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Compressive Strength and Leaching Behavior of Mortars with Biomass Ash

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Abstract: This study investigated the use of a biomass ash produced by a fuel combination made with wood, corn stover, and corn cob as cement replacement for the production of mortar. Biomasses are now widely accepted as a substitute for conventional fuels and are becoming essential for cost-effective production of energy. This study aimed to provide an opportunity for the annual agricultural corn-crop residue, corn stover and cob, which is increasingly being used as fuel for its valuable energy content. Measurements of workability, compressive strength, and leachate properties (pH, salinity, heavy metals and calcium ion release) of mortar specimen, at different cement substitution levels and ages, were evaluated. The results obtained reveal definitive possibilities for such mixed biomass ash to be used in cement-based materials, such as mortars. Moreover, a multiple regression analysis has been reported between the mass of calcium ions leached and the mixture composition with the compressive strength. Data show that further confirmation, on a longer span of time and of other types of mechanical properties and environmental tests, would be necessary to fully implement the use of such biomass ashes in various types of cement-based construction materials, in order to divert them from landfill disposal.

Keywords: biomass ash; biofuel; compressive strength; concrete; fly ash; leaching

1. Introduction

The use of renewable raw materials is continuing to increase. Certain biofuel resources, such as wood, are becoming scarce in some regions [1]. In addition, wood is not available in every area of every country, depending on its climatic and soil conditions; moreover, wood waste is often used for engineering product, such as medium-density fiberboard [2]. With the increased price of fossil fuels, as well as decreased desirability for the use of such fuels, annual agricultural crops, such as straw or whole cereal plants, are reaching closer to economic profitability as a fuel [3]. However, there are many reasons for the presently low use of agricultural crop residues for combustion. Compared to wood, herbaceous plants show some unfavorable fuel properties. Fuel type and its characteristics affect the combustion process in either a technical or an ecological way. Technical problems can, for example, be due to the presence of chlorine, sulfur, potassium, nitrogen, magnesium, and/or calcium that can cause corrosion, and even slagging problems in the combustion boiler-plant and consequentially reduce the useful life of the combustion equipment. Other chemical components, such as heavy metals in the ash, can result in excessive pollutant emissions, or they may remain in the ash, leading to challenges in disposal (Table 1) [4–6].

Table 1. Heavy metal contents of wood, corn stover, and corncob ash (WCSA) compared to the published heavy metal data of coniferous wood and crop of grain straw.

Element	Corn, Whole Plant [7] [mg/kg d. b.]	Coniferous Wood [8] [mg/kg d. b.]	Grain Straw [8] [mg/kg d. b.]
Chrome	4.85	4.50	4.62
Cobalt	0.23	0.35	0.14
Copper	6.08	3.45	2.21
Manganese	46.25	344.70	22.00
Molybdenum	2.53	1.12	0.38
Nickel	0.62	4.23	0.69
Zinc	50.08	37.64	9.42

Ash, therefore, should undergo a broad and critical assessment before being disposed of, with the aim of providing an option for beneficial use, for example, in construction materials. Many attempts have been made in this direction, in order to obtain sustainable building products which can also bring benefit to the occupants, by enhancing the indoor environment quality [9,10]. The assessment should include the evaluation of reducing the cost of construction materials, as well as helping in reducing disposal costs and accounting for long-term and short-term challenges [11–13]. Leaching characteristics are essential in understanding the environmental impact or toxicity, disposal issues, and potential development of beneficial use applications of the wood, corn stover, and corncob ash (WCSA).

The use of coal fly ash for cement and concrete production is a widely adopted method in many countries. The chemical composition of such coal combustion byproduct includes silica, alumina, calcium, iron and magnesium, in different concentration, depending on the type of coal and the combustion and separation technologies adopted. The use of fly ash in concrete has the purpose of obtaining [14–18]:

(1) the reduction of the cement content;
(2) the use of fly ash as a raw material, instead of disposing of it;
(3) the improvement of the concrete durability properties;
(4) a better workability of the concrete mix.

Only recently attention has been paid to the use of ashes from different sources, including: Municipal solid waste ash [19,20], sewage sludge ash [21,22] wheat straw ash [23], olive waste ash [24,25], paper mill sludge ash [26]. However, as the concentrations of the constituents, the elements and the morphology vary depending on the type, it is not possible to predict how the use of a particular ash will affect concrete durability.

Many types of biomass produce ash having similar pozzolanic activity as coal fly ash. For this reason Wang et al. [27] investigated the substitutions of cement with fly ashes obtained from the co-firing with coal, switch grass and sawdust, concluding that: Biomass fly ash concrete has similar compressive strength to that of coal fly ash after the first month (for a substitution of 25% with the concrete); and biomass fly ash concrete has a better performance in mitigating alkali–silica-reaction expansion. Similar results were reported by Cheah and Ramli [28], which they considered wood waste ash.

Since the open burning of biomass constitutes a health hazard in many countries, Memon et al. [23] investigated the optimal calcination temperature of wheat straw and the wheat straw ash grinding duration, concluding that cement could be partially replaced with a percentage of such ash without any negative effect upon its properties.

Although the mechanical characteristics of various fly ash-based concretes have been widely studied in the literature, the impact that these artefacts may have on health is often neglected.

This study presents micro-structural and mechanical behavior, and metal analysis of leachate, obtained from mortars made with various percentages of WCSA (0%, 10%, 20% and 30%).

The hydration behavior of ordinary Portland cement (OPC) in the presence of biomass ash needs to be assessed as a suitable material for mechanical properties and environmental impact [29,30]. An Italian law protocol, designed to assess the environmental impact for biomass ash reuse, is based on a leaching test to be performed, on the ash as such or in the form of its reuse (such as in mortar or concrete), in the range of pH of the leaching, which is presumed to be actual field work of the form recovered in different environmental exposure conditions [30].

In the present research, this leaching protocol was applied to the cement-based mortar cubes, containing WCSA as supplementary cementitious material that was used as a binder replacement for use in mortar production. The importance of this work lies in the evaluation of the effects of the ash content through the comparison of (i) compressive strength as a function of time; (ii) ions leaching release as a function of time; and (iii) as a relationship between compressive strength and leaching. We believe that this is the first work that shows how the leaching behavior of a cement added with biomass-only ash can be a descriptor of both mechanical strength and environmental impact.

2. Materials and Methods

2.1. Specimens Preparation and Procedure

Ordinary Portland Cement, CEM II/A-L 42.5R, and WCSA were used in preparing the mortar mixtures. The test specimens used in this study were 40 × 40 × 160 mm prismatic samples. The WCSA was added to the mortar mixture as a partial replacement of the cement at three levels: 10%, 20%, and 30% by weight. A set of five specimens with water to cementitious materials ratio (w/cm) of 0.42 were prepared for each level of cement substitution; also, another set of five specimens was prepared with a constant 0.5 w/cm. The workability of the mortar was WCSA measured according to UNI EN 1015-3 [31] by the flow table test procedure for fresh mortars, by measuring the increase in mortar diameter. For each mixture, the specimens were made and kept in the sealed steel prismatic molds to allow hardening in the presence of relative humidity (100%) and temperature (26 ± 2 °C) as constant as possible. They were removed from molds at the age of 24 h. After curing for 3, 7, 28, and 56 days they were demolded. An average of three specimens was used to measure the compressive strength at each test age. The leaching release was measured using the tank procedure following the Italian dynamic leaching test procedure [32], at the age of 28 days. This procedure deals with Italian regulations for the reuse of non-toxic materials as by-products. Examples of the similar international standard test include the ISO 6961:82, or the ASTM C 1220 [33]. According to the extraction protocols, a specimen is placed in contact with a precise amount of deionized water for the predetermined extent of time. The solid-to-liquid ratio, expressed as the ratio of the volume of solid to the volume of the leached material, is 1:5. The volume of leaching solution is renewed for each specimen testing to drive the leaching process. At each renewing sequence, the fluid is collected for analysis. The renewing sequence was: 2, 8, 24, 48, 72, 102, 168, and 384 h. The leached material was freshly distilled water with pH of around 6.3 and electrical conductivity of around 3 µS/cm, at room temperature of 20 °C ± 4° C. Because of the physical integrity of the sample matrix (which was maintained during the test), the specimen/leached material properties affect how much material can be leached out, as a function of time. In particular, the surface reactivity of the sample, more than the extraction force, provides the concentrations of the contaminants in the water and the kinetic information about the dissolution process.

2.2. Compressive Strength

Prismatic samples having size 40 mm × 40 mm × 160 mm were cast in triplicates using WCSA as a partial replacement of cement. Mortar composition (in kg/m^3) for all the mixture proportions is presented in Table 3. After casting, all the test specimens were stored at a temperature of about 26 ± 2 °C in the casting room. They were demolded after 24 h and, to allow curing, were put in the presence of relative humidity (100%) for up to 90 days. The compressive strength of these concrete samples with WCSA was determined by the UNI EN 12390-3:2003 at the age of 3, 7, 28, and 56 days.

2.3. Micro-Structural, Chemical and Physical Ash Characterization

Micro-structural analysis of the concrete was conducted using a scanning electron microscope (SEM). Ash samples were dried for 24 h at 40 °C. Then the samples were kept in desiccators overnight for removal of moisture. The samples were coated with gold and analyzed using Philips XL20 scanning electron microscope at 30 kV.

2.4. Heavy Metal Leaching Tests

The environmental impact of cement-based materials may be assessed through characterization of constituent release. Release characterization typically consists of monitoring the flux of constituent for potential concern from a saturated monolithic matrix. For environmental purposes, heavy metals are typically included as an important contaminant. However, cement durability assessment may necessitate a focus on structural species and major ionic constituents (Ca, K, and Na). Simulation of release may utilize models that describe the movement of dissolution fronts within saturated matrices using a diffusion-based released model [34,35].

3. Results and Discussion

Wood, corn stover, and corncob ash was collected from a conventional-boiler after combustion of biomass (wood, corn stover, corn cob, and landfill gas) at about 1000 °C. Main oxides composition is reported in Table 2.

Table 2. Main Oxides Composition from Energy Dispersive X-Ray Analysis (EDXA) of WCSA.

Units	Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe
%	5.62	3.96	8.20	39.94	3.31	6.03	2.63	7.09	16.52	5.02

The average ash content in maize plants shows a relatively low value, more or less within a range between 2.1 and 2.7% [1]. Ash contents are generally inversely correlated with the calorific value, which for a corn stalk is reportedly around 7% lower than wood, ranging from 16.7 to 18.0 MJ/kg for the whole plant. Similar to the ash content, the chemical parameters nitrogen, potassium, and chlorine largely remain constant compared to wood. While the contents of nitrogen are relatively high in comparison to wood or wheat straw, potassium is in between wood and straw, and chlorine is low.

The light-gray colored WCSA used for this research has a specific gravity of 1.90 g/cm^3. The main oxides content of WCSA as analyzed by Energy Dispersive X-ray Analysis (EDXA) is reported in Table 2 and Figure 1.

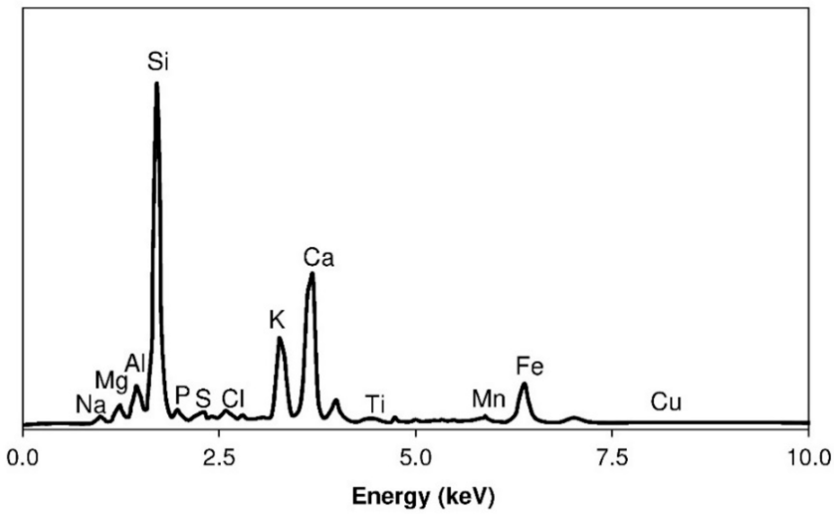

Figure 1. EDXA characterization of WCSA.

The mineralogical characterization for WCSA was determined by X-ray diffraction (XRD) spectroscopy and is reported in Figure 2.

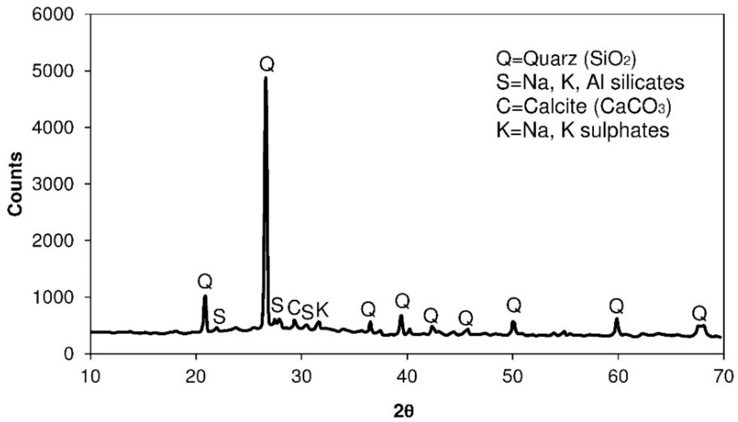

Figure 2. X-ray diffraction (XRD) pattern for WCSA.

Because of their relevance in understanding the hardening performance of WCSA, morphological properties were obtained by Scanning Electron Microscope with Energy Dispersive X-ray (SEM-EDS) Analysis (Figure 3). The analysis revealed that the inorganic portion of the ash sample consisted predominantly of porous irregular particles, mainly Potassium, Sodium, and Calcium with fractions of Sulfur and Chlorine. X-ray diffraction analysis showed the presence of Quartz (SiO_2), Calcite ($CaCO_3$), Arcanite (Potassium Sulfate), Sylvite (Calcium Chloride), and Anhydride (Calcium Sulfate).

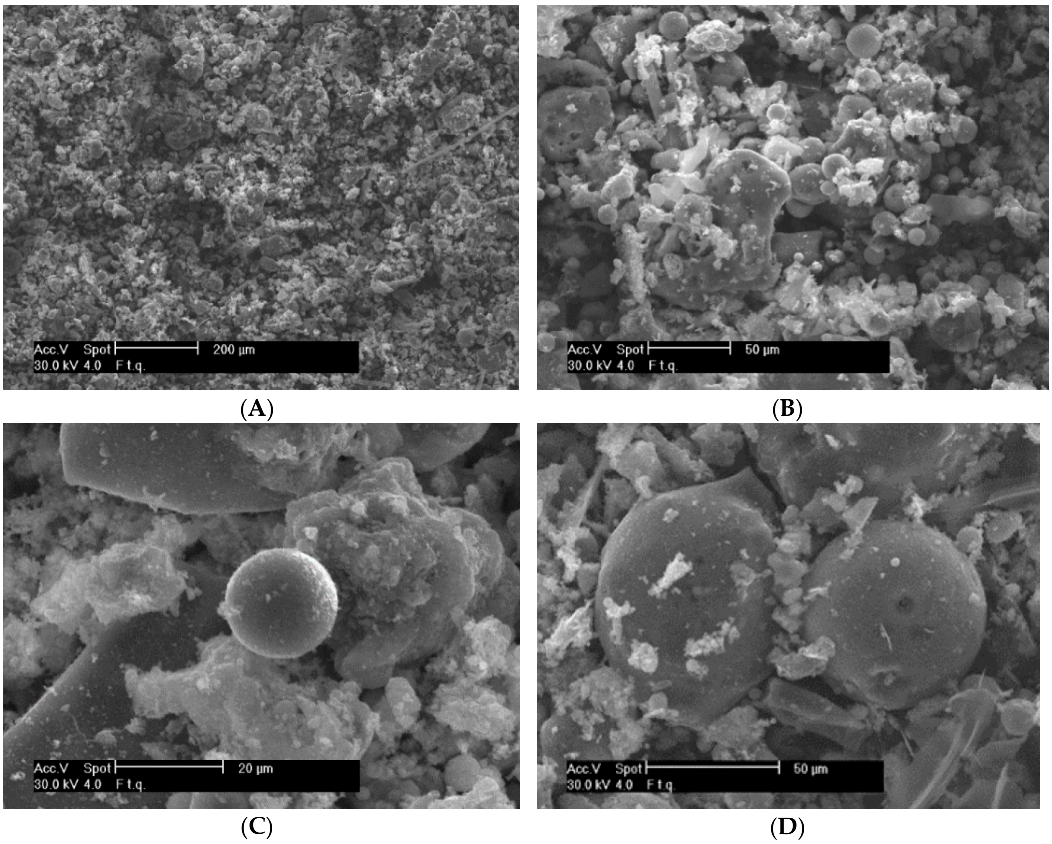

Figure 3. Scanning electron micrograph of WCSA particles. (**A**) Overview of WCSA sample, different zoom areas of the WCSA: (**B**) unburnt particles, (**C**,**D**) fly ash particles.

Measurements of the mortar workability, compressive strength, and leachate properties (pH, electrical conductivity, and calcium ion release) were used for the evaluation of the effect of cement substitution with WCSA, as well as its suitability as a cementitious material. The mixture proportions WCSA and the effect on the workability are reported in Tables 3 and 4 while the compressive strength results are reported in Tables 5 and 6.

Table 3. Mixture proportions of mortars.

	w/cm = 0.42				w/cm = 0.50			
WCSA	**Water (mL)**	**CEM II/A-L 32.5R**	**Ash (g)**	**Sand (g)**	**Water (mL)**	**CEM II/A-L 32.5R**	**Ash (g)**	**Sand (g)**
0	189	450	0	1350	227	450	0	1426
10%	193	405	54	1350	244	405	45.2	1426
20%	197	360	108	1350	262	360	90.4	1426
30%	200	315	152	1350	280	315	135.5	1426

Table 4. Workability of mixture.

WCSA	w/cm	Average Diameter (cm)	w/cm	Average Diameter (cm)
0	0.42	13	0.5	14
10%	0.42	13	0.5	16
20%	0.42	13	0.5	18
30%	0.42	13	0.5	19

Table 5. Compressive strength development (MPa).

Age Days	w/cm = 0.42				w/cm = 0.50			
	Control	**10%**	**20%**	**30%**	**Control**	**10%**	**20%**	**30%**
3	28.0	26.4	26.6	23.3	22.0	17.8	14.6	14.9
7	32.5	35.0	32.3	25.6	27.4	21.3	17.6	19.4
28	38.4	38.8	36.7	37.2	30.6	27.9	25.2	26.7
56	38.7	40.0	37.2	37.3	31.4	28.2	25.7	27.6

Table 6. Parameters fitting Equation (1) to describe the specimen compressive strength.

	w/cm = 0.42		w/cm = 0.50	
Sample Ash%	**$1/b_1$ (-)**	**$1/a_1$ (MPa)**	**$1/b_1$ (-)**	**$1/a_1$ (MPa)**
0	0.047	39.1	0.031	31.9
10	0.036	41.4	0.022	27.4
20	0.036	40.3	0.014	25.1
30	0.022	38.6	0.013	28.6

An attempt is made here to relate the compressive strength to the maturity of mortars cured at 26 °C [36]. Maturity (M) is a product of time and temperature as expressed by $M = T\,(C + 10)$. M is the maturity in °C, T is the curing time in hours, and C is the mortars temperature in °C. The constant 10 is a datum value which assumes that mortar continues to hydrate below the freezing point of water and down to a temperature of −10 °C [37]. The compressive strength (C_{str}), expressed in MPa, and maturity of mortar can be related by the following Equation (1) where a_1 and b_1 are constants.

$$C_{str} = \frac{M}{a_1 * M + b_1} \quad (1)$$

The above equation was used to fit each data set of specimens tested using a least square method, allowing the constants a_1 and b_1 to be determined. The R^2 for all sets is above 0.98 indicating an excellent correlation. It has been suggested that the inverse of constant a_1 ($1/a_1$) is the limiting strength and the inverse of b_1 ($1/b_1$) equals the initial slope of the strength–maturity relationship. Concrete gains strength gradually as a result of a chemical reaction between cementitious material and water; for a specific concrete mixture, the strength at any age is related to the degree of hydration. Since the rate of hydration is a function of temperature, the strength development for a given cement-based material depends on its time-temperature history, assuming that sufficient moisture is available for hydration. This is the basis of the maturity concept, which was developed in the early 1950s to assess the development of in-situ concrete strength during construction. According to this concept, strength development of hardening cement-based materials, such as mortars and concrete, can be estimated at any age by computing the "maturity" based on the temperature and time history of the concrete [33]. In the case of high-performance concrete, however, strength development could become more complex due to the combined physiochemical effects of pozzolans in concrete. The physical influence is in the refinement of the pore structure of the cement paste, while the chemical phase consists of the pozzolanic reaction, which replaces C–H crystals with cementitious C–S–H gel. However, partial replacement of cement in concrete by biomass-based pozzolans may produce an immediate dilution effect, depending upon the type and source of pozzolan [29]. However, generally, it is well established now that coal ash or rice-husk ash do not negatively affect early-age strength [35]. In this paper, an investigation is reported that relate to the strength of concrete mixtures made with biomass pozzolans to the strength of the OPC, used as a control mixture. The parameters involved in this model are the pozzolanic and dilution factors, which depend on the amount of pozzolanic material present in the mixture.

The key feature of this model is its simplicity, since other factors relating to w/cm ratio, age, cement content and temperature can be disregarded because both the pozzolanic and control mixtures have similar material proportions and are assumed to have undergone the same curing history. The results show that, for the w/cm ratio, when fixed at 0.42 and the WCSA-to-cement substitution is set to 20% in the mixture, compressive strength is not reduced. Conversely, fixing a higher w/cm ratio at 0.50, progressive reduction becomes more evident. Hemalata et al. [38] explain that, at low w/cm ratio, unreacted cement and fly ash particles serve as microaggregates, enhancing the compressive strength. However, the substitution effect becomes explicit following the $1/b_1$ parameter, the initial slope of maturity-strength. The sharp decrease observed for both data sets suggests a possible marked effect during the early stages of hydration. In the long term, the compressive strength for the lower w/cm ratio (0.42) increases, while for the higher 0.5 w/cm ratio the compressive strength slopes down. Briefly, the full set of results obtained may be attributed, besides the partial cement substitution with WCSA, to the water-to-cementitious material adopted, along with the pozzolanic effect of the WCSA used.

Heavy Metal Leaching Tests

The environmental impact of cement-based materials may be assessed through characterization of constituent release. Release characterization typically consists of monitoring the flux of the constituent of the potential concern from a saturated monolithic matrix. For environmental purposes, heavy metals are typically included as an important contaminant; however, cement durability assessment may also necessitate a focus on structural species and major ionic constituents (Ca, K, Na). Simulation of releases usually utilizes models that describe the movement of dissolution fronts within saturated matrices using a diffusion-based release model [33,35]. The obtained heavy metal leachate concentrations fully conform to the Italian legal standards. As shown in Table 7, concrete specimens manufactured with or without WCSA showed comparable releases. All leachates, alkaline throughout the testing period with pH values greater than 10, indicate that the interstitial pore fluid in contact with hydrated cementitious materials is buffered by the presence of portlandite and alkaline ions. So far, as the local condition of the leached material remains unchanged, the heavy metals leaching remains very low, as found in a previous publication [36].

Table 7. Cumulated heavy metals (mg/L) in the leaching solution.

WCSA	Ba	Cr	Cu	Ni	Pb	V	Zn
0	0.087	0.008	0.042	0.004	0.003	0.008	0.128
10%	0.082	0.007	0.008	0.002	0.002	0.011	0.113
20%	0.049	0.005	0.009	0.005	0.001	0.007	0.386
30%	0.285	0.006	0.013	0.004	0.001	0.015	0.001
0	0.272	0.008	0.019	0.003	0.002	0.012	0.001
10%	0.079	0.026	0.037	0.007	0.005	0.026	0.116
20%	0.034	0.011	0.015	0.006	0.002	0.026	0.001
30%	0.143	0.008	0.011	0.008	0.003	0.010	0.298
ref. limit	1.0	0.05	0.051	0.01	0.05	0.25	3.0

As, Be, Cd, Co, concentration under detection limit.

The alkaline cations release involves mainly Ca^{2+} and K^+, which are elements that are found in higher concentrations in pore water analysis. This was somewhat expected since, among other things, the major product of OPC hydration includes portlandite ($Ca(OH)_2$). Calcium ions are also abundant in the WCSA, and K^+ is the major alkaline constituent of the ash residue. Adopting the maturity approach, as the hydration model related to the release of the calcium ions and diffused within the pore water, one can obtain from Equation (1) the parameters a_1 and b_1 describing the ionic development. In Table 8, calcium release is expressed as milli-equivalent (meq) of calcium ions for each specimen.

Table 8. Parameters fitting Equation 1 to describe the specimen calcium ion release.

	w/cm = 0.42		w/cm = 0.50	
Sample ash	**$1/b_1$ (-)**	**$1/a_1$ (meq Ca)**	**$1/b_1$ (-)**	**$1/a_1$ (meq Ca)**
0	0.094	10.58	0.049	20.31
10%	0.073	13.61	0.062	16.04
20%	0.124	8.06	0.075	13.23
30%	0.183	5.45	0.074	13.41

The leaching history manifested by each specimen within the two set of w/cm ratio (0.42 and 0.50) comply with the corresponding strength development as shown in Figure 4.

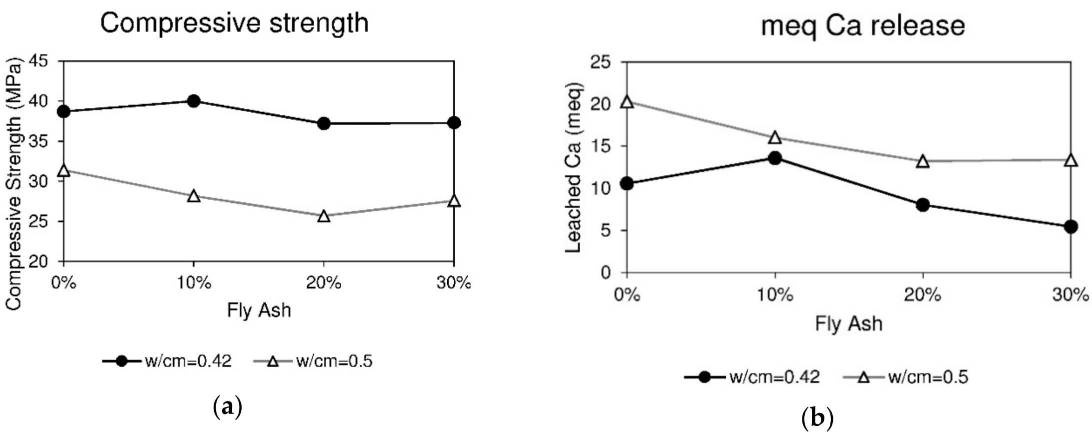

Figure 4. (**a**) Strength (MP) vs. WCSA%, (**b**) mEq Calcium Release vs. WCSA%.

The peculiar symmetry observed reveals a relationship between the specimen characteristics through a multiple regression analysis, taking the mass of calcium ions leached as an independent variable and the mixture composition, expressed in grams, in which water (w), cementitious materials (cm), WCSA and the compressive strength (C_{str}), expressed in MPa, as dependent variables. The results are summarized in Equation (2) and Figure 5.

$$mEq_{Ca} = \frac{C_{str}}{0.739} - 0.5\text{w} + 0.93\text{cm} + 0.73\text{WCSA} \tag{2}$$

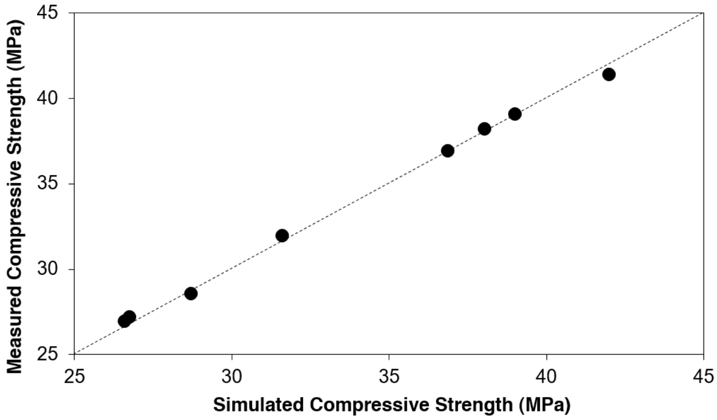

Figure 5. Observed vs. predicted compressive strength adopting Equation (2).

4. Conclusions

The WCSA examined in this study is characterized by high amounts of silicium (39%), calcium (16%), and potassium ions (7%), not different from many other biomass ash compositions found in the literature. For the constant w/cm ratio of 0.42, the compressive strength of prismatic mortars specimens, obtained with WCSA up to 20% as a partial replacement of cement, show a small increase, without losing workability. However, at higher w/cm a clear performance decrease has been observed. The chemical properties of the pore solution given by the leaching tests are characterized by a high concentration of calcium and potassium ions. Particularly the calcium ion leached by WCSA concrete specimens, with respect to the control concrete specimens, indicates a notable alteration of the pore water chemistry. This change has been attributed either to the particular composition of the WCSA examined or to the cement hydration delay as indicated by the kinetics values obtained from the diffusion model adopted.

Cumulated heavy metals in the leaching solutions highlight how these are below the reference concentration limit, so they don't constitute a concern for the health.

In conclusion, the ashes obtained from various sources are already used as a supplementary cementitious material, whereas WCSA can be put to good use in order to divert WCSA from landfill disposal.

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