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Original Research Article

INFLUENCE OF BIOMASS ASH ON THE PERFORMANCE AND DURABILITY OF MORTAR

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A b s t r a c t

This work revolves around the study of the partial substitution of cement by biomass ash (residue generated during incineration of wood waste) in mortar. The introduction of wood ash in the cement formulation allows solving some problems related to the lack of construction materials and protecting the environment. To carry out this work we have introduced wood ash as an addition which partially substitutes cement at three different replacement percentages (5%, 10% and 15%). We carried out tests on mortar in the fresh state (consistency, density and occluded air); evaluated its performance in the hardened state (compressive and flexural tensile strength), dimensional stability and its durability (water absorption by immersion and by capillarity as well as resistance to chlorides and acidic environments followed by XRD). Results obtained will be compared with the results of control samples with 0% substitution rate.

The results of this valorisation show that the incorporation of 5% of ash fillers in the cement improves mechanical resistance as well as certain durability parameters.

Keywords: valorization, substitution, cement, wood ash, additions, performance, durability, XRD

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1. INTRODUCTION

Wood ash (biomass ash) is a residue generated primarily during the incineration of wood scraps (tree bark, trunk, or other). This organic or agricultural waste is in the form of a fine powder of a grey colour similar to the visual appearance of cement. The pulverulent debris called ash is very rich in mineral salts, mainly calcium in the form of lime (20% to 50%), potash (2 to 9%), silica (1 to 4%), magnesium (14%), phosphorus (0.5 to 2%), but no nitrogen [1]. Can this material really represent a potential cement substitute with predetermined percentages? Can it make improvements in physical or mechanical characteristics? We have established this work to answer these questions.

This work falls within the framework of waste recovery as an addition in the cement manufacturing with the aim of eliminating waste and reducing the cost of clinker production and protecting the environment.

Previous years have seen several attempts to use biomass ash in civil engineering and especially the incorporation of these residues in the manufacture of cement; scientists around the world have used the type of ash available locally, such as rice husk ash and bamboo ash in Asia, sugarcane bagasse ash in Central America, olive pomace ash in Africa.

Focusing on wood ash, we find that several researchers have developed the idea of recycling these residues. Dhakulkar et al. (2018) [2] have found that the majority of oxides in wood ash are lime (CaO), portlandite (Ca $(OH)_2$), calcium carbonate ($CaCO_3$) and calcium silicate (Ca_2SiO_4). To this end, it can be affirmed that the substitution of cement with these ashes is feasible.

According to Chowdhury et al. (2015) [3], chemical analysis and X-ray diffractometry have shown that wood ash contains a considerable amount of amorphous silica so the substitution of cement with this residue will not adversely affect the strength properties of the concrete, indeed their investigation has shown promising results and meets the environmental requirements.

In the same concept the researchers Barathan et al. (2013) [4] demonstrated that wood ash has pozzolanic and hydraulic properties, which makes the introduction which makes the use of these residues in mortar or concrete, in combination with cement, the best possible recovery.

In the study published by Leroy et al. (2019) [5], they found that the consistency of the mixtures increases with the increase of the substitution rate during mixing, and consequently we have a significant increase in the amount of water needed to obtain a malleable mortar. In the same work, these researchers found that the initial setting time and the final setting time increased consistently with the amount of ash incorporated into the cement. Their results also showed that the introduction of wood ash improves the resistance to hydrochloric acid attack (HCL).

In the study published by Vu et al. (2019) [6] the results of partial replacement of cement by biomass ash (with the rates of 10%, 20%, 30%, 40% and 50%) in the manufacture of panels, show that the water demand during mixing increases proportionally with the ash content. On the other hand, they found that the ash gave the mixtures a relatively lower loss on ignition and thermal conductivity compared to the control mixtures.

Based on the experimental program of Elangovanand et al. (2018) [7] where they replaced 10%, 20% and 30% of the weight of cement with wood ash, the results indicate that after 7 and 28 days the mortar with 10% ash shows higher compressive strengths than the other percentages and fall within the range of 23 to 26 MPa.

Amrutha et al. (2016) [8] devoted part of their thesis to study the behaviour of concrete with 3%, 5% and 8% wood ash, the results of compressive strength at the age of 7 days are satisfying regardless of the substitution rate.

Othuman Mydin et al. (2014) [9] investigated the possibility of replacing 5%, 10% and 15% of the cement with a combination of wood ash and silica fume, the results showed improvements on the microstructural scale and consequently improvements in mechanical strengths. The researchers also found that this substitution significantly prolonged the initial and final setting time of the mixtures.

The uncontrolled and massive use of non-renewable natural resources has prompted Fapohunda et al. (2018) [10] to study the possibility of substitution of cement and aggregates by sawdust ash with varying percentages, the results show satisfactory strength and durability properties except for vulnerability to carbonation and sulphate attacks. According to these scientists, it is therefore possible to develop structural concrete that meets the requirements, provided that the substitution does not exceed 20%.

The primary objective of this study is to valorise wood ash residues in the manufacture of an ecological mortar in order to save the quantity of cement usually consumed as well as to preserve the environment by minimizing the emission of greenhouse gases (GHG) produced by the cement industry.

2. MATERIALS USED

- The cement used in this study is a class 42.5 CEM I from the cement company of M’sila, Algeria
- Rolled sand (RS) from Oued Zhour dune, Skikda East of Algeria
- The wood ash used in our tests is a residue generated mainly during the combustion of wood scraps. This waste was collected from traditional chimneys and agricultural fields after the burning of branches, leaves, etc. at the end of each harvest season. Even the ashes from the production of charcoal

have been collected. These biomass ashes do not require any prior treatment and are used directly after sieving (to put the non-incinerated parts aside).

– Potable water was used in all the mixes and curing of the specimens.

Fig. 1. The wood ash used

The chemical composition and physical characteristics of cement and wood ash are shown in Table 1.

Table 1. Physico-chemical properties of wood ash and cement

Designation	CEM I	Wood ash
Absolute density (g/cm^3)	2.9	**2.3**
Specific surface (Blaine) (cm^2/ g)	3695	**2528**
CaO	62,85	**33.93**
AL_2O_3	5,02	**5.26**
Fe_2O_3	3,14	**2.21**
SiO_2	21,01	**21.00**
MgO	1,85	**1.44**
Na_2O	0.11	**0.48**
K_2O	0,45	**--**
Cl^-	0.017	**--**
Loss on ignition	1,78	**28.60**
SO_3	2,04	**--**
C_3S	61.00	**--**
C_2S	14.50	**--**
C_3A	02.40	**--**
C_4AF	15.22	**--**

From the results of the characterization tests, we can see that:
- Wood ash has chemical properties similar to those of cement.
- The absolute density of wood ash is slightly less than that of cement.
- The specific surface area (Blaine) of ashes is lower than that of cement.

3. EXPERIMENTAL PROGRAM AND TESTS CARRIED OUT

The objective of this investigation is to study the impact of the partial replacement of a CEM I cement by wood ash on the properties of a mortar with 5%, 10% and 15%.

The formulation of the control mortar (CM) is based on a classic formulation which is 1/3 (1 volume of cement and 3 volumes of sand) with the fixed parameters: the ratio water on cement W/C and the cement dosage; the quantity of water was fixed by preliminary consistency tests.

The compositions of the mortar mixtures (M 5%, M 10% and M 15%) were obtained by substituting 5, 10 and 15% of cement with wood ash (Table 2).

Table 2. Composition of mortar mixtures

Formulations	Cement (g)	Ash fillers (g)	Sand (g)	Water (ml)
CM	450	0	1350	261
M 5%	427,5	22,5	1350	261
M 10%	405	45	1350	261
M 15%	382,5	67,5	1350	261

The tests carried out on the different mixtures in the fresh and hardened state are:
-Density of fresh mortar according to the standard NF EN 1015-6.
-Consistency according to the standard NF EN 1015-3.
-Occluded air measured by an air entrainment meter in accordance with standard NF EN 1015-7.
-Compressive and flexural tensile strength at the age of 2,7, 28 and 90 days on 4x4x16 cm^3 specimens according to the standard NF EN 1015-11.
-Water absorption by immersion measured on cubic samples of 5x5x5cm^3according to the standard NBN EN 1015-18.
- Water absorption by capillarity measured on samples of 4x4x16 cm^3 in accordance with standard NF EN 1015-18.
- Chloride penetration at the age of 28 days measured with the colorimetric indicator method with silver nitrate solution ($AgNO_3$) on 4x4x16 cm^3 specimens according to the standard UNI 7928 and JIS A 1171.

- Resistance to chemical attack on 5x5x5 cm^3 specimens kept for 1,7,14, 21, 28, 56 and 90 days in a solution containing 5% hydrochloric acid (HCL) according to the standard ASTM C-267-97.
- Resistance to chemical attack on 5x5x5 cm^3 specimens kept for 1,7,14, 21, 28, 56 and 90 days in a solution containing 5% sulphuric acid (H_2SO_4) according to the standard ASTM C-267-97.
-X-ray diffractometric test according to standard NF EN 15305 on the residues of specimens immersed in HCL and H_2SO_4 acids.
- Dimensional stability test (shrinkage and swelling), on 4x4x16cm^3 specimens equipped with gauge studs according to the standard NF P15-433.

4. RESULTS AND DISCUSSIONS

4.1. Influence of substitution rate on the properties of mortar at the fresh state

4.1.1. Density

Density is measured by the difference in weight between an empty container and another filled with fresh mortar.

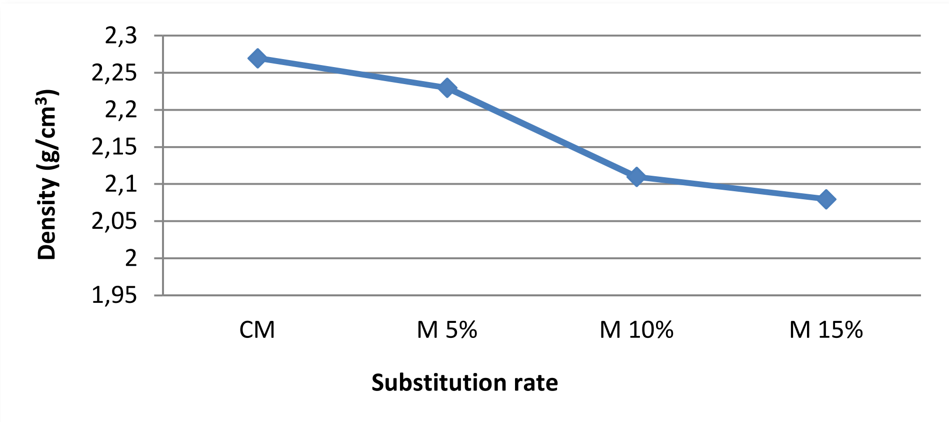

Fig. 2. Density variation according to substitution rate

Fig. 2 displays the results of density;it can be observed that the introduction of wood ash leads to a decrease in density. The lower density value is observed for the mixture with 15% of biomass ash, we noted 2.08 g/cm^3. This reduce is explained by the lower Absolute density of wood ash compared to that of cement [6], which gives us a mortar less dense than ordinary.

4.1. 2. Consistency

This is an essential test to characterise the fresh state and determine the workability of the mortar, this test is carried out using a mini cone placed on a flow table. At the end, the slump obtained is measured.

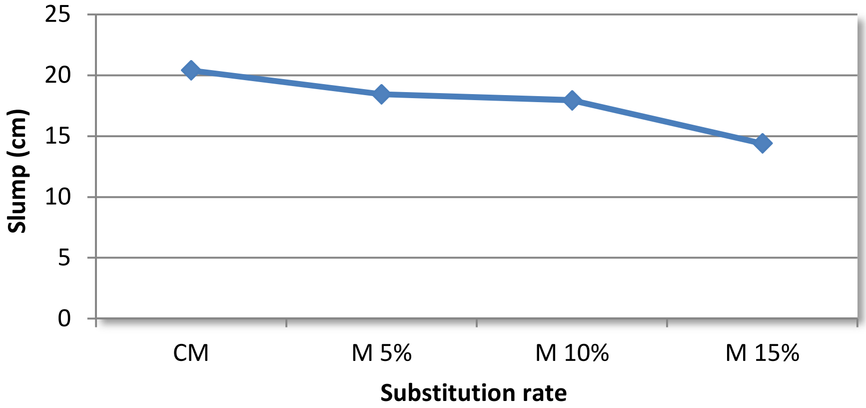

Fig. 3. Influence of substitution rate on workability

According to Fig.3, the incorporation of biomass ash leads to a decrease in workability whatever the degree of substitution. The slump values continue to decrease until they reach the value of 14.4 cm noted for the last percentage (15%). This decrease in slump values is justified by the high organic content of wood ash [3].

4.1.3. Occluded air

The occluded air content is measured with an air entrainment meter on fresh mixtures. The results are shown in Fig. 4.

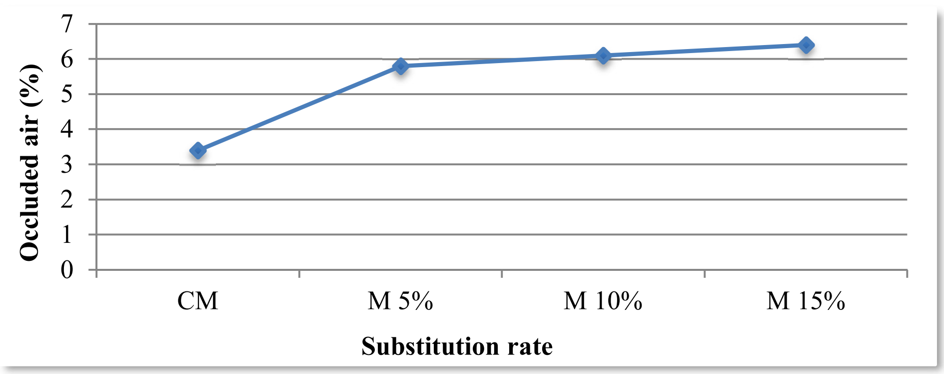

Fig. 4. Effect of substitution rate on the occluded air variation

From Fig. 4 we can observe that the replacement of cement with biomass ash leads to an increase in the content of occluded air regardless of the substitution rate. This phenomenon can be explained by the porosity of the ash particles and the irregular and unrounded morphology of its particles [11], which results in a higher quantity of air in our mortar mixtures.

4.2. Influence of substitution rate on properties of mortar at the hardened state

4.2.1. Compressive strength

The compressive strength of the samples was determined at the age of 2, 7, 28 and 90 days, the results are shown in Fig. 5.

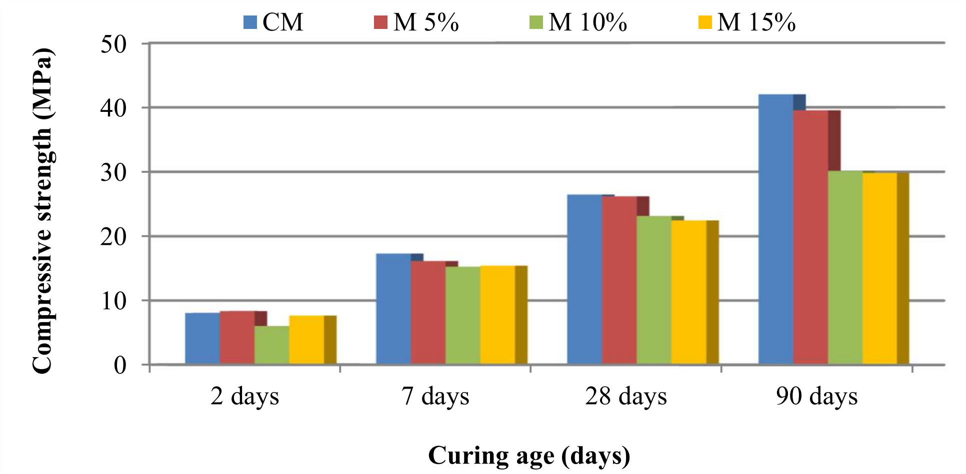

Fig. 5. Effect of the substitution rate on the compressive strength

At early age (2 days), the mortar with 5% of wood ash shows the best compressive strength, according to Akeem et al. (2013) [12] this phenomenon can be explained by the reaction of wood ash with calcium hydroxide released during hydration. The evolution of the compressive strength of the mortars studied increases steadily with time, however, the mixtures based on biomass ash mark a decrease in strength from 7 days but the values remain acceptable, these results are consistent with those found by Subramaniam et al. (2015) [13].

4.2.2. Flexural tensile strength

The flexural tensile strengths of the samples were determined at the age of 2, 7, 28 and 90 days, the results are shown in Fig. 6.

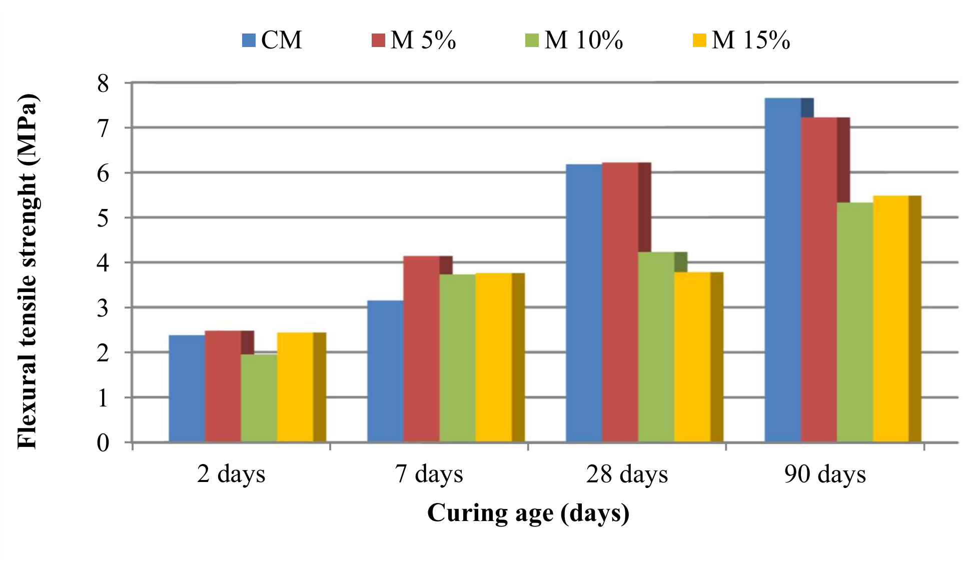

Fig. 6. Effect of the substitution rate on the flexural tensile strength

Fig. 6 shows that the incorporation of 5% of wood ash in the mortar improves the flexural tensile strength at the age of 2, 7 and 28 days. Similar results are observed in the study published by Vaishali et al. (2012) [14], this behaviour can be explained by the amount of lime (CaO) present in the biomass ash which increases the cohesion between the cementitious matrix and the granular skeleton and consequently increases the flexural tensile strength. In the long term (90 days) the control mortar marks the highest value compared to those containing ashes but the values remain acceptable.

4.2.3. Immersion absorption

Water absorption by immersion consists of placing cubic test of the mortar totally immersed in water at 20°C for a period of 24 hours, until the material is saturated, then in the oven at 105°C until a constant mass is reached, then removing them and then weighing them.

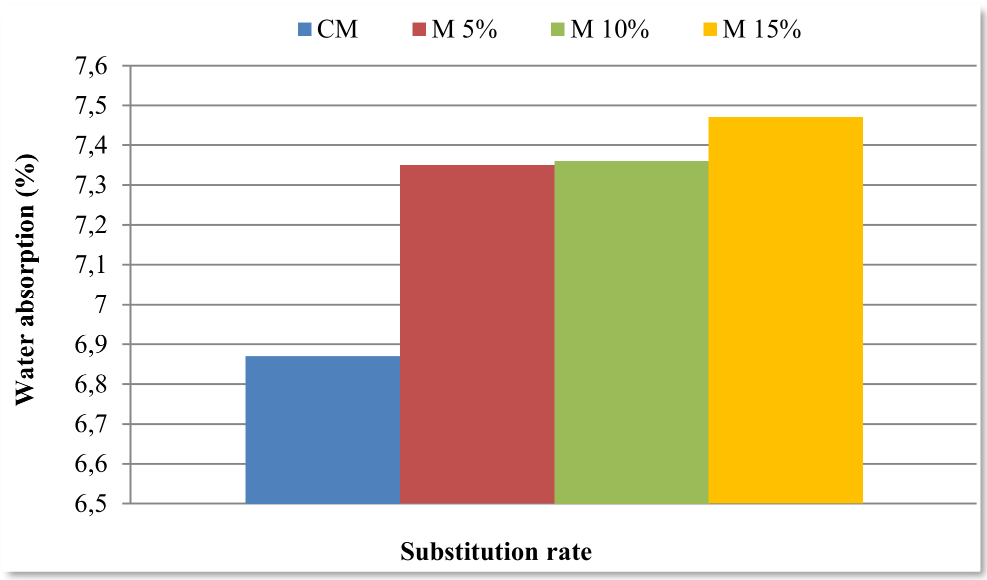

Fig. 7. Effect of substitution rate on water absorption variation

By analysing the results shows in Fig. 7, we can say that the water absorption by immersion increases proportionally with the increasing of biomass ash levels, this result is in line with that found by Vu et al. (2019) [6]. These observations can be explained by the nature of wood ash, this organic and porous material tends to absorb more water than cement.

4.2.4. Capillary absorption

This characteristic reflects the amount of water that can be absorbed by the stone when only one side of the element is in contact with water. The results are displayed in Fig. 8.

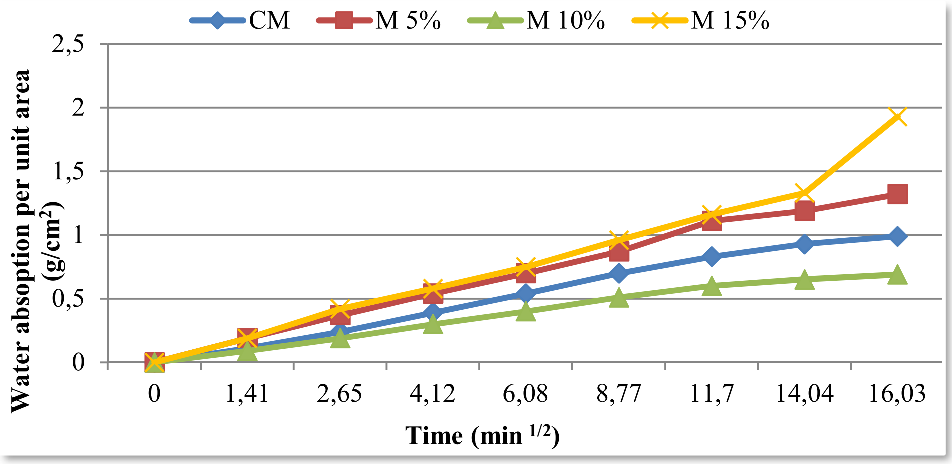

Fig. 8. Variation of capillary absorption as a function of the root of time

According to Fig. 8, the capillary water absorption increases over time, the higher value is given for the mortar containing 15% of biomass ash. According to Fusade et al. (2019) [15], wood ash is one of the hygroscopic materials, which gives the mortar the ability to absorb more water by capillarity. We can also observe that the mortar containing 10% of wood ash marks the lower value.

4.2.5. Chloride penetration

Following the water curing, specimens were preserved in sodium chloride solution (NaCl) with the concentration of 5% for 28 days.

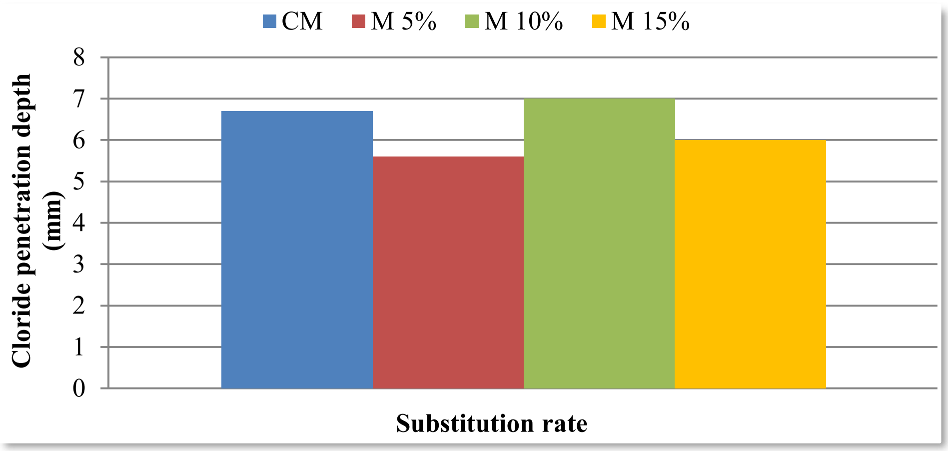

Fig. 9. Variation of chloride penetration depth as a function of the substitution rate

After the conservation in the saline solution, a non-linear variation of the chloride penetration can be noticed. According to Fig. 9 it can be seen that the mortar with 5% of ash addition marks the lower value in term of penetration depth. The chloride resistance of concrete gives an indirect measure of its permeability and internal pore structure [16].

4.2.6. Chemical attack in aggressive environments

- **Hydrochloric acid (HCL) attack**

All mixtures without exception show a permanent but moderate loss of mass (Fig.10),; the same results found by Mambou et al. (2019) [5]. In terms of mass loss, the mortar with 10% of ash addition showed a better performance and degrades the least. This loss of mass is due to the fact that portland cement after hydration, releases some free calcium hydroxide $Ca(OH)_2$ which can leach out internally when exposed to acid attack [17].
The strength of the acid attack depends on the quality and permeability of the mortar. For the mortars studied coming into contact with hydrochloric acid HCL (Fig. 11), this acid latter reacts with calcium hydroxide $Ca(OH)_2$ to form calcium chloride $CaCl_2$ which deteriorates the specimens, causing a loss of mass [18] according to the following formula [16]:

$$Ca(OH)_2 + 2HCL \longrightarrow CaCL_2 + 2H_2O$$

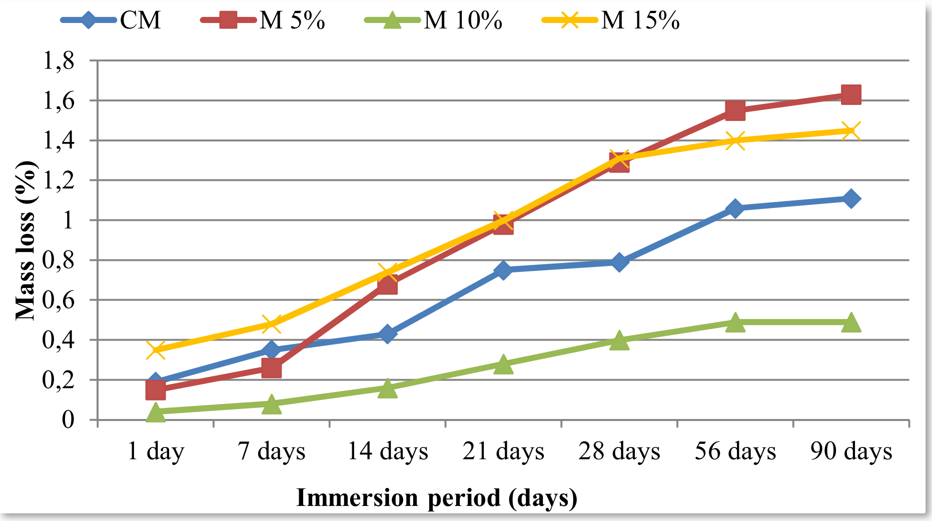

Fig. 10. Loss of mass as a function of immersion period in 5% HCL

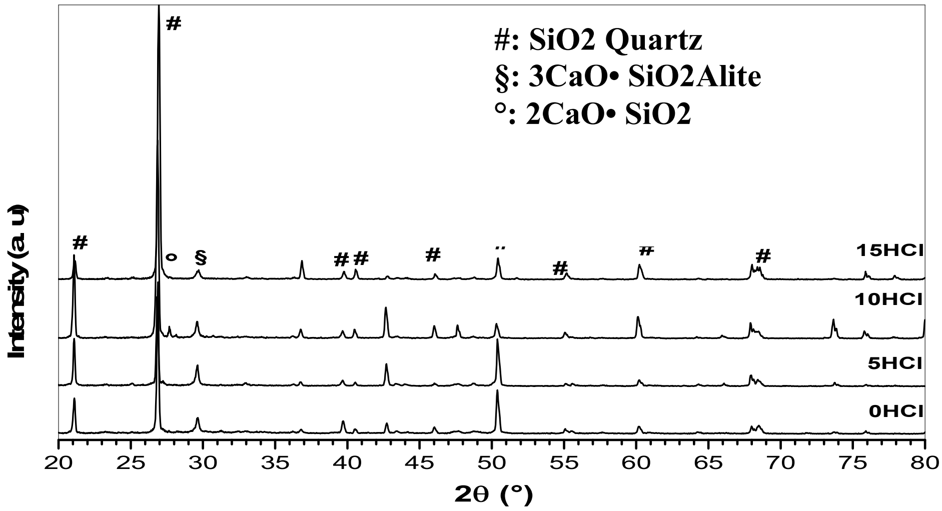

Fig. 11. X-ray diffraction analysis of samples immersed in 5% HCL acid

- **Chemical attack by sulphuric acid (H_2SO_4)**

Figure 12 shows that the incorporation of 5% and 10% of biomass ash leads to a decrease in weight loss in H_2SO_4 acid. Sulphuric acid is a strong inorganic acid whose damaging effect is due to sulphate ions. Its severity increases with its concentration and time. For mortars in contact with H_2SO_4 sulphuric acid, calcium hydroxide $Ca(OH)_2$ (portlandite) reacts with the sulphuric acid to form calcium sulphates $CaSO_4$ which are deposited as gypsum as shown in fig. 13.

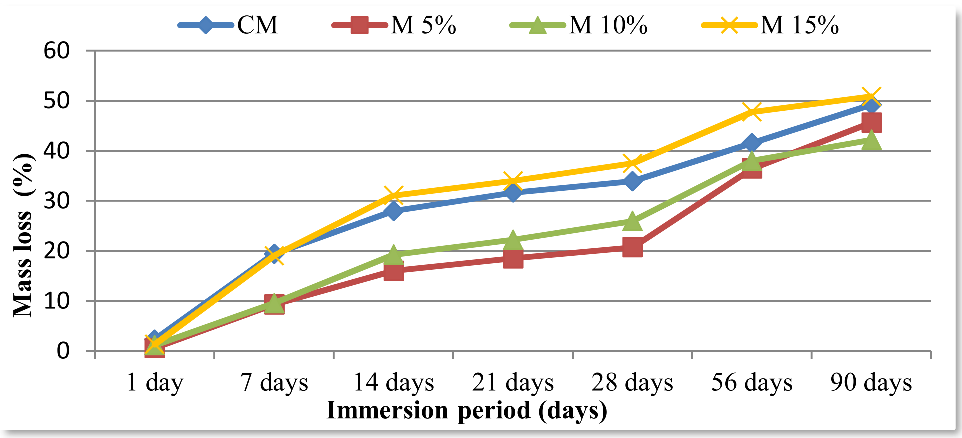

Fig. 12. Loss of mass as a function of immersion period in 5% H_2SO_4

The precipitated gypsum layer is easily leached out resulting in a considerable loss of mass [16].

$$Ca(OH)_2 + H_2SO_4 \longrightarrow CaSO_4 + 2H_2O$$

The deposited gypsum is then leached out, giving us a whitish solution [18]. After chemical degradation, it is observed that the sample not only had an increase in porosity but also a decrease in volume and mass.

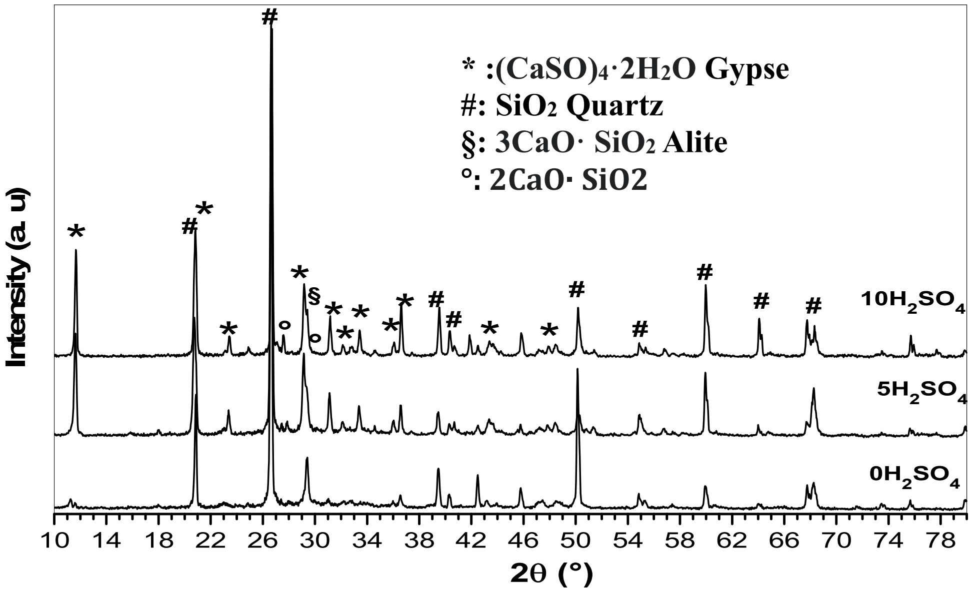

Fig. 13. X-ray diffraction analysis of samples immersed in 5% H2SO4

4.3.7. Dimensional stability (shrinkage and swelling)

- **Shrinkage**

The objective of this test is to measure, as a function of time, the variation in length due to the effects of hydration and desiccation of cement materials.

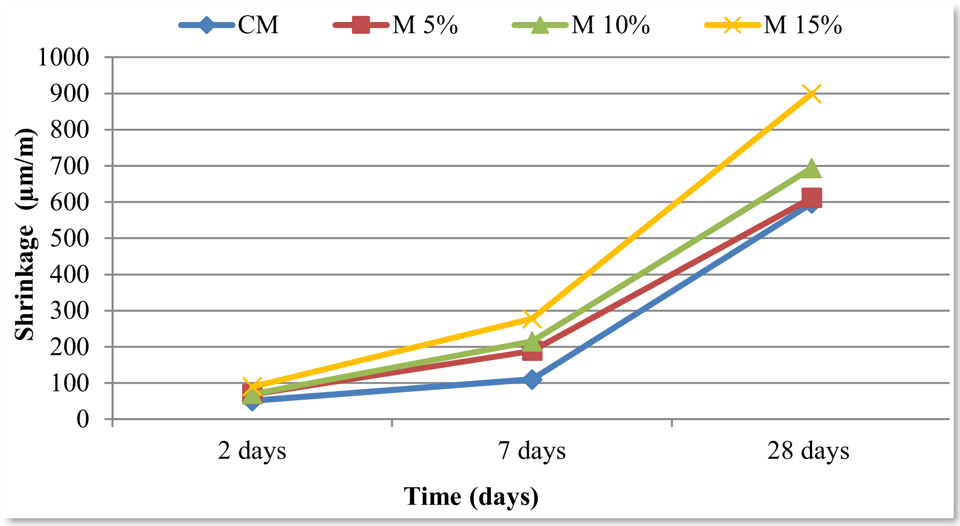

Fig.14. Variation of total shrinkage over time

The analysis of the dimensional stability results (Fig. 14), shows different behaviours of shrinkage according to the substitution rate; mortars containing biomass ash show a higher shrinkage values than those recorded for the control mortar. Indeed, the shrinkage increases proportionally with the age of the mortar, this is due to the hydration of the cement which generates a fine network of capillary pores of the hydrate clusters formed and to the desiccation, but this phenomenon is accentuated compared to the mortars with substitution rates. The addition of microfine materials such as ash can increase the shrinkage phenomenon [19].

- **Swelling**

This test has the same concept as the previous one, the length variation of the specimens is measured. After mixing, mortars swell in the presence of water. Some swellings lead to disorders in the structures (reduced mechanical strength, fissures, etc.).

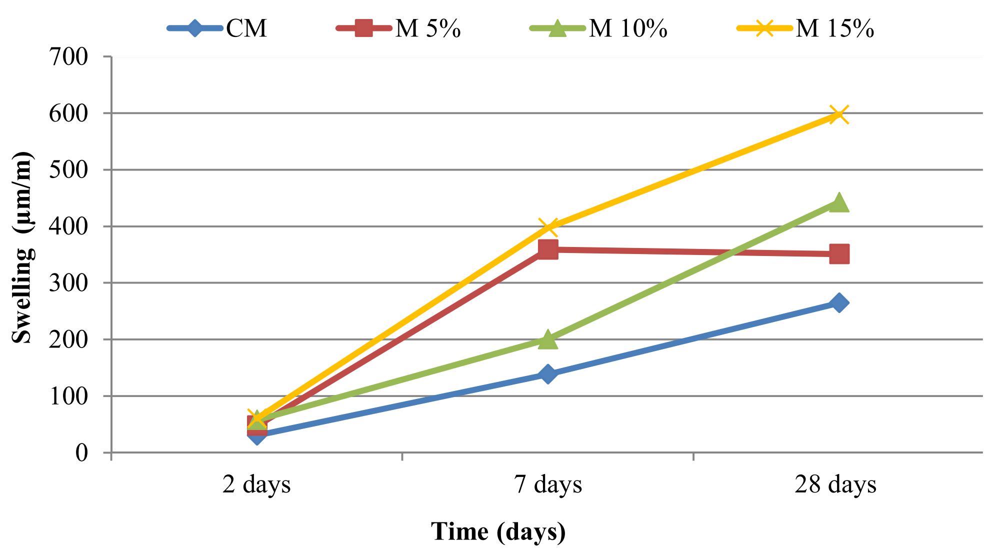

Fig. 15.Variation du gonflement en fonction du temps

From Fig. 15, it can be seen that the swelling values develop with time and the substitution rate. This variation in volume (swelling), is due to the consumption of $Ca(OH)_2$ portlandite and the combination of magnesium oxide and the depletion of free lime during the hydration reactions with the admixture incorporated in the cement [20].

5. CONCLUSION

Based on the results of this study, we can draw the following conclusions:

- The introduction of wood ash leads to a decrease in density, which can be beneficial in certain cases such as lightweight structures and insulating structures.
- The use of biomass ash tends to decrease workability.
- The introduction of biomass ash affects the amount of occluded air.
- Mortar with 5% of wood ash has the best compressive strength at early age.

- The addition of 5% of biomass ash improves flexural tensile strength at the age of 2, 7 and 28 days.
- The use of wood ash increases the immersion absorption of the mortar.
- The introduction of wood ash leads to an increase in capillary absorption.
- Mortar with 5% of wood ash is the most resistant to chloride penetration.
- The mixture with 10% of biomass ash is the most resistant to the hydrochloric acid (HCL) attack.
- Mixtures containing 5% and 10% of ash addition show a better performance in the sulphuric acid (H_2SO_4) and lose mass the least.
- The cement-ash combination has an influence on shrinkage.
- The addition of biomass ash affects the swelling.

Generally speaking, it can be concluded that the valorization of wood ash improves some characteristics and has a negative impact on others, as with the valorization of all wastes, but the results remain acceptable given that it's a subject of replacing the cement, binder and key of construction with residues. This process allows saving raw materials, energy and protecting the environment which is very promising.

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