

Physical and Long-Term Mechanical Properties of Agricultural Ash Blended Cement

H.H.M. Darweesh^{1*}

¹ Department of Refractories, Ceramics and Building Materials,
National Research Centre, Dokki, Cairo, 12622, EGYPT

*Corresponding Author: hassandarweesh2000@yahoo.com

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Abstract

Walnut shell ash (WSA) is a new supplementary cementitious material (SCM) for cement. The tests for water of consistency, setting time (Initial and final), pozzolanicity, free lime content, flexural and compressive strength were conducted. Results demonstrated that the cement pastes incorporating up to 25 wt. % WSA possesses suitable setting times, i.e. not high and not low, physical, pozzolanic activity and mechanical properties. The addition of WSA increased the w/c ratio and setting times. The total porosity gradually decreased with the increase of WSA content (26.13 %) while the bulk density increased (2.0112 g/cm³). The strength improvements were due to of formation of fibrous CSH gel, high hydration level, low porosity, high density and pozzolanic reactions of WSA with the resulting free lime from the hydration process. The free lime contents of the OPC (W0) increased with the curing times (8.78 %), while with WSA the free lime content decreased due to the pozzolanic activity of WSA (4.23 %). The increase of WSA content > 25 wt. %, the specific properties were adversely affected, i.e. the optimum added content of WSA does not exceed 25 wt. %.

1. Introduction

CO₂ emission during the manufacture of cement is the main anthropogenic contributor to global warming which may lead to human casualties and substantial economic losses [1-10]. Cement is the main building material that permeates nearly all sides of our built environment, from dams and bridges to houses and the various buildings [1-4]. The cement industry is also a major source of about 5–7% of global CO₂ emissions, where one ton of cement emits about 560–630 kg of CO₂ [3-8, 11]. The emissions often cause severe climate change worldwide that is being detrimental to human health and well-being. Carbonization in cement industry is to improve the energy efficiency of cement production processes, and to use alternative materials to partially replace cement in cement structures [12-14]. However, the wider acceptance of these alternatives faces significant barriers. Utilization of partially replacing CO₂-intensive cement is one of the most effective and practical long-term solutions to reduce the heavy environmental burden relative to cement production [15-18]. Various industrial by-products as granulated blast furnace slag (GbfS), silica fume (SF), fly ash (FA), sugar can bagasse ash (SCBA) and many others could be used as cement substituents [19-36]. These wastes and its diverting away from landfills could be promoted its value [37,38], which it is a step to meet the reduction purposes for excess wastes [39,40]. Using of SCMs for construction can reduce the environmental pollution due to the accumulation of materials, save the costs of both waste disposal and producing concrete [41-44]. The bio-derived ashes, such as walnut shell ash, have the potential to act as a SCM alternative in cementitious composites due to their similar physicochemical properties to conventional SCMs. As biomass ash, walnut shell ash has abundant silica and calcium dioxide contents that play a vital role in hydraulic and pozzolanic reactions [45–47]. Many studies on the various agricultural wastes as rice husk ash [48,49], cotton

stalk and palm leaf ashes [50], rice straw [51], olive waste ash [52], and nano-palm oil fuel ash [53] as SCMs have reported promising results. Moreover, the finely ground agriculture waste ashes act as filler materials, improving its density and microstructure of cementitious composites [54,55]. The reliable utilization of walnut shell ash as a SCM and enrich the role of biomass-derived ash in cementitious materials.

The potential reduction emission of CO₂ due to the partial addition of an agricultural waste (WSA) to cement is a step of a more sustainable construction practice. Moreover, it is essential to mitigate environmental concerns related to cement production. This study deals with WSA as a valuable resource because it provides a feasible and an economical solution to waste management. This contributes to the improvements of the mechanical durability. Thereby, it enhances its long-term performance. The increased demand of alternative building materials for construction, WSA could be considered as a suitable and efficient alternative. This research also deepens the scientific understanding of the role and potential applications of biomass ash in cementitious materials.

2. Experimental

2.1 Raw Materials

The used raw materials in the current study are Portland cement (OPC) which was delivered from Sakkara cement factory, Giza, Egypt. The elementary phases of the OPC are tabulated in Table 1, while the oxide composition of the OPC and Walnut shell ash (WSA), as measured by an X-ray fluorescence technique (XRF) are summarized in Table 2. The physical properties of the used OPC cement and WSA are shown in Table 3. The Walnut shell (WS) was first dried in a drying oven and kept at 100 °C for 48 hours. The dried WS was calcined in a furnace up to 800 °C for 2 hours. Then, it was put into a ball mill for 2 min to obtain WSA [56-58]. The particle size distributions of the OPC and WSA are shown in Fig. 1, where the WSA is the higher fineness, and the OPC is the lower. Table 4 shows the constitutions of the various cement mixtures. The WSA was substituted for cement to prepare cement mixtures.

Table 1 Mineralogical composition of OPC sample, wt. %

Material	C ₃ S	β-C ₂ S	C ₃ A	C ₄ AF
OPC	46.81	28.43	5.90	12.56

Table 2 Chemical composition of materials (%)

Materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	LOI
OPC	20.58	5.03	3.38	63.32	2.01	1.23	0.68	2.06	1.76
WSA	56.12	17.26	7.88	9.22	3.29	0.96	2.98	1.60	1.65

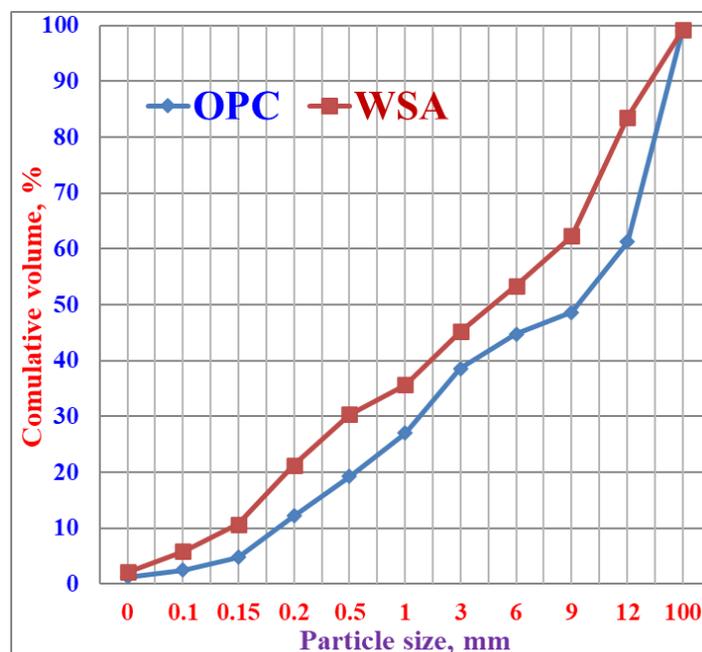


Fig. 1 Grain size distribution of the raw materials

Table 3 Physical properties of the raw materials, wt. %

Materials	Specific gravity	Density, g/cm ³	Blaine surface area, cm ² /g
OPC	3.15	3.12	3564
WSA	2.66	2.87	5683

Table 4 Composition of cement mixtures, wt. %

Materials	W0	W1	W2	W3	W4	W5	W6	W7
OPC	100	95	90	85	80	75	70	65
CSSA	0	5	10	15	20	25	30	35

2.2 Preparation and Methods

During the preparation of cement mixtures, different dosages of WSA (0, 5, 10, 15, 20, 25, 30 and 35 wt. %) were added to the OPC. These OPC/WSC mixtures were categorized into eight batches as W0, W1, W2, W3, W4, W5, W6 and W7, respectively. The blending process of the various cement blends was done in a porcelain ball mill containing 2-4 balls for two hours to assure the complete homogeneity of all cement blends.

2.3 Water of Consistency and Setting Times

Water of consistency (w/c- ratio) and setting time are measured using Vicat apparatus [36,59,60]. Water of consistency (WC) was determined from the following relation [61].

$$WC, \% = A / C \times 100 \quad (1)$$

Where, A is the amount of water taken to produce a suitable paste, C is the amount of cement (300 g). The initial setting time (IST) is the time taken to reach the initial set, while the final setting time (FST) is the time taken to reach the final set of the paste [62]. The cement pastes were then cast using the predetermined water of consistency, i.e. during mixing, the measured w/c-ratio was poured into the cement portion inside the mixer and then run the mixer for about 5 minutes at an average speed of 10 rpm in order to have a perfect homogenous mixture. The cement pastes were then molded into one-inch cubic stainless-steel molds of dimensions 2.5 x 2.5 x 2.5 cm³ using about 500 g cement mix, vibrated manually for three minutes and then on a mechanical vibrator for another three minutes [63]. The surface of the molds was smoothed using a suitable spatula. Thereafter, the molds were kept in a humidity chamber for 24 hours under 95±2 RH and room temperature of 20 ± 2 °C, demolded in the next day and soon immersed in water till the time of testing at 1, 3, 7, 28, 90, 180 and 270 days.

2.4 Water Absorption, Bulk Density, and Total Porosity

Water absorption, bulk density and total porosity [21,64] of the hardened cement pastes could be calculated from the following relations:

$$WA, \% = (W1 - W2) / (W3) \times 100 \quad (2)$$

$$BD, \text{g/cm}^3 = (W1) / (W1 - W2) \quad (3)$$

$$\varepsilon = (0.99 \times W_e \times BD) / (1 + W_t) \quad (4)$$

Where, W1, W2, W3, ε , W_e and 0.99 are the saturated, suspended, dry weights, total porosity, free or evaporable water content and specific volume of free water, respectively.

2.5 Mechanical Properties

2.5.1 Flexural Strength

Flexural strength (FS) was calculated [65,66], whereas the samples were marked at three points adjusting to place them on the correct point of contact (Fig. 2). Then, the FS was obtained from the following equation:

$$FS = 3 (PL) / 2 (b) (d) / 10.2 \text{ (MPa)} \quad (5)$$

Where, L: load taken, P: beam or loading of rupture, b: width, d: thickness.

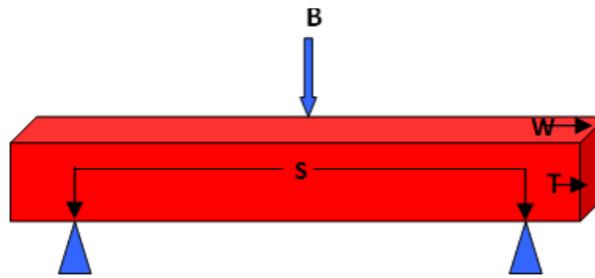


Fig. 2 Schematic diagram of bending strength, B: Beam or loading of rupture, S: Span, W: Width and T: Thickness

2.5.2 Compressive Strength

The compressive strength (66,67) could be also calculated from the following relation:

$$C_s = L \text{ (kN)} / S_a \text{ (cm}^2\text{)} \text{ kN/m}^2 \times 102 \text{ (kg/cm}^2\text{)} / 10.2 \text{ (MPa)} \quad (6)$$

Where, C_s : Compressive strength (MPa), L: load (kN), S_a : surface area (cm²).

2.6 Chemically Combined Water Content

The chemically-combined water content at each hydration age was determined on the basis of ignition loss [30,31,68,69] as follows:

$$W_n, \% = W_1 - W_2 / W_2 \times 100 \quad (7)$$

Where, W_n , W_1 and W_2 are combined water content, weight of sample before and after ignition, respectively.

2.7 Free Lime Content

The free lime contents (FL_n) of the hydrated samples pre-dried at 105°C for 24h were also measured [20,21,31,36,45,47,48]. The X-ray fluorescence (XRF) was used to analyze the chemical and mineralogical composition of OPC and WSA raw materials as well as identifying crystalline phases of OPC cement. Subsequently, based on the quantitative analysis of Ca (OH)₂ consumption and the degree of cement hydration was conducted to explore the effect of WSA on the long-term strength of cement pastes.

2.8 FTIR and SEM

Some selected samples were investigated using infrared spectroscopy (FTIR) to determine the functional groups in a material and scanning electron microscopy (SEM) to determine the surface morphology of a material. The FTIR spectra were performed by Pye-Unicum SP-1100 in the range of 4000-400 cm⁻¹. The SEM images of the fractured surfaces, coated with a thin layer of gold, were obtained by JEOL-JXA-840 electron analyzer at accelerating voltage of 30 KV. Each group comprised three specimens where the arithmetic mean was considered for each group to determine the corresponding strength values, noting that any abnormal data must be excluded.

3. Results and Discussion

3.1 Water of Consistency and Setting Times

Fig. 3 demonstrates the water of consistency and setting time of cement pastes with and without WSA. The water of consistency has been increased with the increase of WSA content. Also, the setting times (initial and final) are displayed the same trend as water of consistency. When the WSA content increased from 5 to 35 wt. %, the initial and final setting times increased from 3 to 4.5 hours and from 5 to 6 hours, respectively. The extended workable life can be ascribed to that silica-rich content of WSA that is not involved in the early-age hydration of cement. This is mainly due to the reduction of Ca (OH)₂ for secondary cement hydration. This phenomenon also occurs when applying other SCMs as silica fume [70,71], fly ash [71,72], and sewage sludge ash [73,74] to OPC cement. This retardation action due to the higher addition of WSA on setting time may be inhibited its practical application. Therefore, the higher content of its addition must be prevented. It is stipulated that the initial setting time should

be not less than 50 min. and not more than 2.5 hours, whereas the final setting time should be not less than 2 hours and not more than 8 hours [75]. So, the optimum substitution level of WSA for OPC should not exceed than 20 wt. %.

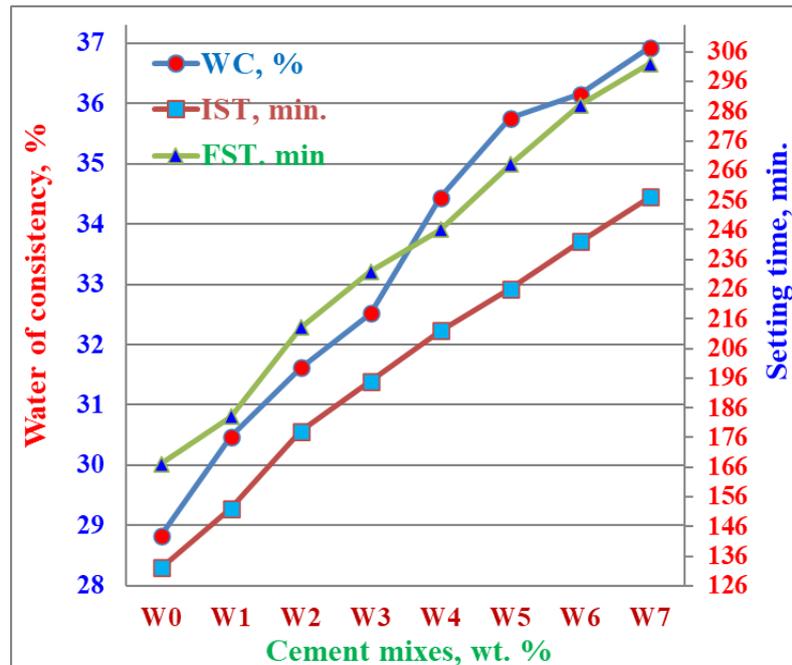


Fig. 3 Water of consistency and setting times of the various cement pastes blended with different ratios of WSA

3.2 Flow Value

Fig. 4 demonstrates that the flow value of cement pastes with WSA. It shows that as the WSA content increases, the flow value is progressively decreased. So, the flow value was shortened from 189 mm to 110 mm with increasing of WSA up to 35 wt. %. The same trend was obtained fluidity other cement pastes blended with other agro/waste ashes [70-73]. This is essentially contributed to the surface properties of WSA, where the specific surface area of WSA ($5400 \text{ cm}^2/\text{g}$) is higher than that of OPC ($3600 \text{ cm}^2/\text{g}$). The higher specific surface area of WSA enables it to absorb more water molecules to form large water film areas [73]. This strong water demand of WSA often decreases the amount of water in the surface of cement clinker [31,74]. This impedes the moving of fresh cement particles. The addition of the high specific surface, angular and irregular shape surface WSA (Fig. 3d and e) increases the viscosity of WSA particles in cement mixtures. This is contributing to the decreased work ability of WSA-based cement paste [70-72,76-79].

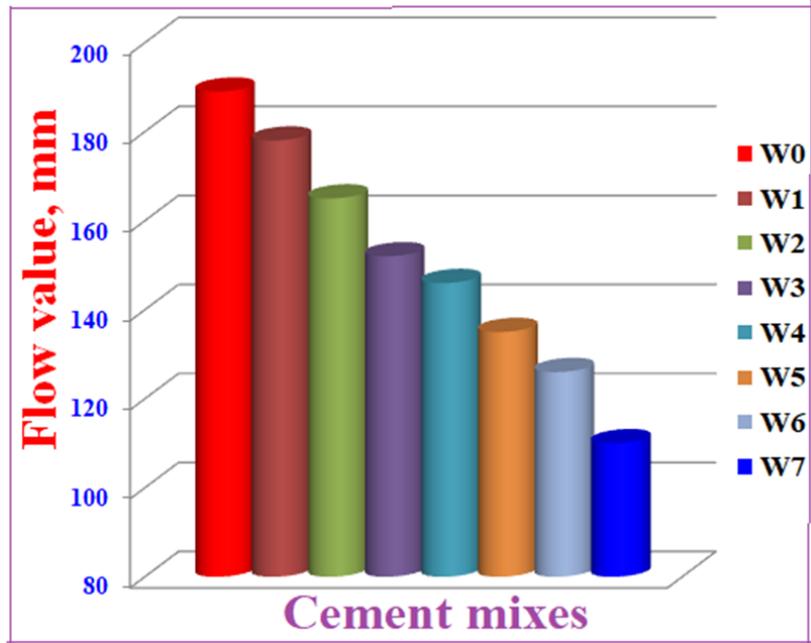


Fig. 4 The flow value of the various cement pastes blended with different ratios of WSA

3.3 Hydration and Pozzolanicity

To assess the hydration and pozzolanicity of cement pastes containing different ratios of WSA, Fig. 5 shows the experimental results of free lime analysis up to 365 days. The concentration of calcium ions, Ca^{2+} or free lime content, $Ca(OH)_2$ decreased with the substantially increasing substitution of WSA. The reduced Ca^{2+} of the cement suggests that Ca^{2+} or portlandite released during the hydration of cement phases have been gradually consumed due to the promoted pozzolanic reaction of WSA with Ca^{2+} on account of increasing time of hydration and replacement levels of WSA [80]. In addition, the increasing addition of WSA from 0 to 25 % led to a gradual loss of portlandite due to pozzolanic reactions of WSA with both di- and tricalcium silicate cement phases, which are much similar to other bio-based ashes, such as palm ash [53,81]. However, the increase of portlandite content was observed with any further increase of WSA content [W6 and W7]. This is essentially attributed to that the high volume of WSA possessed a great percentage of unhydrated cement phases, i.e. this impedes cement hydration with the formation of insufficient hydration products for pozzolanic reactions [82]. These results indicated that the WSA with a replacement level of 0–25 % for OPC in this study can be qualified as pozzolanic cement.

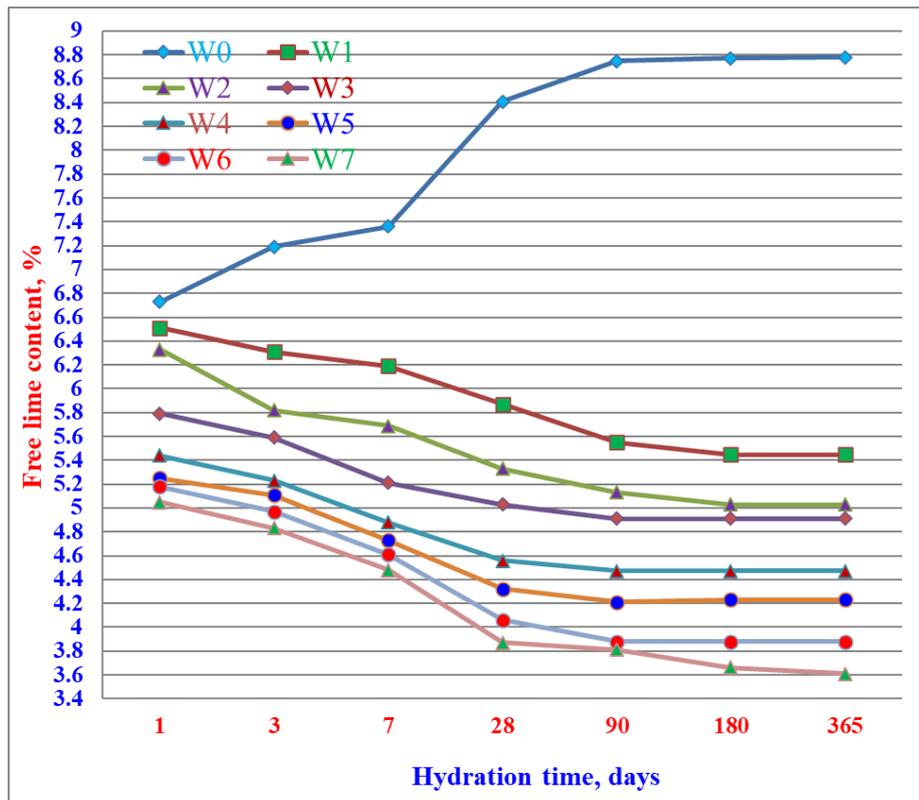


Fig. 5 Free lime contents of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

3.4 Total Porosity and Bulk Density

The total porosity (TP) and bulk density (BD) of the various cement pastes blended with different ratios of WSA hydrated up to 365 days are represented in Fig. 6 and Fig. 7, respectively. Generally, the BD of the various blended cement pastes improved and enhanced with the substitution of WSA until the cement pastes incorporated 25 wt. % WSA (W5), i.e. 2.0112 g/cm³, and then the BD started to decrease with any further addition of WSA (W6 and W7), i.e. 2.0064 and 2.0059, respectively. On contrast, the TP gradually decreased slightly as the WSA content increased in the cement mix. This continued up till 25 wt. % WSA (W5), and then the TP started to increase (W6 and W7). The increase of BD is mainly due to three factors. The first is the normal hydration process of the main major phases of the cement [16,17,21,62], the second is the pozzolanic property of the WSA with the evolved Ca (OH)₂ from the first process [28-30,39,50,68], while the third is the filling action of the free fine WSA that close the available pore spaces between particles [62,68,79]. The decrease of BD is essentially contributed to two factors too. The first is the deficiency of the main hydrating portion of the cement [62,62,67,74,79,83]. The second factor is that the higher incorporation of WSA particles at the expense of OPC hindered the hydration process, i.e. the rate of hydration is adversely affected and decreased. So, the BD decreased. This would be reflected negatively on the TP where it tended to increase. Hence, the higher content of WSA 25 wt. % (W5) must be avoided.

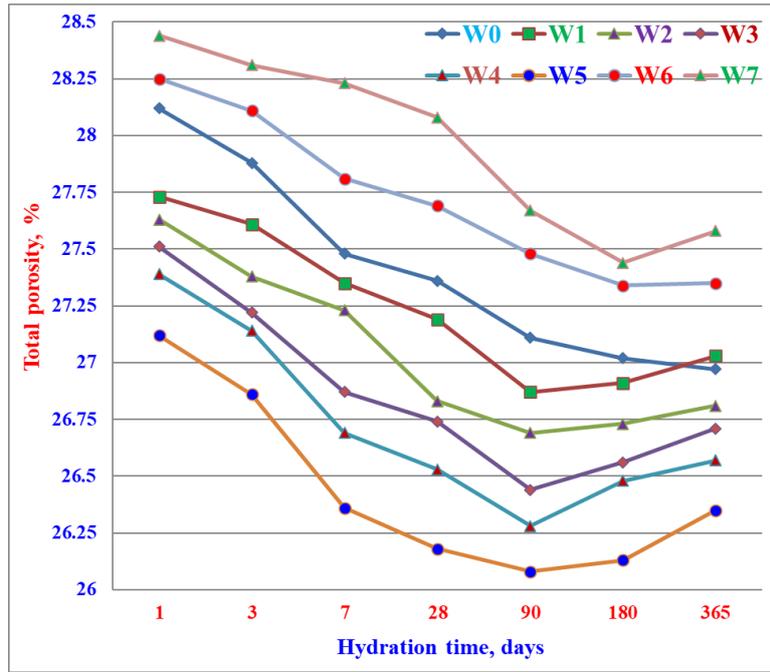


Fig. 6 Total porosity of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

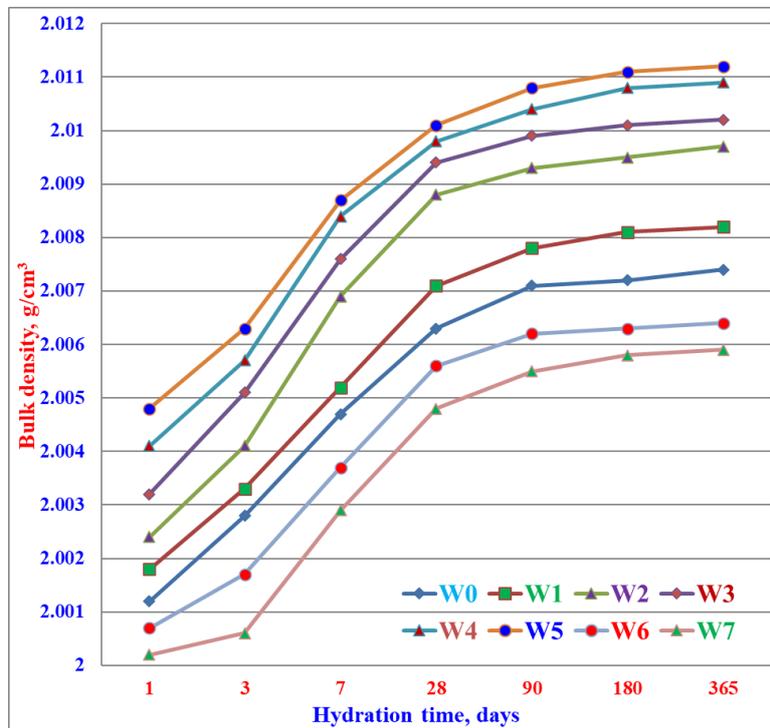


Fig. 7 Bulk density of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

3.5 Heat of Hydration

Experimental results of heat of hydration (HH) of the various cement pastes blended with different ratios of WSA hydrated up to 365 days are represented in Fig. 8. The HH of the OPC (W0) is generally increased up to 90 days of hydration (12.58 kj.g.10⁵) and then seemed to be constant up to 365 days (12.43 kj.g.10⁵), whereas the other blended cements (W1-W7) only increased up to 90 days and then decreased onward up to 365 days [62, 67,69]. The HH of cement batches W1-W5 are slightly higher than those of the blank (W0) at all hydrating times, while that of batches W6 and W7 incorporating higher WSA contents is lower than those of the other cement batches [53.61,69]. Moreover, it even exhibited lower values than those of the blank (W0).

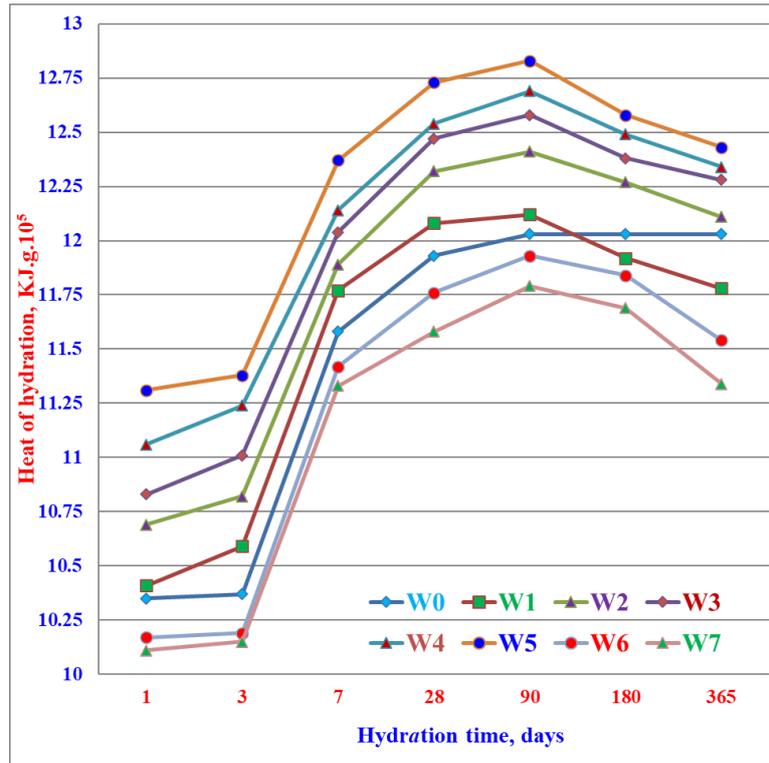


Fig. 8 Heat of hydration of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

3.6 Mechanical Properties

3.6.1 Flexural Strength

The flexural strength of the various cement pastes containing different ratios of WSA hydrated up to 365 days is presented in Fig. 9. Generally, the flexural strength improved and enhanced with the progressive of hydration times due to the normal hydration process of the cement phases [21,28,29,62,83]. The flexural strength of cement pastes blended with WSA slightly improved and enhanced during the early hydration ages up to 7 days, and then faster and higher during the later ages of hydration. This is essentially due to both the normal hydration process of the main phases of the cement and the pozzolanic activity of WSA with the resulting $\text{Ca}(\text{OH})_2$ from the first process, i.e. the flexural strength increased due to the decrease of CaO ion concentration inside the cement matrix [39,61,68,79]. This confirms the pozzolanic reactivity of WSA [83-86]. With the increase of WSA content, the flexural strength also improved and enhanced, but only up to 25 wt. %, i.e. 24.93 MPa and then adversely affected and decreased, i.e. 24.82 MPa [62,67,74]. There was a linear mapping of CaO reduction. This is also attributed to the generation and creation of additional CSH gels brought on by CaO reduction [87-89]. The decrease of flexural strength with the higher amounts of WSA [W6 and W7] is due to the higher percentages of the unreacted or hydrated cement particles. These desirable strength properties indicated that WSA is an attractive supplementary cementitious material for construction applications in terms of long-term mechanical properties. The inhibited cement hydrations with larger replacing of WSA can be attributed to the physical, chemical origins of WSA and its composition which lowers the concentration of Ca^{2+} and OH^- ions in the cement mixtures [90]. Moreover, the decrease of the major phases of the cement (C_3S , C_2S , C_3A and C_4AF) due to the addition of WSA led to retard the rate of hydration of the cement paste [91], and increases the interparticle pores or spaces between cement particles and limits the moving of the reactive ions in the cement [92]. So, the optimum content of WSA is 25 wt. %, and the higher addition of WSA > 25 wt. % (W6 and W7) must be eliminated.

3.6.2 Compressive Strength

Fig. 10 demonstrates the compressive strength of the different cement pastes containing different ratios of WSA hydrated up to 150 days. The compressive strength is generally increased due to the normal hydration process of the major phases of the cement [62,90,91]. The compressive strength values of the cement pastes incorporated WSA marginally increased during the early ages of hydration up to 28 days, and then largely improved and enhanced during the later ages of hydration, i.e. it exhibited better values compared with those of the reference

(W0). This is essentially contributed to that the WSA has pozzolanic characters that are promoting the strength development at later ages of hydration. Also, the filling action played an important role to decrease the open pores which affected positively on the total porosity and often reflected positively on the compressive strength. As the compressive strength values increased with the hydration time, it also increased with the increase of WSA content, but only up to 25 wt. %, i.e. 57.36 MPa and then adversely affected with the further increase of WSA, i.e. 47.08 MPa [29,30,67,92]. This is essentially contributed to the larger amounts of the unhydrated or unreacted cement particles. This is consistent with many previous studies [93-95]. Therefore, the optimum content of WSA is 25 wt. %, and the higher addition of WSA > 25 wt. % must be prevented.

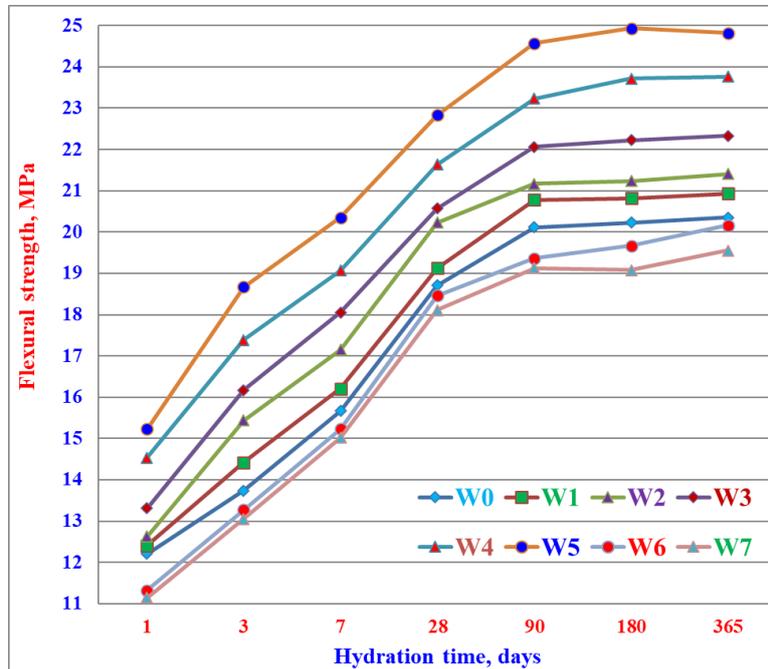


Fig. 9 Flexural strength of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

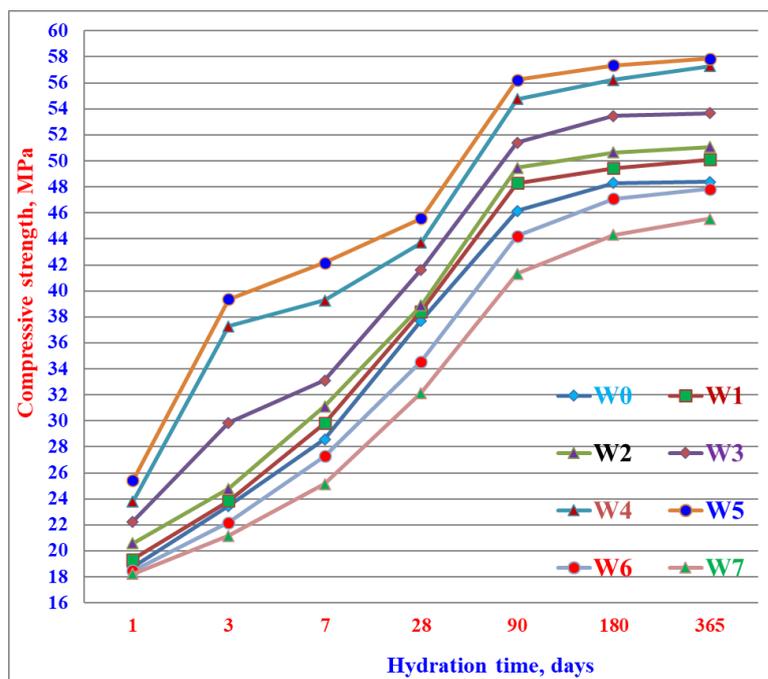


Fig. 10 Compressive strength of the various cement pastes blended with different ratios of WSA and hydrated up to 365 days

4. Conclusion

Walnut shell ash (WSA) has been successfully utilized as a new supplementary cementitious material (SCM). The use of WSA led to increase of W/C-ratio, the elongation of setting time and the reduction of the fluidity of cement paste due to the inactive WSA and its irregular surface. The substitution of WSA for OPC must be ≤ 25 wt. %, beyond which both W/C-ratio, setting time and fluidity are undesirable. The free lime content of the OPC (W0) increased due to the hydration of the di- and trisilicate phases of the cement, but decreased with the addition of WSA due to the pozzolanic action of WSA. The total porosity gradually decreased whereas the bulk density enhanced, but only up to 25 wt. % WSA (W5). With any further addition more than this ratio the total porosity increased, while the bulk density decreased. The use of WSA > 25 wt. % to replace OPC resulted in lower later mechanical properties, where the addition of 25 wt. % WSA exhibits the optimum flexural and compressive strength results. The strength enhancement originated from the high rate of hydration, the low porosity, the reduction of free lime content and the formation of additional fibrous CSH gel in cement paste with 25 wt. % WSA. The obtained results confirmed that the WSA is a qualified SCM. The partial substitution of WSA for cement up to 25 wt. % has a great potential to help in mechanical performance enhancement and good densification of cement pastes.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The author confirms sole responsibility for the following: study conception and design, data collection, analysis and interpretation of results, and manuscript preparation.

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