



The Effect of Adding Barley and Oats Husks Ashes on the Properties of Ordinary Portland cement

Masuoda Farhat ^{1*}, Taha Abdullah ², Fares Fenniche ³, Hassina Hadj Kouider ⁴

mas.ali@sebhou.edu.ly

^{1,2} Materials and Corrosion Engineering Department, Faculty of Engineering, Sebha University, Libya

³ Process Engineering Department, Dynamic, Interactions and Reactivity of Systems Laboratory, Kasdi Merbah Ouargla University, Algeria

⁴ Process Engineering Department, faculty of Science and Technology, Ghardaïa University, Algeria

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Abstract

In this scientific study, the researcher's analysed agricultural waste materials, namely barley husk ash (BHA) and oats husk ash (OHA), as possible substitutes for ordinary Portland cement (OPC). The primary aim was to improve OPC production capacity while mitigating ecological and economic problems linked with traditional OPC production. A range of cement blends, comprising different proportions (0, 5, 10, 15, 20 and 25 wt. %) of BHA or OHA additives in OPC, were prepared and analysed.

Raw material analysis was conducted through X-ray fluorescence technique (XRF), complying with established standards. Cement mixes, resulting from the blends, were scrutinised for their cementing properties and mechanical performance according to international standards. The properties analysed were standard water of consistency (WOC), bulk density, apparent porosity and compressive strength after a hydration period of 28 days. A 15% ash inclusion resulted in the maximum compressive strength of hardened cement pastes, specifically, a 42% increase for reinforcing with BHA (strength value rose from 358 to 510 Kg/cm³) and a 38% increase in strength value for OHA incorporation (strength value increased from 340 to 470 Kg/cm³).

The study's findings showed that the incorporation of 15-20 wt. % of BHA or OHA led to a substantial improvement in the cementing properties examined. It is noteworthy that BHA was found to be more advantageous as cement mixes containing BHA demonstrated superior cementing properties in comparison to the other investigated samples. The superior performance of the cement blends can be attributed to the relatively higher pozzolanic nature demonstrated by BHA, which had a positive impact overall.

Key words: Barley Husk Ash; Oats Husk Ash; Ordinary Portland Cement; standard water of consistency; waste materials

Introduction

Cement is an inorganic material that acts as a hydraulic binder. Upon mixing with water, it forms a paste that can set and harden through hydration reactions. Once solidified, it maintains its strength and stability, even when completely submerged [1]. At present, the global production rate of cement is roughly 4.3 billion tonnes per year, with a projected increase to around 5 billion tonnes per year by 2023. To meet increasing demand and reduce environmental impact, supplementary cementing materials are becoming more commonly used. This is due to the fact that the production of one ton of Portland cement clinker is associated with CO₂ emissions [2–4].

Modern cement formulations often include pozzolanic materials as supplementary cementing agents in Portland cements, replacing some of the clinker to improve the performance of the hydrated cement. These composite or blended cements provide economic, ecological, and technological benefits by reducing energy consumption and CO₂ emissions [5]. The utilization of supplementary cementing materials reduces the amount of lime present in hydrated Portland cement and substitutes it with pore-filling cement hydrates. These hydrates have established improvements in the final strength, impermeability, and ability to resist chemical degradation of the cement [6,7]. Technical term abbreviations have been explained during their first use. Consistent citation and adherence to style guides have been followed. Various supplementary materials, like pozzolans (e.g. natural pozzolans, low calcium fly ash, and silica fume), auto-pozzolans (e.g. high calcium fly ash and blast furnace slag) and crystalline additives (commonly referred to as fillers) are utilized. The vitreous or amorphous structure of these materials primarily accounts for their pozzolanic activity or hydraulicity [8–10]. The level of pozzolanic activity in husk ashes is mostly affected by the amount of the amorphous phase that exists [11]. It has been noted that the ash obtained from this process typically possesses a crystalline structure, which results in unsatisfactory pozzolanic properties. Nevertheless, Mehta's research in 1973 proved that heating rice husks to 600 °C produces ash with an ideal composition for displaying pozzolanic characteristics [8,12,13]. The strength and resistance of cement, according to research [14], mainly depend on the compounds of dual calcium silicate and tri-calcium silicate. Composite tri-calcium silicate is especially important for providing strength after 28 days and sustaining resistance for one year. Furthermore, it is worth noting that the quad-calcium iron compounds, as well as the tri-calcium aluminate, exhibit a minimal contribution to the overall strength, with the latter aiding in early strength development. Through the consideration of the mentioned fundamental components' proportions earlier, it is possible to produce several types of Portland cement [15].

Portland cement, commonly known as OPC or Ordinary Portland Cement, is a fundamental construction material widely used in various everyday applications such as concrete, mortar, stucco, and non-specialty grout. As a result, it is one of the most frequently used cement types globally [16]. The composition of Ordinary Portland Cement (OPC) consists of mainly Portland cement clinker (over 90%), a small portion of calcium sulfate utilized to regulate setting time, and other minor ingredients that maintain permissible limits as per various standards such as EN197.1 in Europe [17, 18]. The chemical structure of OPC is primarily made up of lime (CaO), silica (SiO₂), alumina (Al₂O₃), iron oxide (Fe₂O₃), and sulfur trioxide (SO₃). Explanation of technical abbreviations have been provided at their first use. The production of cement consists of grinding raw materials with suitable ratios of the requisite oxides to attain a desirable fineness, followed by incipient fusion burning in a kiln. This causes the formation of clinker through solid-state chemical reactions [19,20].

To obtain a novel Portland cement component, various bio-based materials are commonly incorporated. Barley husk ash is a significant additive of interest, as barley is a top-ten global crop. In 2021/2022, approximately 145.9 million tons of barley were produced worldwide. However, Libya's grain production is restricted to wheat and barley

due to agricultural land limitations and climatic constraints. These crops are limited to a narrow coastal strip with rainfall and a few irrigated areas in isolated oases. Autumn-sown wheat and barley can be cultivated due to the availability of two primary water sources [21]. Silica is the predominant component found in the ash, making up approximately 90% of its weight. The ash can generally be regarded as impure silica. Ash with minimal or no residual carbon possesses numerous potential applications, extending from soil conditioning to operating as an abrasive constituent in toothpaste [22,23]. The composition of silicon-containing products derived from plants varies based on input and recovery processes, primary product (SiO₂) content, contaminants, particle size, specific surface area, pore volume, and other attributes [24–26]. In addition, there has been significant research interest in utilizing fly ash as a constituent in cement production [27, 28, 29]. The size and fineness of granulomere fly ashes tend to have a significant impact on the strength of the mortar. The hydration reaction is initiated and hastened by tiny particles serving as the reaction's nucleus [30].

The study investigated the effect of adding local barley husk ash and oats husk ash to ordinary Portland cement to decrease CO₂ emissions from the cement manufacturing process. Agricultural waste materials improve ordinary Portland cement quality as they contain a substantial proportion of aluminum oxide, silicon, and iron, which make them potential natural pozzolanic materials. The addition of these materials aims to improve the characteristics of standard Portland cement whilst decreasing its ecological impact by decreasing CO₂ emissions.

2. Experimental Procedures

2.1. Materials

The materials utilized in this investigation are as follows:

1. BHA, Barley husk ash.
2. OHA, Oats husk ash.
3. OPC, Ordinary Portland cement.

Portland cement (Type 42.5 N), which was produced under Libyan specifications (No. 340/2009), was utilized in the experiments. The cement was manufactured at Zliten Cement Factory in Zliten, Libya.

2.1. Processing of Oats and Barley Husk Ashes

The husks of oat and barley treated individually were washed repeatedly with running water and rinsed with distilled water. They were later dried in the sun and roasted for five hours at 800°C.

2.2. Preparation of Cement Pastes

The optimal water content for formulating various cement blends with Ordinary Portland Cement (OPC) using varying weight proportions of Barley Husk Ash (BHA) and Oat Husk Ash (OHA) was determined based on Figure 1's graphical representation. The resultant pastes were moulded into 4x4x4 inch cubic samples within steel containers, following a two-minute thorough dry mixture for a duration of ten minutes.

After molding, the molds containing the paste were placed in a 100% relative humidity environment. After one day, an additional set of samples were removed from the molds to obtain cubic samples that represented the matured cement paste. These samples were subsequently submerged in water until the investigation commenced. Over the course of 10, 14, 21, and 28 days, we extracted three samples from each cement blend in order to evaluate certain aspects including bulk density and apparent porosity.

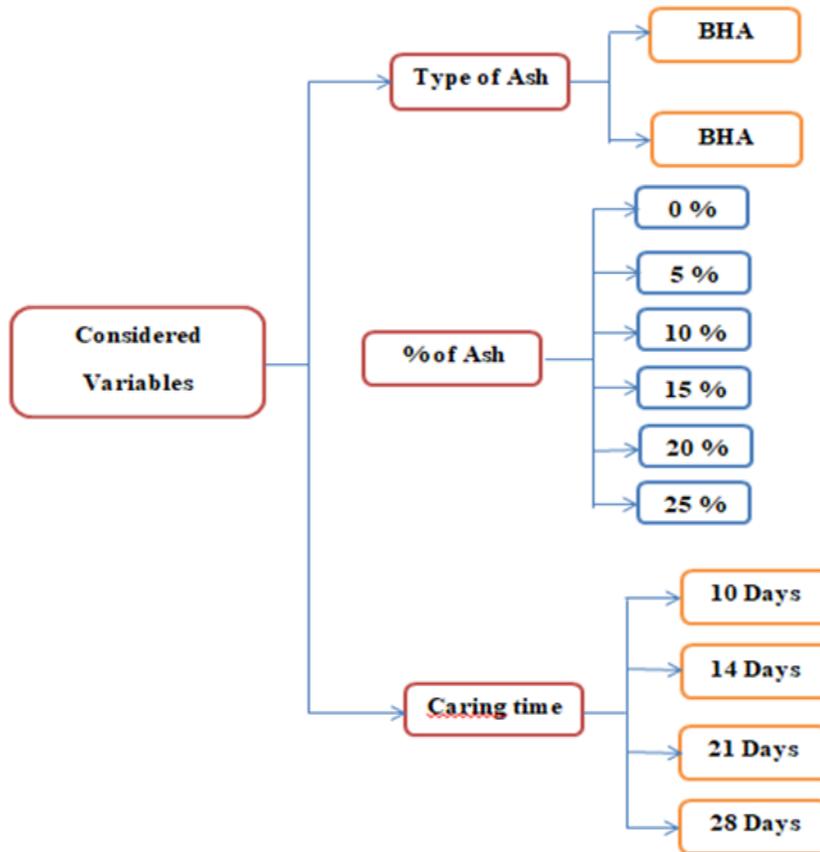


Fig.1 – Methodology (Tested Variables) of research experiments.

During the density test, it was observed that the density of the ash slab was lower than that of OPC.

Table 1 – Density of OPC, BHA and OHA.

1. Materials	2. Density g/cm ³
3. Cement	4. 2.031
5. Barley Husk Ash	6. 0.753
7. Oat Husk Ash	8. 0.702

2.3. Instrumentation

The chemical compositions of Ordinary Portland Cement (OPC), Barley Husk Ash (BHA) and Oats Husk Ash (OHA) were examined via X-ray fluorescence (XRF) analysis. Additionally, the surface area and particle size distribution of the ash materials were evaluated.

The cements were characterized by their water of consistency (WOC) and bulk density (B.D).

(iii) Apparent porosity (A.P).

(iv) Compressive strength of the hardened cement pastes.

3. Results and Discussions

3.1. Chemical composition of Barley ash (BAH) and Oat ash (OAH)

Table 2 displays the primary composition of the cement, with a strong prevalence of CaO (64.4%) and SiO₂ (20.5%), as well as a significant amount of Fe₂O₃ (4.49%). In addition to these major components, minor elements are present, including Al₂O₃ (4.7%), SO₃ (4.6%), and K₂O (0.493%). This comprehensive characterization of the cement composition serves as a basis for understanding its properties and potential utilization.

Table 3 presents a detailed chemical composition analysis of Barley Husk Ash (BA) and Oats Husk Ash (OA) using X-ray fluorescence technique (XRF). The results indicate that both ash samples consist mainly of silica (SiO₂) – BA has 22% and OA has 15.3%. Moreover, varying proportions of Fe₂O₃ are observed, with BA containing 3.81% and OA containing 1.66%. The ash samples contain a range of constituents, including K₂O, P₂O₅, CaO, MnO, CuO, ZnO, and SrO, in varying proportions. A comprehensive chemical analysis provides insights into their elemental makeup, enhancing our understanding of their potential impact on cementitious materials and related industries.

Table.2 – Chemical analysis of the used cement.

Compounds Formula	Content (%)
CaO	64.4
SiO ₂	20.5
Al ₂ O ₃	4.7
Fe ₂ O ₃	4.49
SO ₃	4.6
TeO ₃	0.441
K ₂ O	0.493
MnO	0.0805
NiO	0.0708
CuO	0.0209
PbO	0.0045

Table.3 – Chemical Composition of BAH and OAH.

Compounds Formula	Content (%)	
	BAH	OAH
K ₂ O	26.2	24.1
P ₂ O ₅	24.4	33.7
SiO ₂	22.7	15.3
CaO	6.38	2.34
Fe ₂ O ₃	3.81	1.66
TeO ₂	2.48	1.45

Sb ₂ O ₃	0.07	0.417
NiO	0.547	0.611
MnO	0.382	0.811
ZnO	0.238	0.492
CuO	0.213	0.313
SrO	0.0563	0.0159

3.2. Cementing Properties being Prepared for Cement Pastes

3.2.1. Water Requirement (Water of Consistency)

Figure 2 highlights the changes in standard consistency percentages at varying levels of Barley Husk Ash (BHA) or Oats Husk Ash (OHA) usage as substitutes for conventional cement. The graph shows a steady increase in the water requirement for standard consistency with greater ratios of BHA or OHA replacing cement. Technical term abbreviations such as BHA and OHA are explained when first used. This occurrence stems from two important factors: the ashes exhibit a relatively high specific surface area, ranging from 11,000 to 12,000 cm²/g, which is significantly greater than that of OPC and its value of 3100 cm²/g; and the ashes possess an inherent hygroscopic nature, which enables them to absorb more substantial quantities of mixing water. These empirical trends align with the findings of previous researchers [31–33] and confirm the consistency of the observed behaviour across distinct investigations. The water demand trends highlighted here emphasise the complex chemistry of ash properties and their impact on the fluidity and workability of the cementitious mixture. This study not only provides insight into the behaviour of these materials, but also offers valuable guidance for developing more effective and customised cement blends.

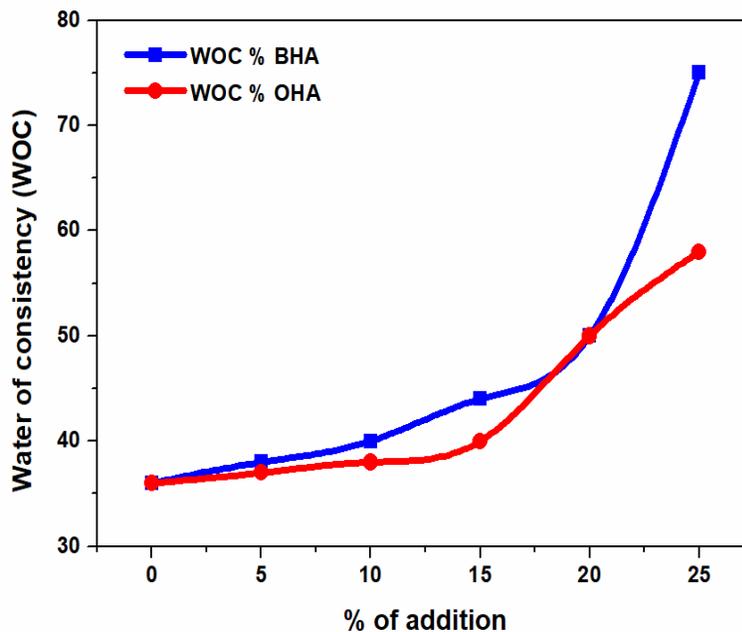


Fig.2 – Water of consistency (WOC) of different cement mixes.

3.2.2. Bulk Density and Apparent Porosity

Figures 3 and 4 depict the data concerning the bulk density measurements of solidified cement pastes, distinguished by different levels of ash content. Furthermore, Figures 5 and 6 present a comprehensive overview of the corresponding ratios of apparent porosity. The investigation's outcomes highlight a clear trend: a decrease in bulk density coincides with a corresponding increase in apparent porosity, providing evidence for a direct correlation between the rise in Barley Husk Ash (BHA) or Oats Husk Ash (OHA) concentrations and these physical properties. The observed trend can be explained by the intrinsic characteristic of bulk density showing a linear decrease, in contrast to the converse linear increase evident in apparent porosity, as the concentrations of BHA or OHA increase within the composite matrix. This affiliation is highly corroborated by the considerably reduced density of either BHA or OHA when compared to that of Ordinary Portland Cement (OPC), which has been recognised as a crucial factor in previous research [34].

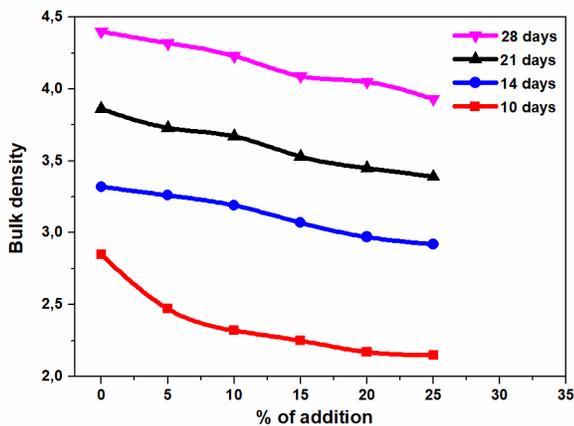


Fig.3 – Bulk density of the hardened cement pastes containing different contents of BHA at different curing times.

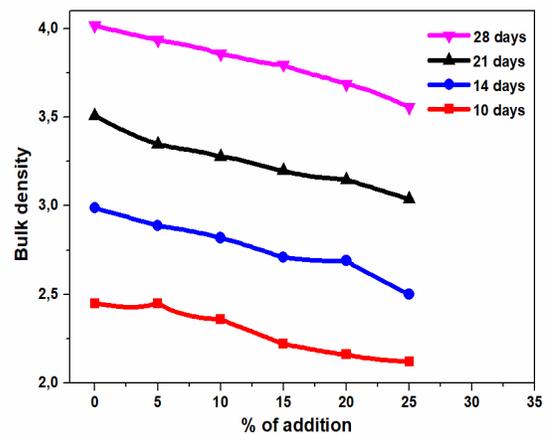


Fig.4 – Bulk density of the hardened cement pastes containing different contents of OHA at different curing times.

It should be noted that the addition of ash has differing effects on the porosity of solidified cement pastes. Specifically, BHA has a stronger influence than OHA. Additionally, the order of bulk density enhancement is as follows: cement with BHA exceeds that of cement with OHA. It should be noted that an observable trend towards higher bulk density values is apparent as the curing duration progresses from 10 to 14 days, and further to 21 days, even up to 28 days of hydration. This is accompanied by a gradual reduction in apparent porosity percentages, observed across specified levels of BHA or OHA, as well as in the control sample without ash content. The time evolution of these physical characteristics illuminates the intricate interplay between the cementitious matrix's reaction to hydration and ash inclusion. This comprehensive understanding not only enhances our fundamental grasp of these materials' behaviour but also expands their potential applications in various engineering domains.

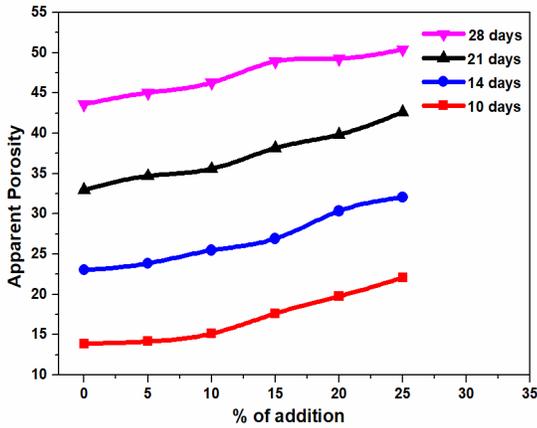


Fig.5 – Apparent porosity of the hardened cement pastes containing different contents of BHA at different curing times.

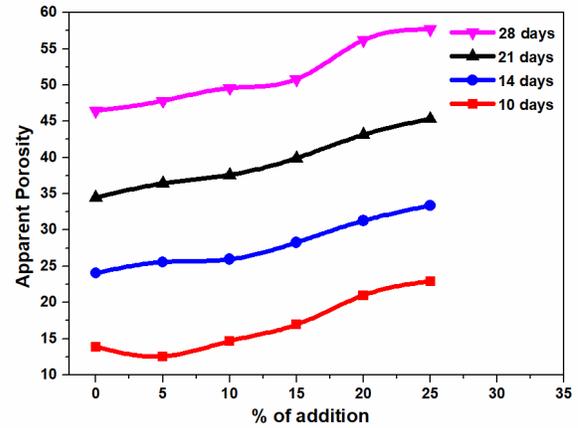


Fig.6 – Apparent porosity of the hardened cement pastes containing different contents of OHA at different curing times.

3.2.3. Compressive Strength of the Hardened Cement Pastes

Figure 7 displays the compressive strength values observed in hardened cement pastes after a 28-day hydration period for different proportions of BHA and OHA. The results demonstrate a noticeable enhancement in the compressive strength of the cement solidified as the concentrations of BHA or OHA increase. This trend persists until either ash type is incorporated into the cement at a percentage of 15 wt.%. This section indicates the peak compressive strength of solidified cement pastes. This corresponds to a 42% growth in compressive strength for BHA reinforcement (the strength value rose from 358 to 510 Kg/cm³) and a 38% increase for OHA addition in the cement paste (the strength value rose from 340 to 470 Kg/cm³). Moreover, the strength slightly decreases but keeps on being higher than the control sample, with only an 8% increase at a 20 wt. % BHA ash level. However, reducing the ash content from 20 wt. % to 25 wt. % becomes detrimental to the compressive strength of both BHA and OHA. Consequently, solidified cement pastes exhibit values of compressive strength closer to the control samples without any ash. This is evident from the results found in ref. [35], wherein the addition of 15% WSA (wheat straw ash) led to the highest level of compressive strength in the concrete mix. However, a reduction in compressive strengths was observed when other increments (5%, 10%, 25%) were applied.

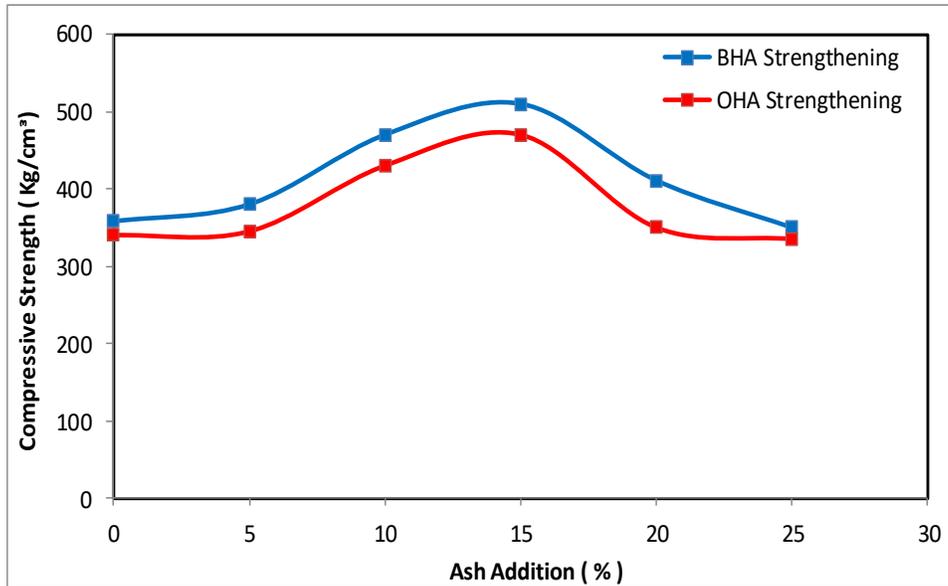


Fig.7 – 28 days compressive strength of the hardened modified cement pastes.

These findings indicate that the optimal amount of either BHA or OHA for achieving greater compressive strength lies within the 15–20 wt. % range. Alternatively, a compressive strength level that is sustained within the control sample's spectrum may be maintained. Additionally, it is evident that cement samples containing BHA display significantly higher compressive strength values compared to those containing OHA. The addition of pozzolanic materials to cement results in the formation of compounds that enhance the strength and other important properties of the solidified cement pastes. This increase in compressive strength is attributed to both the increased pozzolanic character of the ashes and a filler effect. The evidence for this enhancement is supported by previous work [36,37,38–39]. As previously mentioned, the complicated procedure can be attributed to the creation of crucial hydration products, specifically $\text{Ca}(\text{OH})_2$ and calcium silicate hydrates (C–S–H), within the specimens containing ash [40,41]. These findings furnish comprehensive explanations of the intricate correlations among ash proportion, pozzolanic treatments, and the ensuing compressive potency, which considerably enhance our comprehension of the mechanical conduct of these cementitious materials augmented with ash.

The increase in strength can be attributed to the finer particle size of the ash additives compared to Ordinary Portland Cement (OPC). This smaller particle size results in the separation of larger pores and facilitates an increase in the number of nucleation sites, leading to the precipitation of pozzolanic reaction products within the cementitious matrix [30]. Thus, this elevation stimulates a heightened pozzolanic reaction, refining the paste's overall pore structure. The amount of calcium hydroxide present in the paste concurrently reduces with greater hydration [42]. The reinforcement of the cementitious composite's strength properties follows from this intricate interplay of particle size, pozzolanic processes, pore structure, and hydration dynamics.

4. Conclusions

The experiment yielded significant findings on the effects of adding Barley Husk Ash (BHA) and Oats Husk Ash (OHA) to cementitious matrices. Firstly, an increase in the proportion of the additive (BHA or OHA) directly resulted in a rise in standard water of consistency (WOC). This indicates a linear correlation between the amount of additive and water demand. Secondly, it was observed that with the increase in ash content, the bulk density reduces proportionally. This can be attributed to the lower densities of the ash additives in comparison to Ordinary Portland Cement (OPC). Conversely, apparent porosity displays an opposite trend, where it increases with the increase in ash content. Additionally, an evaluation of cement compositions containing either BHA or OHA demonstrates that formulations containing these additives up to 20 wt. % consistently conform to international standard specifications, confirming their adherence to established industry regulations.

With an increase in ash content up to 15 wt. %, there is a gradual improvement in compressive strength. This point represents the maximum compressive strength of the hardened cement pastes, resulting in a 42% increase in compressive strength for BHA reinforcement (from 358 to 510 Kg/cm³) and a 38% increase for OHA incorporation in cement paste (from 340 to 470 Kg/cm³). At 20 weight per cent, a reduction in compressive strength is visible, but it falls within the range of values seen in the control sample. Nevertheless, a slight decrease in compressive strength values is noticeable when the ash percentage surpasses 20 wt. % (at 25 wt. %).

Ultimately, upon comparing the efficacy of Barley Husk Ash (BHA) and Oats Husk Ash (OHA) with cement pastes, the former is revealed to be the superior alternative. Compared to the tested samples, cement blends that include BHA exhibit improved characteristics for cementing. This improvement can be partly attributed to the considerably enhanced pozzolanic nature of BHA. The detailed investigation has highlighted the complex relationships between ash content, physical characteristics and performance. This information can be used to create more effective and customised cementitious composites.

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