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Article

Influence of the Mass Percentage of Bottom Ash and Its State of Maturation on the Mechanical Performance of a Bio-Composite

Asmahan Taleb-Ahmed ^{1,2,*}, Nicolas Montrelay ³ , Koffi Justin Houessou ³, Michèle Quéneudec-t'Kint ³, Nassim Sebaibi ²  and Rose-Marie Dheilly ¹ 

¹ Laboratoire des Technologies Innovantes, Department of Civil Engineering, Picardie Jules Verne University, 80000 Amiens, France; rose-marie.dheilly@u-picardie.fr

² Builders Ecole d'Ingenieurs, Builders Lab, ComUE NU, 1 Rue Pierre et Marie Curie, 14610 Epron, France; nassim.sebaibi@builders-ingenieurs.fr

³ Research Unit Eco-Processes, Optimization, and Decision Support, Picardie Jules Verne University, 7 Street Moulin Neuf, 80000 Amiens, France

* Correspondence: talebahmedasmahan@gmail.com

Abstract: This study examines the influence of the mass percentage and maturation stage of bottom ash on bio-composite mechanical strength. Two kinds of bottom ash were used: matured and non-matured. To elaborate the composites, several different percentages of bottom ash were mixed with an organic matrix. Casein, starch, alginate, polyethylene glycol, pre-vulcanized natural latex, and water are the components of matrix. The idea was to use as much bottom ash as possible, since it can be used as 80% or more as the main charge, which was in prismatic form for mechanical testing after drying. The results show that whatever the state of maturation of the bottom ash, the resistances present a maximum for a percentage of bottom ash equal to 85%. It could also be noticed that non-matured bottom ash composites have better mechanical strengths than composites with matured bottom ash. This is due to the decrease in porosity of the composites and to the improvement in their structural integrity. These new composites could solve some of the solid waste problems created by bottom ash production. As the matrix is made from organic resources, it saves energy and reduces the carbon footprint. This bio-composite contributes to the circular economy by giving waste a second life.

Keywords: bottom ash; bio-sourced matrix; bio-composite; maturation



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1. Introduction

Bottom ash, solid waste resulting from the incineration of non-hazardous waste, has long been abandoned in landfills, posing environmental and logistical problems. However, due to the growing awareness of the environmental consequences of this practice, various works have been undertaken over the past thirty years to transform this potentially hazardous waste into a valuable resource. The results of these studies have led to the promotion of the use of alternative materials in road technology under controlled environmental conditions. Several countries, including France, Belgium, Sweden, Finland, the Netherlands, and Japan, have developed specific techniques to use bottom ash as materials for subgrade and embankment layers in the construction of roads, structures, car parks, and other things [1,2]. The drawback of this recovery method is the maturation period, which is necessary to stabilize the bottom ash. This step generally lasts between 2 and 4 months on average, allowing for the chemical stabilization of the bottom ash through the carbonation of the lime it contains.

However, since the beginning of the 21st century, in order to meet the community objectives of circular economy and waste recovery, additional requirements must be met to avoid any risk to health and the environment. Other sustainable recovery methods of bottom ash have also been studied, in particular to reduce the volume of stored bottom ash

and maximize the efficiency of this resource. Regarding the extraction of natural aggregates, which also has an impact on the environment, especially on wildlife and flora [3], studies have been conducted to determine the potential to use bottom ash as a partial or total substitute for natural aggregates in the preparation of cementitious materials. Percentages have varied from 10 to 50% depending on the desired characteristics and the desired concrete performance. Provided that the size of the bottom ash is less than or equal to 6 mm and does not exceed 30% by mass of sand substitution with bottom ash, the studies of [4–8] have shown that concrete made with bottom ash is lighter than ordinary concrete with equal performance. The work of Perra et al. [9] and of Pecqueur et al. [10] showed that concrete is firmer when bottom ash is substituted for 50% of the mass of sand. Cohesion is not impacted, as compared to ordinary concrete. In any case, it is necessary to wait 28 days to obtain reliable and accurate results of the properties and performance of the cementitious materials thus developed [11]. Some reactions can then occur and lead to concrete cracking or swelling of concrete structures [12]. In 2020, works of Courard and Martin [13] proved that the setting time for bottom-ash-based concrete is faster, despite the higher air content, and that this does not affect the strength, which increases in the long term. To achieve the best mechanical performance in concrete made from bottom ash, it is essential to carefully pre-treat these materials before they are used. This treatment begins with a thorough sorting process designed to eliminate undesirable elements, such as metals, organic matter, or other impurities, which could adversely affect the quality of the final concrete. This ensures that only the most suitable and homogeneous elements are incorporated into the mixture. Secondly, the grinding of the bottom ash plays an important role. Depending on the application, it may be necessary to reduce the granulometry of the bottom ash to obtain a fine, regular texture, as was applied in the various studies cited. Proper grinding improves the integration of bottom ash into the cementitious matrix, thereby increasing inter-particle adhesion and overall material cohesion. This optimization of bottom ash processing can lead to a significant improvement in the mechanical properties of concrete, such as compressive strength and durability. Indeed, studies have shown that these treatments can potentially double or even triple concrete performance, compared with concrete made from untreated bottom ash [14]. This makes the treatment process essential for anyone seeking to maximize the structural capacities of bottom-ash-based concrete, while at the same time reclaiming a waste material in an efficient and sustainable way. This same preparation of bottom ash is also important for improving its use in road engineering, as Mkahal [15] demonstrated in her research [16,17].

In all these works, cement is used as a binder. However, cement is obtained from fossil resources, through a high-temperature thermal process, around 1300 °C, which consumes energy and generates large amounts of CO₂ [18]. Just like in the extraction of aggregates, the production of cement is therefore not without impacts on the environment. It also suffers from resource depletion due to population growth over the past 50 years. In order to best respect the goals of the circular economy, which encompass the environment, the economy, and society, and to optimize resource management, the objective set for this study is to explore the feasibility of substituting 100% of the cement with an organic binder and using only bottom ash as the aggregate. However, the matrixes usually used in the production of composites are based on formaldehyde, polymethyl methacrylate, a polymer of unsaturated polyester, and epoxy [19]. Some of these matrixes are expensive or toxic, such as formaldehyde, as it is a volatile organic compound [20,21]. Polymers with binding properties derived from natural resources are therefore being increasingly studied [22–25].

Thus, the bio-based matrix used for the development of the composites is a biopolymer matrix that was developed at the University of Picardie Jules Verne and has been incorporated in mass proportions of 10 to 15%. The influence of the maturation state of bottom ash has also been examined.

The results of this study highlight the opportunities offered by a new sustainable approach to valorize bottom ash. This approach perfectly addresses current environmental

challenges while offering economic benefits, as it allows avoiding prolonged storage of bottom ash while promoting a change in its waste status.

2. Materials and Methods

2.1. Materials

2.1.1. Bottom Ash

The bottom ash used in this study came from the incineration of non-hazardous household waste in a plant located in Rouen (France). This plant is operated by the Joint Union for Waste Disposal in Rouen, and the bottom ash is then marketed by the company Valenseine (Rouen, France).

At the end of the incineration process, after quenching it in water, the bottom ash must be stored in a special storage area for a period of two to four months. This storage allows the bottom ash to undergo a natural maturation process. During this stage, chemical reactions, notably the carbonation of lime, take place, stabilizing the residue and modifying its physical and chemical properties. This process contributes to improving the quality and performance of bottom ash for future applications, in line with French regulations on the use of bottom ash in road construction.

To determine the impact of bottom ash maturation on the properties of bottom ash–bio-sourced matrix composites, bottom ash was sampled directly at its site.

2.1.2. The Bio-Sourced Matrix

The bio-sourced matrix used in this study is an innovation developed by a researcher team of the University of Picardie Jules Verne (France). It is the subject of a patent (No. 20 12870) entitled “Process for preparing bio sourced elastomer polymers” (ref FR3117118A1 (B1) 2022-06-10 UNIV PICARDIE FR) [26]. It is a heat- and water-resistant bio-elastomer that can be used at room temperature to agglomerate mineral and/or organic particles. It owes its “bio-sourced” designation to its main components: a phosphoprotein compound (casein), a polysaccharide (starch), a pre-cured, natural elastomer (natural latex), a thickening agent (alginate) and/or plasticizer (polyethylene glycol), and water. It has a pH of 7.7. Its bulk density ranges from 0.995 g/cm³ dry to 1.08 g/cm³ wet.

2.2. Experimental Techniques

2.2.1. Production Techniques for Bottom Ash–Bio-Sourced Matrix Composites

Preliminary tests were carried out to determine the optimum water content of the bottom ash so that it would not absorb any of the water from the bio-sourced matrix during composite production. The results obtained show that the water content should be between 14 and 15% of the dry mass of the bottom ash. Matured bottom ash was therefore used in its raw state after carbonation, with a water content equal to 14.29%. Non-matured bottom ash was placed in an oven at 25 ± 5 °C to reduce its water content to 14.67%.

A second preliminary study based on the evolution of composite mechanical strength as a function of percentage led us to choose five MINDN mass percentages (80, 82, 85, 88 and 90%) for this study; the composites are named 80, 82, 85, 88, and 90 respectively (Table 1). To ensure that the bottom ash was homogenized, each batch was quartered before the bio-composites were produced.

Table 1. Mixing ratios for bio-composite (g/1 m³).

Bio-Composite Names	Bottom Ash		Bio-Sourced Matrix	
	%	g	%	g
80	80	1354.2	20	338.6
82	82	1388	18	304.7
85	85	1438.8	15	253.9
88	88	1489.6	12	203.2
90	90	1523.5	10	169.3

The mass quantities of the two components required to produce a composite were fed into a mixer. The latter was chosen with a hook configuration (Figure 1b) to avoid altering the granulometry of the bottom ash, as had been observed with a paddle blender.



Figure 1. Mixer accessory: (a) K-shaped flat mixer; (b) hook.

The mixture was mixed for 60 s at 150 rpm, then for 120 s at 250 rpm (Figure 2).

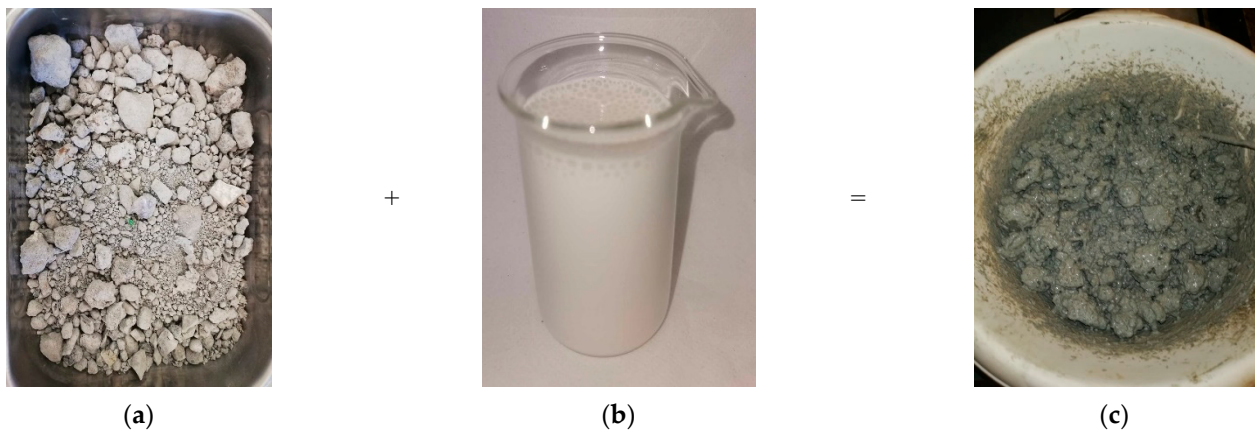


Figure 2. Photographs of the various components (bottom ash (a) and bio-sourced matrix (b)) and the elaborated biopolymer composite (c).

To determine the mechanical strengths of the composites, the mixture was then poured into $(4 \times 4 \times 16) \text{ cm}^3$ specimens, the surface of which was levelled but not compacted (Figure 3).



Figure 3. Photographs of $(4 \times 4 \times 16) \text{ cm}^3$ test species: (a) mixture poured in molds and (b) full test specimens.

The specimens were then stored in the open air for 24 h. They were then removed from the molds and placed in a ventilated oven at a temperature of $50 \pm 5 \text{ }^\circ\text{C}$ in order to reduce the drying time without causing deterioration.

The average time taken for these specimens to reach constant mass was 5 ± 1 days. To guarantee accurate results, the tests were carried out 24 h after the specimens reached constant mass and then brought back to room temperature.

2.2.2. Characterization Techniques

- Bottom ash characterization techniques

To characterize the bottom ash used in this study, several analyses were carried out in accordance with established standards.

The granulometric analysis of bottom ash, whatever its nature, was carried out by sieving in accordance with standard NF 933-1 [27]. This method makes it possible to determine the particle size distribution of bottom ash, essential for assessing its suitability for various applications, particularly as an aggregate in composites.

The bulk densities of bottom ash were determined in accordance with standard NF EN 1097-3 [28]. Bulk density was determined after drying the bottom ash samples, matured or not, to constant mass. In accordance with standard NF EN 1097-3 (ANFOR 1998), for each type of bottom ash, the measurement was repeated 3 times. Sample preparation began with the selection of a representative bottom ash sample. The bottom ash was introduced into the standardized container using a funnel. Before the mass of the empty container was measured (M_1 in g), its volume (V in cm^3) was already known. Finally, the total mass of the filled container was measured (M_2 in g). The bulk density was therefore calculated using the following formula: ρ (g/cm^3) = $(M_2 - M_1)/V$. This parameter is one of the most important we will be looking at later due to its influence on our composites. The water content of each batch was determined in accordance with NF EN 1097-5 [29]. This is an important parameter for composites, as it influences the proportion of water available for the chemical reaction with the bio-based binder.

The pH of bottom ash is an important indicator of its chemical reactivity and compatibility with other materials used. To assess the alkalinity of bottom ash, pH paper was used, and the pH was measured using a Hanna Instruments electronic pH meter to guarantee the accuracy of the results.

A thermogravimetric analysis (TA) coupled with a differential thermogravimetric analysis (dTGA) was carried out on both types of bottom ash (matured or non-matured) (STA 449 F5 JUPITER) to determine their degree of hydration and provide valuable information on various components, such as water and carbonates. About 125 mg of bottom ash was introduced into porcelain crucibles. The analysis started at 20 °C and went up to 1200 °C at a rate of 10 °C/min under a helium atmosphere. The variation in a sample's mass over time as a function of temperature was measured.

- Composite characterization techniques

The characterization of composites is essential for assessing and understanding the physical, chemical, and mechanical properties of these complex eco-materials.

Among the techniques used, there is mainly the determination of mechanical strengths in bending and compression (Figure 4). This characterization enables us to determine the performance of composites in various applications. A multifunction machine AG-X, Shimadzu, with a 10 kN sensor was used. The different strengths were obtained according to the NF EN 196-1 [30] standard. Measurements were repeated 3 times in bending and 6 times in compression per formulation.

To compare the mechanical strength results, the bulk density of the composites was calculated. The specimen of each composite was weighed using a balance to obtain its mass (g), then the length, width, and height were measured to obtain the volume, which is V (cm^3) = $L \times W \times H$. The density was therefore calculated according to the following formula: ρ (g/cm^3) = m/V .



Figure 4. Flexural (a) and compressive (b) strength tests carried out on one of the biopolymer composites.

The morphology of various samples was analyzed using scanning electron microscopy (SEM). A high-resolution Quanta 200 FEG Scanning Electron Microscope (FEI Company, Hillsboro, OR, USA) was employed for this purpose. This microscope is equipped with a Peltier plate, allowing for the observation of a wide range of sample types. The SEM analysis enabled the visualization of the macro-porosity in the various composites. To enhance the observation, the samples were first dried and then coated with a thin layer of gold using a spray technique, which served as a conductive layer.

The microscope was also equipped with an X-ray microanalysis system, specifically the INCA OXFORD SDD 80 mm² detector. Energy-dispersive X-ray spectroscopy (EDS) was used to determine the chemical elements present in the samples.

3. Results and Analysis

3.1. Effect of Bottom Ash Maturation on Its Properties

The thermogravimetric and differential scanning calorimetry analyses of the bottom ash are shown in Figure 5.

The TG-dTG analysis of the matured and non-matured bottom ash samples show two significant differences related to main weight losses. The results are summarized in Figure 5. First, the TG analysis of the bottom ash (Figure 5) revealed a significant loss in mass at the beginning of the analysis. This is due to the remaining adsorbed water evaporation. In order to cool the bottom ash, when exiting the oven, the bottom ash undergoes quenching with water. These conditions also promote the hydration of CaO in portlandite.

The observed mass loss between 200 and 700 °C is greater in the case of the matured bottom ash. During its maturation, bottom ash undergoes various physicochemical transformations, the main one being carbonation. The work of Gervais 1999 [31] and Verrecchia 2002 [32] showed that this carbonation results from the dissolution of minerals, such as portlandite, which releases large amounts of calcium and hydroxide. The high alkalinity of the pore water promotes the solution of atmospheric CO₂ and causes the precipitation of carbonates. The observed mass loss between 700 and 850 °C is therefore due to the decomposition of calcium carbonate. It is more important for matured bottom ash.

The impact of the carbonation reaction on the bottom ash properties is significant. First, this reaction leads to a gradual decrease in the pH of the bottom ash, as the lime is converted into calcium carbonate (Table 2). This decrease in pH can have important implications for the chemical and physical properties of bottom ash and its behavior when used in various applications.

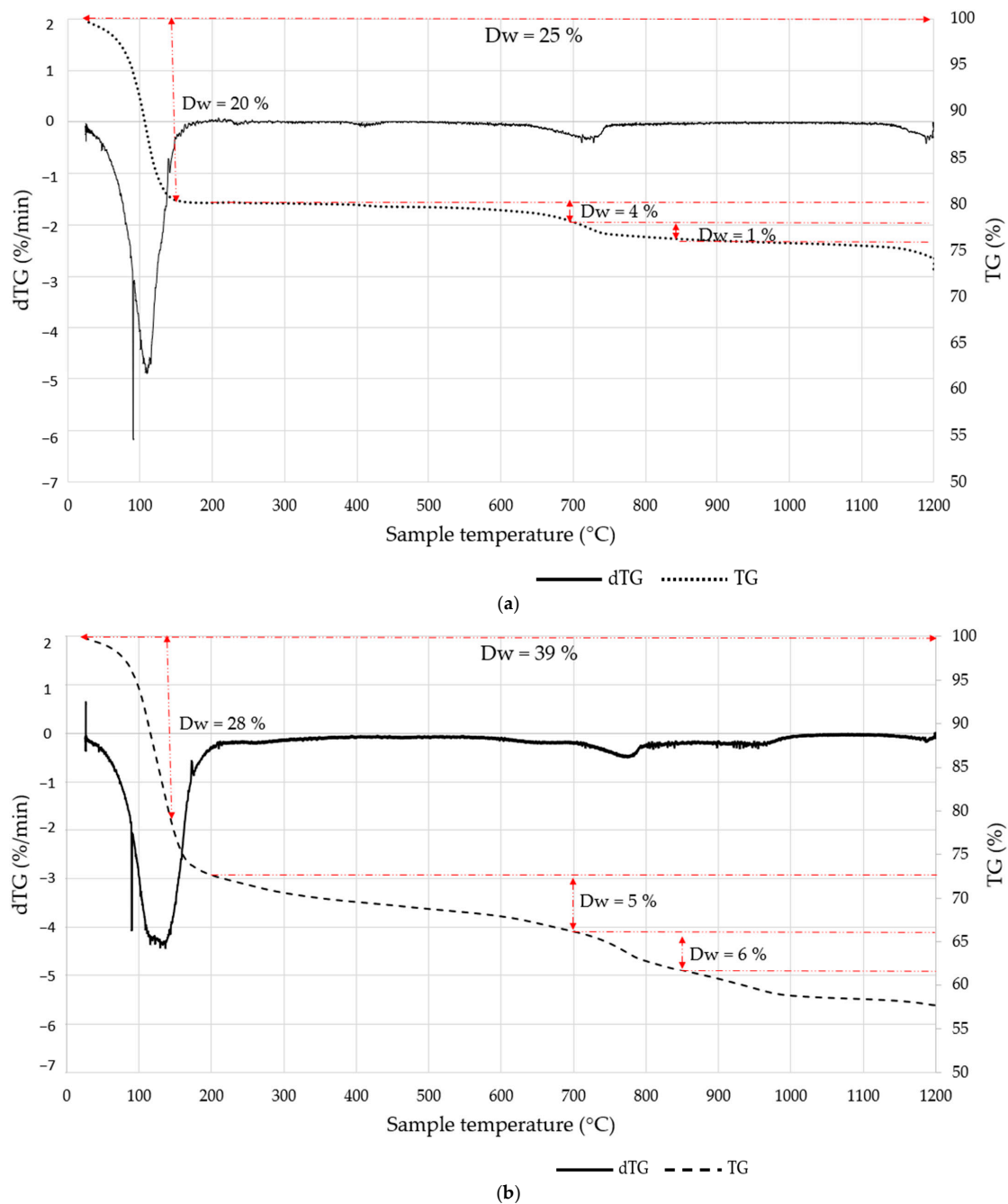


Figure 5. Thermogravimetric analysis (TG) and derivative thermogravimetric analysis (dTG) of non-matured (a) or matured (b) bottom ash, showing the mass loss D_w (%).

Table 2. Main physical characteristics of bottom ash modified during maturation.

Bottom Ash	Particle Size Class (mm)	Bulk Density (g/cm^3)	Water Content (%)	pH
Non-matured	0/14	0.98	24.2	12
Matured	0/40	1.25	14.29	7.7

The particle size analysis (Figure 6) revealed significant differences in the particle size distribution of the two bottom ash types. It can be noticed that the granular beams of the two categories of bottom ash are identical for dimensions less than 4 mm. Beyond 4 mm, the matured bottom ash has a higher cumulative percentage. The granular class is 0/14 mm for the matured bottom ash and 0/40 mm for the non-matured bottom ash (Table 2).

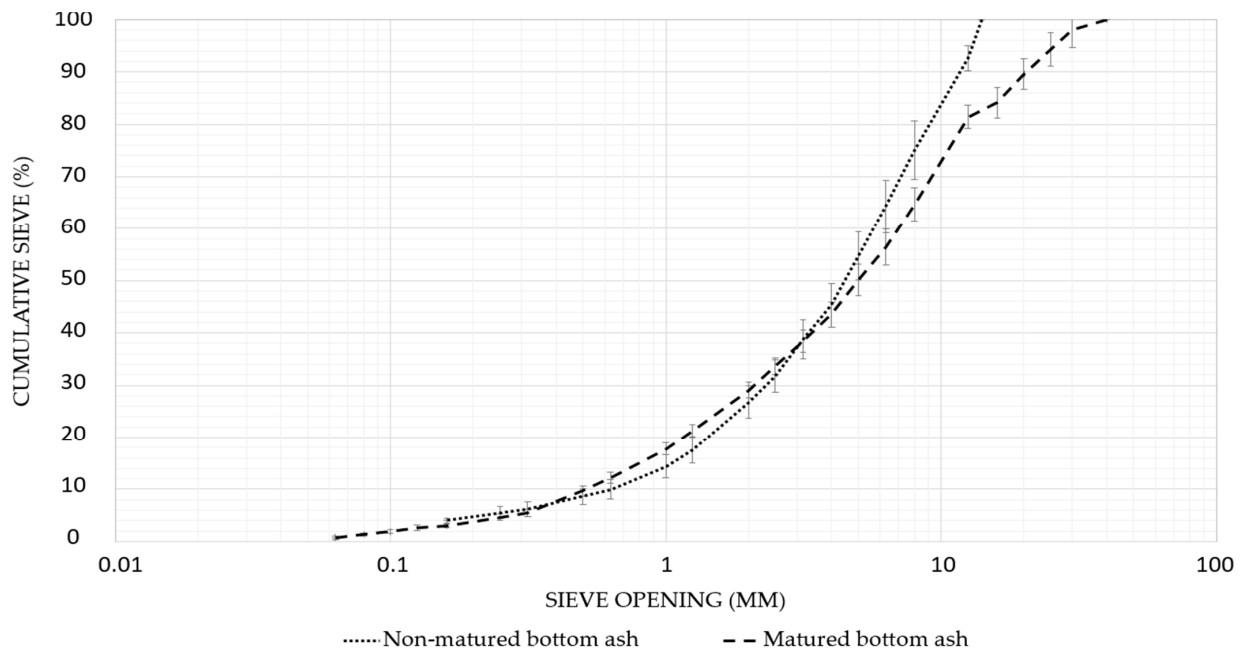


Figure 6. Particle size analysis of bottom ash samples.

The bulk density of the bottom ash also increased during maturation. The results of Table 2 show that this increase is close to 28%. However, regardless of the category of bottom ash used in this study, the samples are light aggregates, since their bulk density is less than 2 g/cm^3 [33]. Thus, despite the changes resulting from carbonation, the bottom ash continues to offer performance as a light aggregate.

3.2. Influence of Bottom Ash Maturation on Composite Characteristics

3.2.1. Influence on Bulk Density of Composites

Figure 7 shows that the bulk density of the bottom ash and bio-sourced matrix composites increases with the percentage of bottom ash used, whether matured or not.

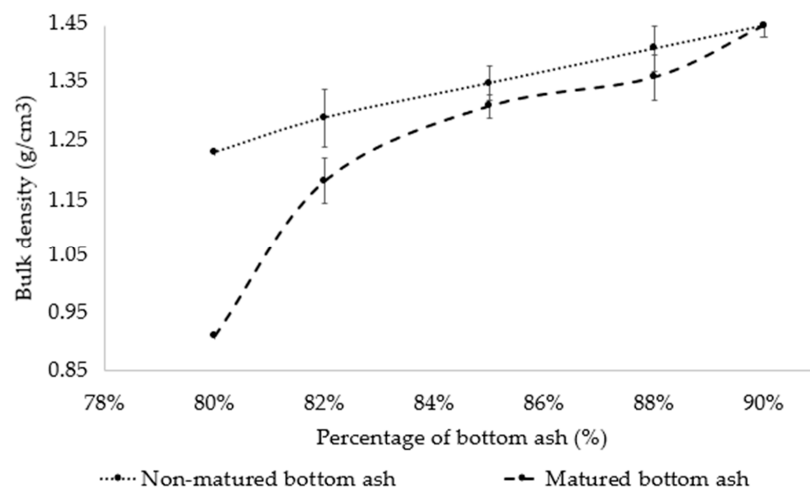


Figure 7. Evolution of the bulk density of composites as a function of the mass percentage of bottom ash.

This increase in the density of biopolymer composites is logical because the bulk density of the bottom ash is higher than that of the bio-sourced matrix. Therefore, the integration of a greater amount of bottom ash in the composite leads to an increase in the bulk density of the whole composite.

It can also be noticed that biopolymer composites containing non-matured bottom ash have a higher bulk density than those containing matured bottom ash. That could

be explained by the particle size of the non-matured bottom ash, which unlike matured bottom ash does not have particles with a size greater than 14 mm.

The fineness of non-matured bottom ash particles reduces the porosity of the composite by allowing for a denser arrangement of aggregates, resulting in higher compactness and, consequently, a higher bulk density (Figure 8a,b,e).

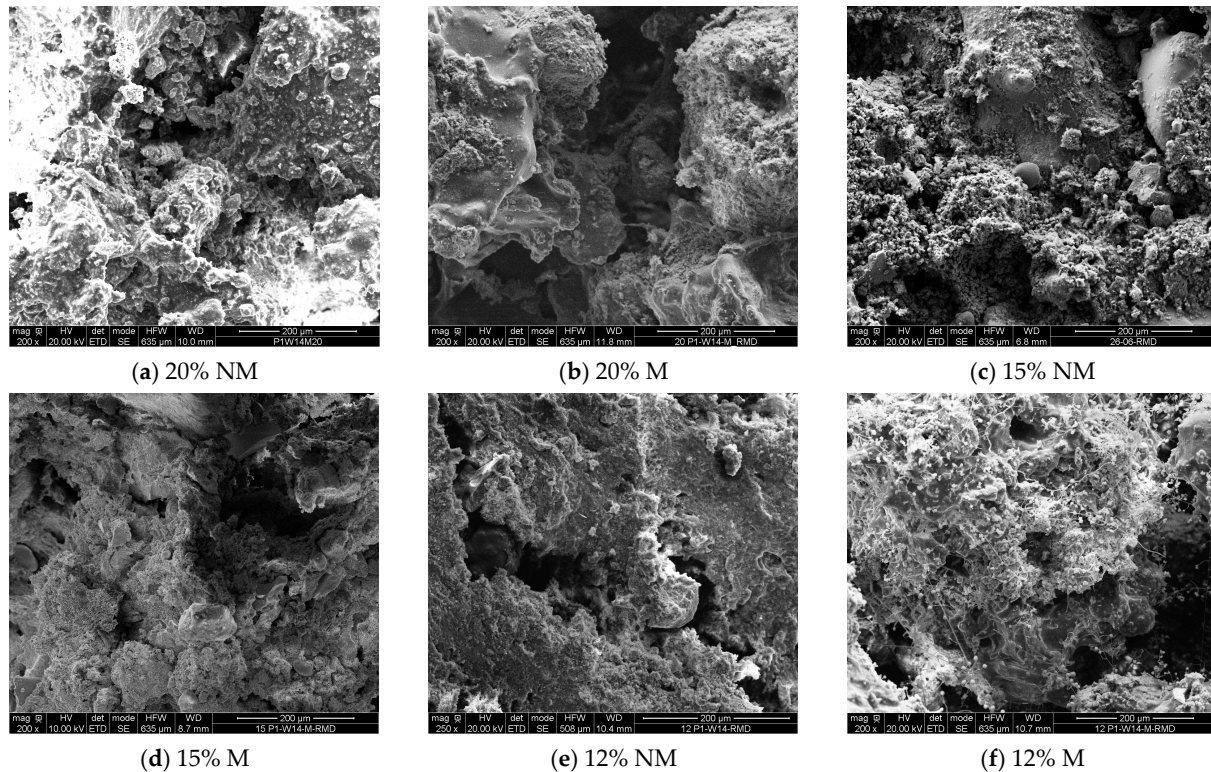


Figure 8. Scanning electron micrographs of composites elaborated with non-matured (a,c,e) or matured (b,d,f) bottom ash; magnification = 200.

On the other hand, matured bottom ash, with larger-diameter aggregates, introduces more voids and porosity into the composite, leading to a reduced apparent bulk density (Figure 8b,d,f).

3.2.2. Influence on the Mechanical Strengths of Composites

The evolution of the mechanical strengths of biopolymer composites as a function of the percentage of bottom ash and the apparent bulk density are represented in Figures 9 and 10.

The Figures 9a and 10a clearly show that the mechanical strengths of the composites vary with the percentage of incorporated bottom ash. In addition, the curves present a maximum for composites developed with 85% bottom ash, regardless of the nature of the latter. If referring to the functional classification of lightweight concrete, this composite with a density less than 2000 kg/m^3 and a compressive strength equal to $3.98 \pm 0.17 \text{ MPa}$ could be used as a lightweight building element [34].

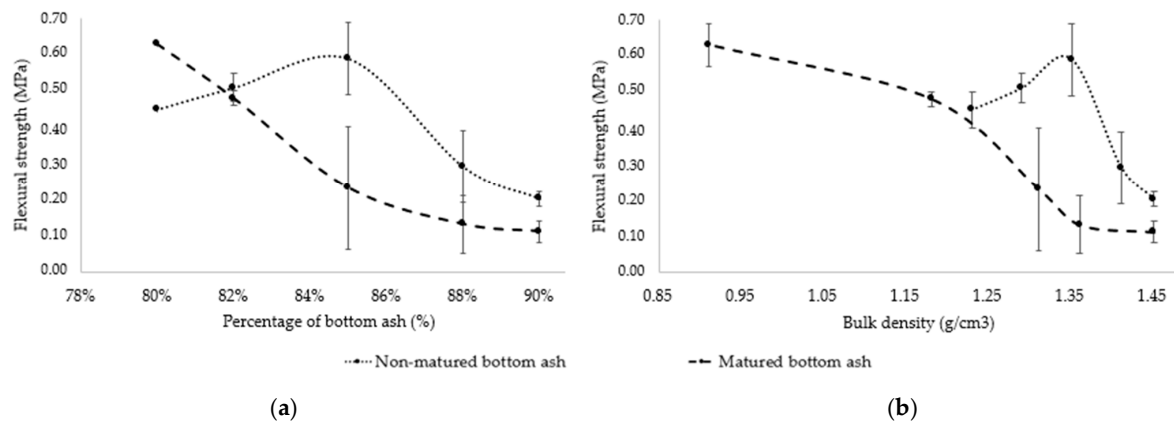


Figure 9. Evolution of the composites' flexural strength as a function of the mass percentage of bottom ash (a) and the bulk density of composites (b).

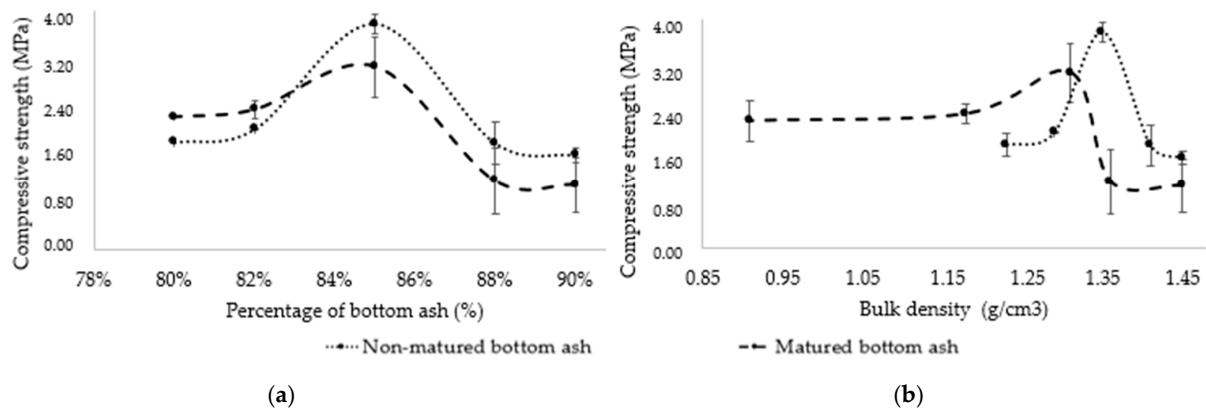
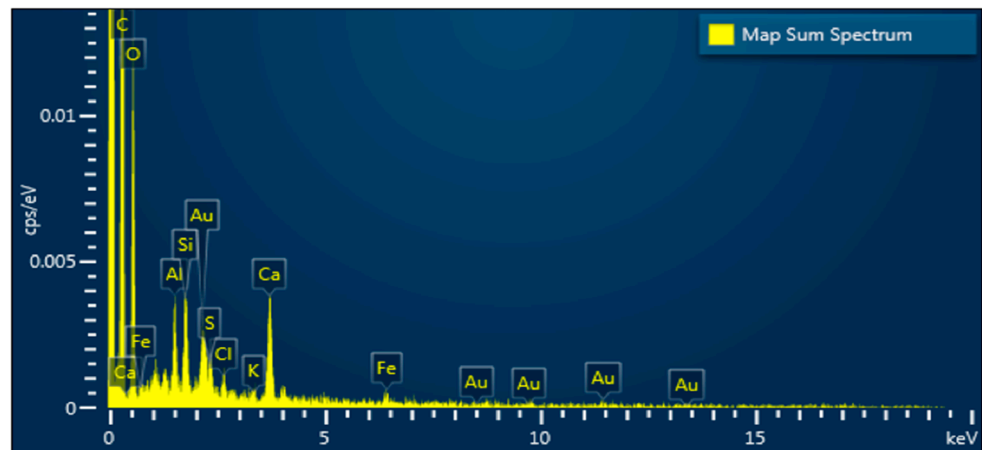


Figure 10. Evolution of the composites' compressive strength as a function of the mass percentage of bottom ash (a) and the bulk density of composites (b).

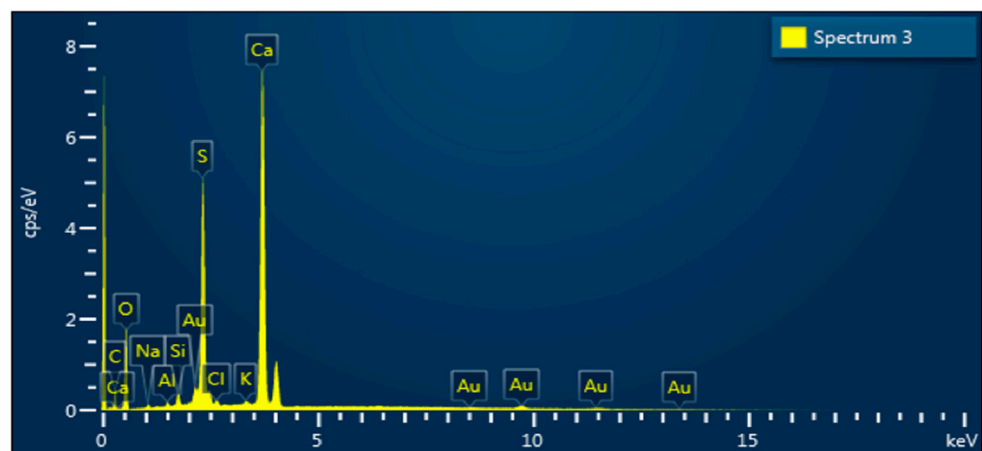
It can also be noticed that the maturation of the bottom ash has an influence on the strength of the composites. Carbonation, the main reaction that occurs during the maturation of bottom ash, seems to have a negative effect on the mechanical strengths of the eco-materials developed in these works. In fact, in the composites made with matured bottom ash, calcium sulphate is the predominant species (Figure 11a), while in the case of the composites made with non-matured bottom ash, the presence of other calcium species such as calcium silicates can be noted (Figure 11b). These silicates are known to improve the mechanical resistance of cementitious composites.

The bio-sourced matrix exhibits mechanical properties (increased flexibility and different failure modes) that are not directly comparable to the rigidity and compressive strength generally observed in cement-based composites. Therefore, the characteristics of the composites developed in this study are not comparable to those of cementitious materials.

In the case of mortars made with 15% matrix, the interfacial zone appears to be of good quality regardless of the stage of maturation of the bottom ash (Figure 12). In addition, the coating of aggregates by the bio-sourced matrix is better in the case of the composites made with matured bottom ash.

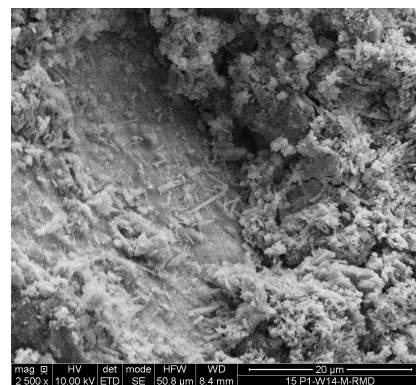


(a)

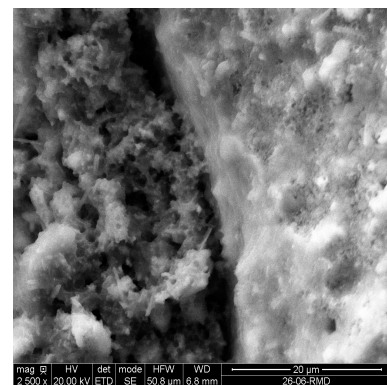


(b)

Figure 11. EDS spectra of composites elaborated with non-matured (a) or matured bottom ash (b).



(a)



(b)

Figure 12. Scanning electron micrographs of composites elaborated with matured (a) or non-matured (b) bottom ash. The mass percentage of bottom ash is equal to 85%, and the magnification is equal to 2500.

However, a greater amount of bottom ash leads to a less good adhesion of the matrix (Figure 13a). And in the case of a greater amount of matrix, a significant presence of calcium sulfate is observed in the case of matured bottom ash (Figure 13b).

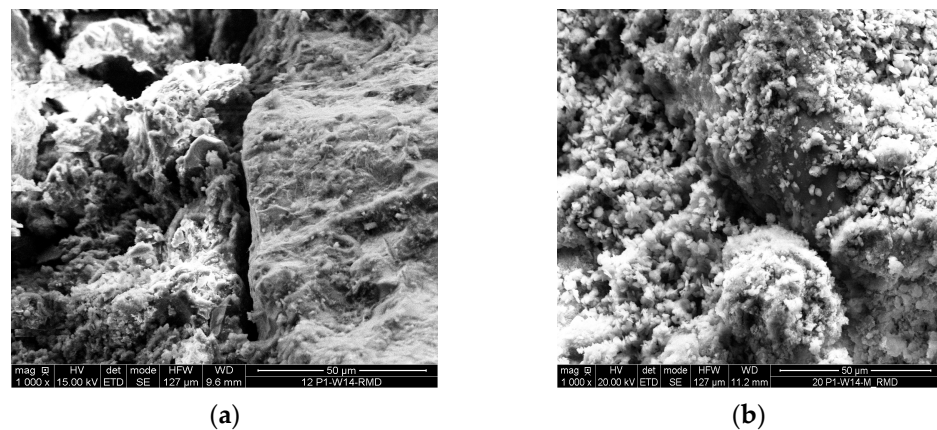


Figure 13. Interfacial transition zone in the case of composites elaborated with (a) 12% of matrix and non-maturated bottom ash or (b) 20% of matrix and matured bottom ash.

All these observations can explain that the mechanical strengths of composites made with matured bottom ash are lower than those of non-matured bottom ash composites and that the maximum amount of bottom ash that can be added is 85%.

3.2.3. Influence on the Preservation of Composites

On the other hand, bottom ash is a combustion residue containing various oxides and hydroxides that can influence the pH of biopolymer composites. Initially, non-matured bottom ash has a high pH, often around 12, due to the presence of calcium hydroxide (portlandite) and other alkaline compounds. This high pH helps to chemically stabilize the composite by creating an environment that is unfavorable to microbial growth, as shown in Figure 14.

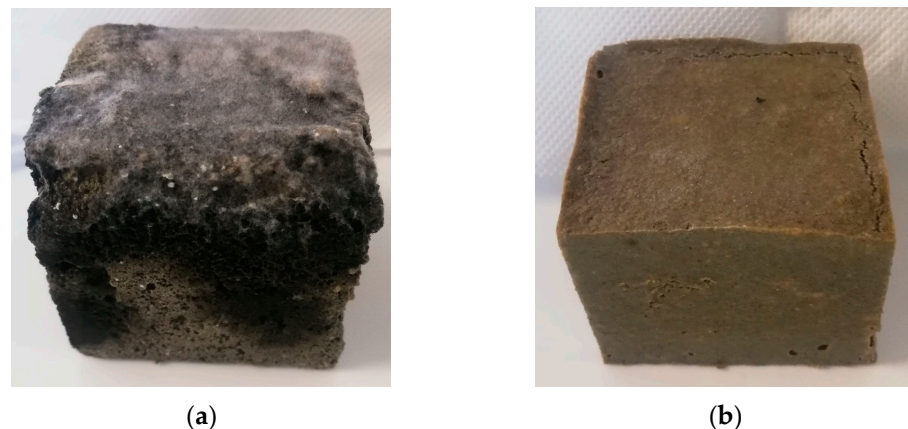


Figure 14. Biopolymer composites elaborated with matured (a) and (b) non-matured bottom ash and preserved for 12 ± 3 days in free air.

Alkaline environments can inhibit microbial proliferation by damaging cell membranes and disrupting the metabolic processes of germs.

As bottom ash matures, its pH decreases, and the environment becomes more conducive to colonization by molds and mushrooms (Figure 15). A lower pH allows spores of mushrooms to germinate and grow, especially in the conditions of heat and humidity often present in practical applications of biopolymer composites.

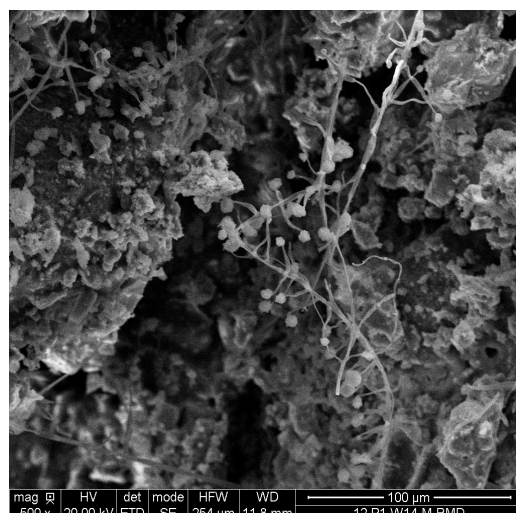


Figure 15. Scanning electron micrographs of a composite made with 12% of matrix ($G = 500$).

4. Conclusions

This study has shown that non-matured bottom ash exhibits superior interactions between the bottom ash particles and the bio-sourced matrix within the composites. It better connects the non-matured bottom ash together. This leads to the formation of more homogeneous and robust materials.

The use of non-matured bottom ash offers interesting properties in terms of mechanical strength compared with matured bottom ash since the maximum compressive strength can reach more than 4 MPa with 85% non-matured bottom ash, which is very promising for a composite with the bio-sourced matrix derived from biomass with a flexural strength that can exceed 0.65 MPa. Moreover, this matrix preserves the bio-composites.

It has been observed that the maturation of bottom ash leads to a decrease in its pH, which significantly promotes the appearance of microorganisms on bio-composites. The high pH of non-matured bottom ash therefore plays an important role in the stabilization of the bio-composite.

In conclusion, the incorporation of non-matured bottom ash into bio-composites proves to be a promising approach to maximize material performance while minimizing costs and environmental impacts, making this practice an advantageous and innovative alternative in the field of composite materials.

This new method of valorizing bottom ash offers a significant opportunity to transform a waste issue into a useful resource, thereby contributing to more sustainable waste management and the conservation of natural resources by being more environmentally friendly, making it a sustainable solution for a variety of industrial applications.

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