.33 MPa (25BBA-OL-CA). The strength reduction was 16% and 25% for mixtures with 25% replacement (BBA-OL and BBA-EU) and 50% for 50% replacement.

Analysing the simple compressive strength values, they ranged from 10.62 MPa (50BBA-OL-CA) to 27.15 MPa (25BBA-OL-CA), with strength reductions of 25% and 38% for mixtures 25BBA-OL-CA and 25BBA-EU-CA, and 70% and 57% for mixtures 50BBA-OL-CA and 50BBA-EU-CA.

Figure 7 shows the increase in strength over time due to the carbonation of BBA. The samples with 25% replacement exhibited a 32% (25BBA-OL-CA) and 40% (25BBA-EU-CA) increase in 28-day simple compressive strength. The 50BBA-OL-CA mortar mixture showed very similar strength values to the non-carbonated mortar, with a slight decrease of 5%. The 50BBA-EU-OL sample exhibits a 55% increase.

The positive effect of carbonation shown in previous studies (58) was not clearly seen in the compressive and flexural strength values obtained in this work. Mainly due to the need to use a higher amount of water in mortars with BBA-CA. The strength reduction in the 50BBA-OL-CA sample was due to the large amount of extra water added to maintain the workability of the sample, significantly increasing the water/cement ratio. The remaining mixtures exhibited a considerable increase in simple compressive strength at all ages due to the carbonation effect. Carbonation increases the density of BBA, reduces their absorption, and improves the physical properties of the mortars. Similar behaviours have been described applying carbonate recycled aggregates (59) .

3.2. Determination of the capillary water absorption coefficient of hardened mortar

The water absorption coefficient (WAC) due to capillary action was determined for hardened mortars containing BBA as a replacement for sand or cement, with and without carbonation treatment according to EN 1015-18. The calculated coefficients are presented below in Figure 8.

A preliminary analysis based on the percentage of aggregate replacement with BBA reveals that mixtures with 50% BBA exhibit higher water absorption coefficients compared to mortars with 25% BBA. Studies have demonstrated that the incorporation of highly porous industrial by-products significantly increases the porosity in hardened cement mortars (47) and that low substitutions (10-20%) can improve mortar durability by reducing water absorption due to decreased porosity. However, higher substitutions (30-50%) tend to increase these values (60)

As observed in table 2 and table 3, untreated BBAs exhibit high absorption percentages, and the accelerated carbonation process to which they were subjected improved this characteristic, resulting in significantly lower absorption percentages for BBA-CA. Based on the above and analyzing the data in Figure 8, it can be seen, particularly with BBA-EU samples, how the use of BBA-CA as an aggregate replacement reduces the WAC of hardened mortars, resulting in less permeable and more resistant mortars, as shown in figure 6 and previous research (61).

On the other hand, replacing part of the cement with BBA shows a notable decrease in WAC (62) compared to mortars where aggregates were replaced with BBA, with the p-BBA-OL sample presenting the lowest value at 0.13.

3.3. Dimensional changes

Dimensional changes of the mortar mixtures were recorded throughout the curing process following the guidelines of UNE 83831. Square section samples measuring 2 cm per side and 28.5 cm in length were used, and length variations were measured with micrometric precision at different maturity stages: at 2, 7, 14, and 28 days. During the hardening process, these specimens are embedded with two metal pieces which serve as reference points for subsequent length measurements. Once the specimens have been fabricated, their changes in length are measured using a length comparator. This comparator consists of an adjustable-height steel frame equipped with a micrometre with a precision of 0.003 mm. A steel bar is used to calibrate the apparatus. To evaluate the dimensional changes, all mixtures were subjected to two curing environments: one in a Dry Chamber (60% humidity and 20ºC) and the other underwater condition. The results of these changes are presented in Figures 9 and 10.

In the dry chamber, the mortars experience a loss of moisture to the dry environment, which causes significant contraction, as evidenced in most samples shown in Figure 9. This contraction results from the evaporation of water contained in the mortar pores, reducing the total volume of the material and generating internal stresses that contribute to further dimensional reduction. Specifically, for the present work, the dimensional changes were found between -697µm/m and 4189µm/m, with mortars containing BBA-EU substitutions, both carbonated and non-carbonated, showing the highest shrinkage values at 28 days.

On the other hand, when mortars are submerged in water, they have the capacity to absorb additional water, causing the material to swell due to the water entering the pores, which promotes hydration products and thus increases the total volume. The results presented in Figure 10 confirm this, as previously analyzed, with mortars containing higher percentages of incorporated BBA showing a higher water absorption coefficient and therefore presenting the greatest expansion values.

In summary, shrinkage in dry conditions is mainly due to moisture loss, while expansion underwater is attributed to water absorption and the continuous hydration of the cement. The dimensional changes of the mortar in these two environments depend on how the material interacts with moisture, the specific curing conditions, and the physicochemical properties of the substitutions in the mixtures.

It was observed that the dimensional changes produced in the mortars at early ages were more variable in dry chamber curing conditions than submerged, similar results were obtained in previous studies (Manu). The use of BBA as a partial sand substitute led to a higher dimensional instability in the mortars compared to the results obtained with the control mortar. The carbonation of the material produced a decrease in these dimensional variations, mainly due to the reduction of the absorption of the material at

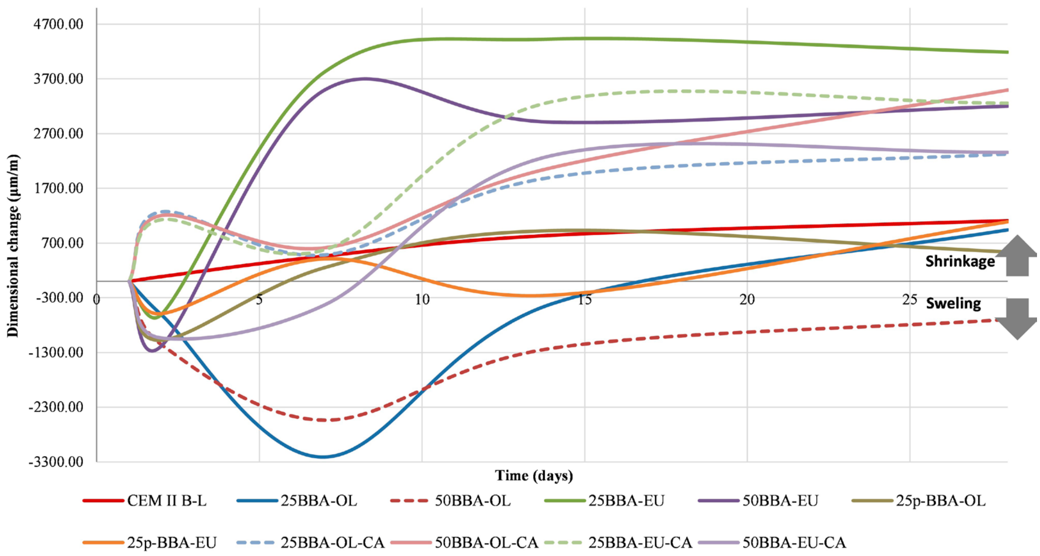


Figure 9. Dimensional change (dry chamber).

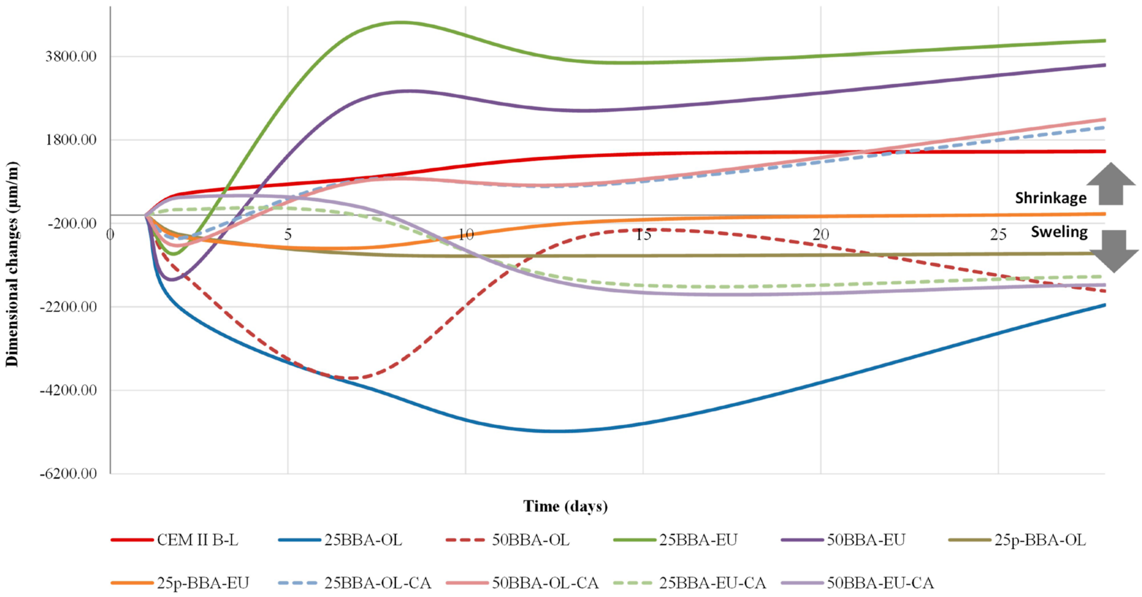


Figure 10. Dimensional change (under water).

source. The use of p-BBA as a partial cement substitute did not lead to significant modifications with respect to the control mortar (50).

4. CONCLUSIONS

The following conclusions can be drawn from this study:

- The application of ground biomass bottom ash (BBA) as supplementary cement material in a 25% proportion allows the production of cement with compressive strength values similar to or higher than those of a CEM II B-L. The BBA-EU exhibited values superior to the reference cement due to its demonstrated pozzolanic activity.
- The application of biomass bottom ash as a sand substitute results in a decrease in compressive strength due to the lower density and higher absorption of the particles.
- Carbonation of the BBA results in an increase of between 35-55% in 28-day compressive strength values compared to mortars made with non-carbonated BBA (25% replacement), due to improvements in the physical properties of the BBA particles.
- Carbonation of BBA results in a decrease in the workability of mortar mixtures, necessitating the use of extra water for their production. The use of additives could reduce the extra water required, thereby improving the mechanical properties of the mortars.
- Mortar mixtures with a higher percentage of aggregate substitution with BBA show increased water absorption.
- The use of BBA-CA reduces water absorption and enhances the strength of the mortars compared to non-carbonated BBA, specifically when using BBA-EU.
- The greatest dimensional changes under both curing conditions are observed when aggregates are replaced with BBA-EU.

As a general conclusion, the application of biomass bottom ash for the production of cement-based materials is technically viable. However, processing methods such as grinding, or carbonation are necessary to improve its physical and chemical properties for use as a supplementary cement material or as a sand substitute.

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Authorship contribution statement

Manuel Rosales: Data curation, Investigation, Writing - original draft, Formal analysis.

Leena Ahmed: Data curation, Investigation, Writing - original draft.

Reinier Rodríguez: Investigation, Writing - original draft.

Manuel Cabrera: Conceptualization, Writing - original draft.

Francisco Agrela: Supervision, Validation, Funding acquisition.

Santiago Moreno: Data curation, Writing - original draft.

Julia Rosales: Conceptualization, Supervision, Writing-review & editing.

José Luis Díaz-López: Conceptualization, Formal análisis, Writing - original draft.

Declaration of competing interest

The authors of this article declare that they have no financial, professional or personal conflicts of in-terest that could have inappropriately influenced this work.

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