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Cementitious Composite Containing Diatomaceous Earth Waste as a Pozzolanic Component

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

Results showed that diatomaceous earth waste (DEW) which is rich in SiO_2, has a highly porous surface and contains more amorphous phases. DEW consists of a mixture of quartz sand and DEW particles with a high specific surface area (8755 cm^2/g) and a lower apparent specific mass compared to cement, and moreover uniform particle sizes distribution. Ratio of cement/water (c/w) and setting time increased continuously with the increase of DEW addition. Water absorption and apparent porosity reduced as the addition of DEW increased, while bulk density improved and enhanced, but only up to 24 wt. % DEW, and above this it was adversely affected. Flexural and compressive strengths also improved and enhanced with enhancing DEW addition also up to 24 wt. % and then adversely affected with more addition of DEW. Therefore, the optimum addition of DEW is 24 wt. %. Portlandite or free lime test indicated that DEW showed and exhibited a strong pozzolanic reactivity due to its high content of amorphous silica. So, it could be used in the production of pozzolanic cements, mortars, and/or concretes.

1. Introduction

Recently, the environmental impact of construction sector has become a growing concern due to its high CO_2 emissions [1]. Not only the supplementary cementitious materials (SCMs) reduce the carbon emission, but it also contributed to the sustainable management of industrial wastes [2,3]. However, the availability of traditional SCMs as granulated blast furnace slag (GbfS), fly ash (FA), silica fume (SF), ceramic sludge (CS), etc. is increasingly limited in many countries. The challenge for meeting the high demand of these wastes is to utilize in the production of cement and/or concrete [4-8]. This needs to search and explore new SCMs. A potential solution is to use the diatomaceous earth (DE) as SCMs [9]. The DE created from the fossilized remains of unicellular aquatic plants (diatoms) that consisted of sedimentary rocks due to biogenic origin. Its excellent physical and chemical properties like high porosity, high specific surface area, and low density which enhanced its value in various industries [9,10-14]. Both high concentration of amorphous silica and high specific surface area (1900 cm^2/g) of the DE particles promoted its reactivity to form new hydrated compounds as calcium silicate hydrate (C-S-H) [2,9]. These properties make DE to behave as a natural pozzolan [15-18]. The DE is highly valuable to produce cement-based composites [19]. Approximately 2.2 million tons of DE were produced worldwide as in [20-26]. Most of the produced DE is of a poor quality due to its lower SiO_2 content and presence of impurities [27]. This is mainly due to the turbulent nature of the aquatic environment, where the pores of the diatomaceous earth are filled with the surrounding impurities (organic matter, sand, clay, etc.) [28]. As a result, large quantities of diatomaceous waste are produced, which are usually dumped in open spaces, causing serious environmental, social, and economic problems related to the waste of natural resources and the lack of space to absorb the large quantities of waste produced [29]. Ferreira et al. 2024 [9] demonstrated that the use of DE in construction materials could be used as a cement substitute. Only the low-grade DE with less SiO_2 was used. The lack of research on the properties of DE waste has led to its underutilization in construction, resulting in the waste of valuable resources.

The study is essential not only for filling this research gap, but also for encouraging the use of this waste because it often has no other field to be applied. The main objective of the current article is to evaluate physical, chemical and mineralogical properties, as well as the pozzolanic activity of DE waste. This approach could promote environmental sustainability by preventing the improper disposal of DE waste and offer an innovative solution to the scarcity of natural resources.

2. Materials and Methods

The used raw materials are Ordinary Portland cement (OPC) Type I- CEM I 42,5R, pulverized fly ash (PFA), Granulated blast furnace slag (GbfS) and diatomaceous earth waste (DEW), respectively. Samples of OPC and pulverized flay ash (PFa) were provided by the new cement factory, Sohag, Egypt, GbfS sample was obtained from Iron and Steel Company, Helwan, Cairo, Egypt. DEW Sample was supplied by Agricultural Research Center (ARC), Giza, Egypt. Blaine surface areas of the four raw materials were 3564, 5685, 4385, and 8755 cm^2/g, respectively (Fig. 1). The high fineness diatomaceous earth waste (DEW) sample with beige color containing sand and an organic matter having particle size distribution 73 µm was obtained from a local company, Cairo. Manufacture of DEW passed through several steps as extraction, calcination, grinding, classification, and, dispatch. Extraction of DEW occurs in an open-pit mining, either in ponds or waterways [30-31]. Once the material is formed, the product is mainly DEW. Calcination process occurs in kilns up to 1000 °C for 24 hrs. Finally, it turned and processed to big bags, which could be sold to the beverage and paint industries. These samples were then dried in an oven at 105-110 °C for 48 hrs and then stored in plastic containers till using. DEW sample was quartering till the required amount of material was obtained. Mineral constituents of OPC sample are shown in Table 1. Physical properties of the raw materials are illustrated in Table 2, whereas the oxide composition of these four materials were determined by semi-quantitative X-ray fluorescence (XRF) spectrometry, is shown in Table 3, and then recalculated considering the loss on ignition (LOI) at 1000 °C. Electrical conductivity for the used four raw materials was conducted to explore its fineness and pozzolanic activity. Also, particle size distribution of these raw materials is illustrated in Fig. 2, where DEW is much finer than those of the other two materials (PFa and GbfS), while PFa is finer than GbfS.

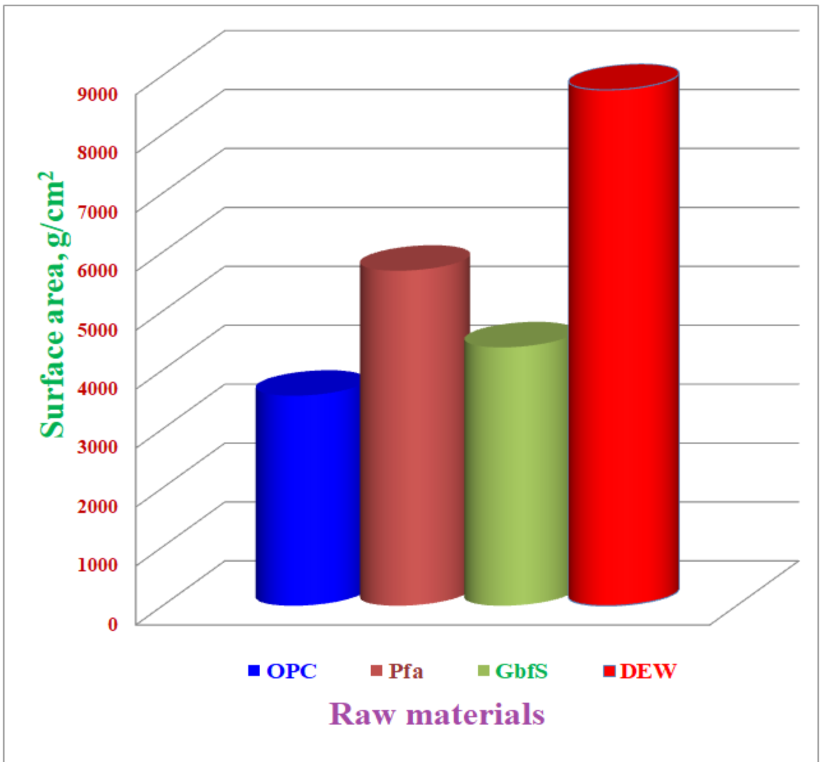

Fig. 1 *Surface area of raw materials*

Table 1 *Phase constituents of Cement specimen, %*

Phase / Material	C_3S	β-C_2S	C_3A	C_4AF
OPC	49.47	29.19	4.83	11.85

Table 2 *Physical properties of raw materials, %*

Properties / Materials	Spec. gravity	Density, g/cm^3	Fineness, cm^2/g
OPC	3.15	3.12	3564
PFa	2.66	2.87	5683
GbfS	2.87	2.65	4385
DEW	2.79	2.59	8753

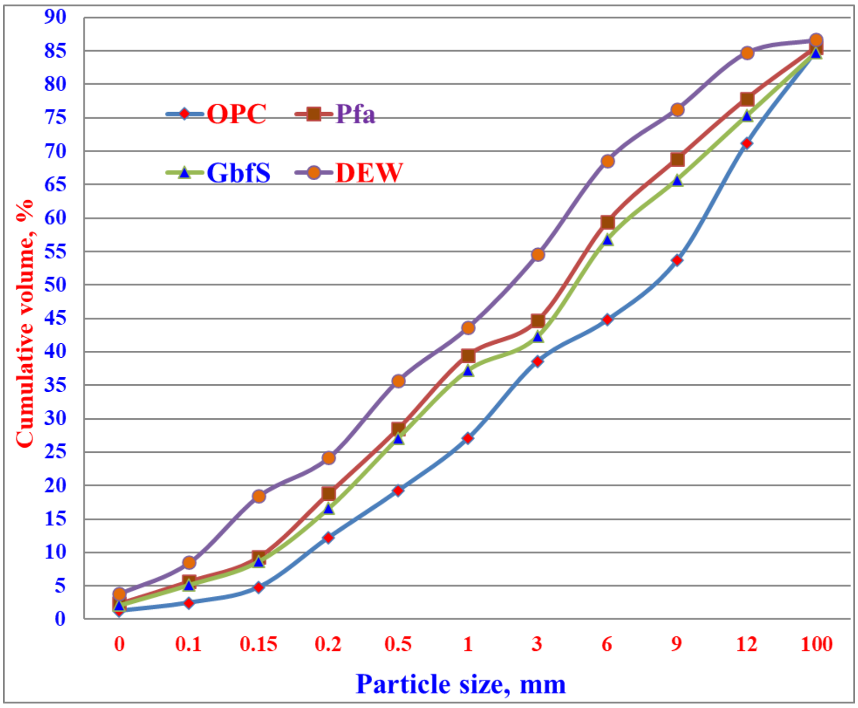

Fig. 2 *Particle size distribution of the used raw materials, wt. %*

Table 3 *Oxide contents, wt. % of raw materials using by XRF technique*

Oxides / Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	ZrO_2	TiO_2	LOI	Total
OPC	21.49	5.89	2.97	63.62	2.01	1.23	0.68	2.06	-----	0.03	1.76	100
PFa	51.12	31.31	5.84	4.83	1.38	0.68	1.17	0.16	0.02	0.01	1.48	100
GbfS	42.73	10.67	1.49	30.46	2.23	1.47	0.67	2.68	0.01	-----	0.51	100
DEW	82.18	7.69	1.31	3.65	-----	1.68	0.37	0.12	0.01	0.15	2.84	100

Table 4 *Physical properties of raw materials, %*

Mixtures / Materials	D0	D1	D2	D3	D4	D5	D6	D7
OPC	94	91	88	85	82	79	76	73
PFa	3	3	3	3	3	3	3	3
GbfS	3	3	3	3	3	3	3	3
DEW	0	3	6	9	12	15	18	21

The base batch is composed from OPC, PFa and GbfS by the ratios of 94:3:3, respectively. During the preparation of cement mixtures, different dosages of DEW (0, 4, 8, 12, 16, 20, 24 and 28 wt. %) were added to the base batch. These mixtures were categorized into 8 groups as D0, D1, D2, D3, D4, D5, D6 and D7, respectively. Mixing of the various cement blends has done in a porcelain ball mill containing 3-5 balls for one hour to assure the complete homogeneity of blends. Firstly, the w/c-ratio and setting time are measured using Vicat apparatus [33,34]. Pastes of the various blends were prepared with the determined w/c-ratio, i.e. the right w/c-ratio was added to the cement powder in the mixer. Then, the mixer was run for about 5 minutes at an average speed of 10 rpm to have a perfect homogenous mixture. Cement pastes were then molded into one inch cubic stainless steel molds (2.5 cm^3) using about 500 g cement mix, vibrated manually for two min. and then on a mechanical vibrator for another two min. [33]. The surface of the molds was smoothed using a suitable spatula. The molds were kept in a humidity cabinet for 24 hrs. at 95±2 RH and room temperature 20 ± 1 °C. In the following day, the molds were demolded and the samples were immediately placed in water till the time of testing for water absorption, bulk density and total porosity [35,35].

Flexural strength (FS) was calculated [37,38], whereas the samples were marked at three points adjusting to place them on the correct point of contact (Fig. 3). The compressive strength of the different cement pastes also was measured [39,40].

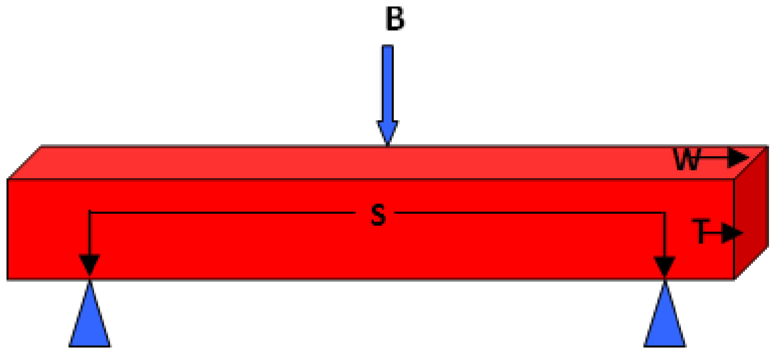

Fig. 3 *Schematic diagram of bending strength, B: Beam or loading of rupture; S: Span; W: Width and T: Thickness*

Portlandite or free lime content (FLn) of the hydrated specimens pre-dried at 105°C for 24h was also measured [32,39-44]. Heat of hydration has been studied to assure the obtained results [45-49]. Degree of cement hydration was explored the influence of DEW on the long-term strength of cement pastes. The X-ray fluorescence (XRF) was analyzed the chemical and phase composition of OPC and DEW, identifying crystalline phases of OPC cement.

3. Results and Discussion

Electrical conductivity (EC) of raw materials was conducted to follow its pozzolanic activity are represented in Fig. 4. Pure water was used because it is free from any salts and/or contaminants. Water heating temperature was based on the initial temperature observed during the exothermic reaction of Portland hydration [50]. Electrical conductivity of a saturated solution of $Ca(OH)_2$ at 40 °C and DEW material in two minutes was proposed [51,52]. Electrical conductivity of DEW is higher than those of PFa and GbfS and PFa is higher than that of GbfS. This test indicated that DEW has a pozzolanic reactivity with the formed portlandite due to hydration more than PFa and/or GbfS. On the side, the Pfa has pozzolanic action more than that of GbfS [51-53]. But, OPC sample is nonpozzolanic material. This helped to modify and improve the characteristics of the cement.

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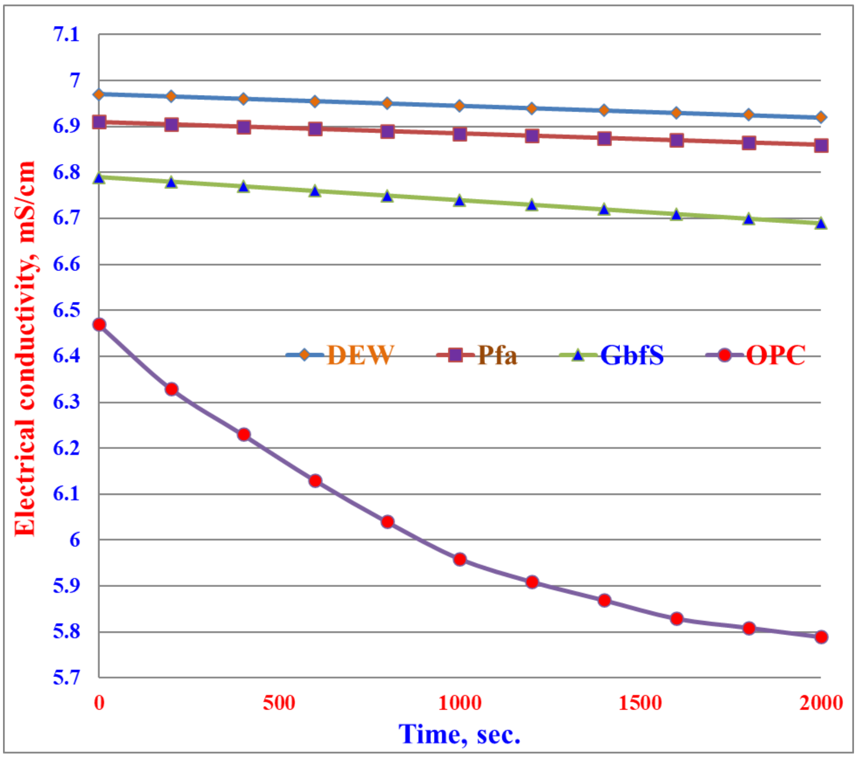

Fig. 4 *Electrical conductivity of OPC, PFa, GbfS anf DEW materials*

Water of consistency (WC) and setting times (ST) of OPC composite (D0) blended with various percentages of DEW (D1-D7) are shown in Fig. 5. It is clear that WC of the control (D0) was 31.56 %. This value was increased as the content of DEW enhanced. Also, both Initial and final setting times of the blank (D0) were 145 and 171 min, respectively. These values were enhanced as the DEW addition enhanced. It is mainly contributed to the high fineness of DEW [54-57]. Furthermore, its pozzolanicity with the produced portlandite due to hydration of OPC, which is in need to more water to occur improved its physical and chemical characteristics [58-61].

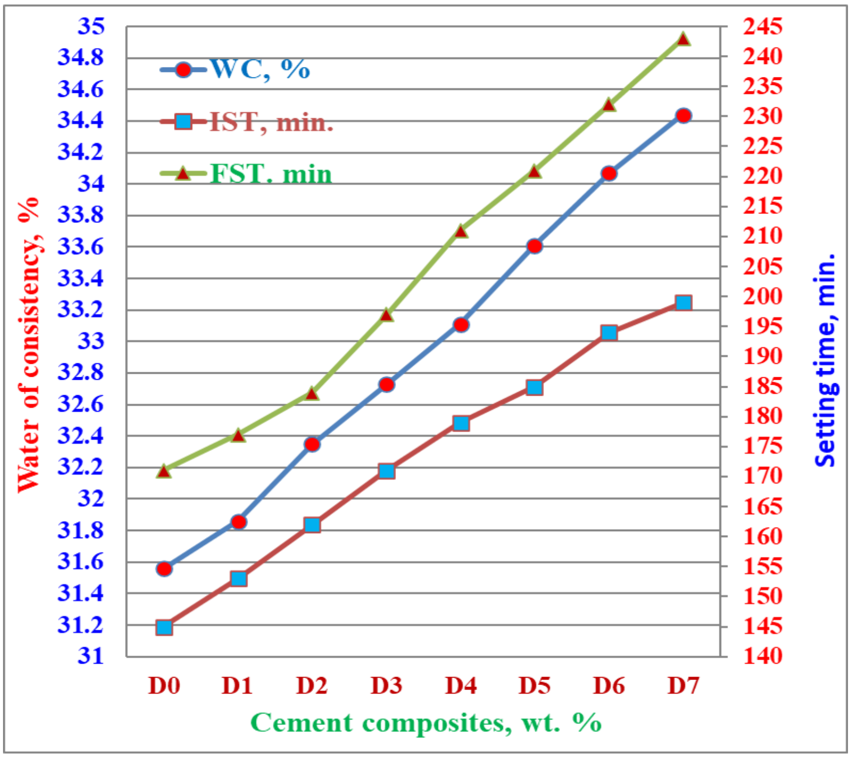

Fig. 5 *Water of consistency and setting times of cementitious composites containing DEW waste material*

Water absorption (WA) of a cementitious composite blended with various contents of DEW hydrated up to 90 days is represented in Fig. 6. Results illustrated that the WA of control (C0) decreased all over hydration times. It is surely attributed to the mechanism of hydration of cement phases [62-64]. As DEW content increased, the WA decreased. This firstly may be attributed to the increased compaction of the hardened cement pastes due to the decreased pore volume. Furthermore, the pozzolanic property of DEW with the producing portlandite that was coming from the hydration of silicates phases of the cement help to decrease the WA [65-67]. This was continued till 18 wt. % DEW (D6). But, with any further DEW addition, the water absorption tended to increase (D7). This is essentially contributed to that the higher content of DEW at the expense of the main binding material negatively reflected on the WA results [68-72]. Accordingly, the higher content of the additive material must be refused.

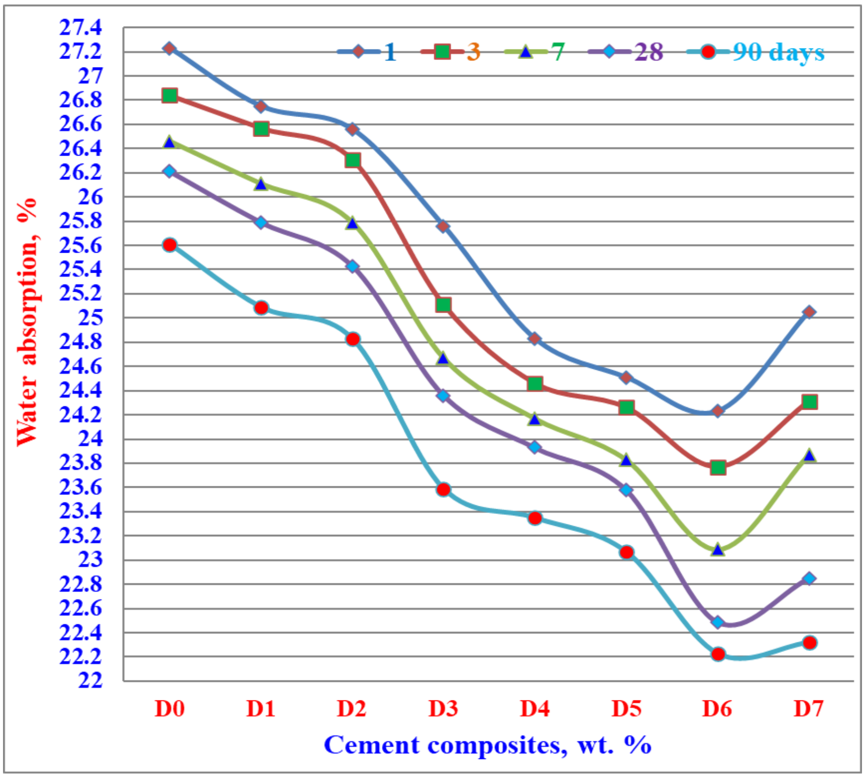

Fig. 6 *Water absorption of cementitious composite containing DEW hydrated up to 90 days*

Fig. 7 shows the bulk density (BD) of a cementitious composite consists of OPC, PFa and GbfS (D0) mixed with various ratios of DEW (D1-D7) hydrated to 1, 7, 28 and 90 days. Results indicated that the BD of all cement composites increased with both curing times and DEW addition [60,66,70]. This is mainly due to the normal hydration of cement phases and also the pozzolanic effect of the three pozzolanic materials (PFa, GbfS and DEW). This continued till the cement composite (D6) containing 24 wt. %. But, with any increased addition of DEA › 24 wt. %, the BD tended to decrease. The increase of BD is due to the high pozzolanic action of DEW besides the good compaction of the hardened cement composites due to the precipitation of the unhydrated particles of the three waste materials (PFa, GbfS and DEW) inside the pore volume of the specimens, i.e. DEW acted as filler [60,71]. As a result, the apparent porosity seemed to decrease [61,64,66,72]. Consequently, the BD improved and enhanced. The decrease of BD is attributed to that the higher contents of DEW hindered the hydration as well as the pozzolanic effect of DEW, or even ceased it. Accordingly, the high quantities of DEW must be prevented.

Apparent porosity (AP) of a cementitious composite from OPC, PFa and DEW (D0) mixed with various proportions of DEW (D1-D7) hydrated till 90 days are represented in Fig. 8. It is clearly that the AP of all cement composites gradually decreased, with the hydration ages till 90 days. Moreover, it also lowered with the increase of DEW addition, but this continued merely up to 24 wt. % (D6). Then, it enhanced with any other CSA addition. Decreasing of AP is mainly due to the pozzolanic properties of DEW with the produced portlandite from the hydration of C_3S and C_2S phases of the OPC cement [59,71,73]. But, the increased values of AP are mainly contributed to stopping of hydration that was resulting from the incorporation of larger amounts of DEW (D7), which decreased not only the normal hydration process, but also the pozzolanic activity of DEW [70-73]. As a result, the optimum addition of DEW is 24 wt. %. Furthermore, the higher quantities of DEW must be refused due to its adverse effect. Results of AP are in a good agreement with those of BD and WA to a great extent.

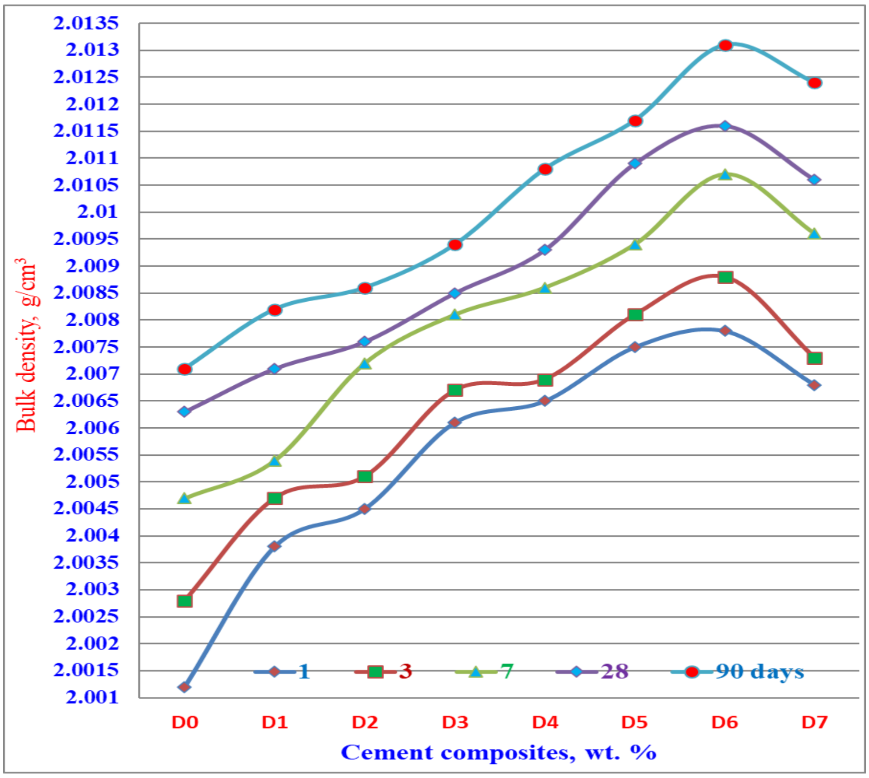

Fig. 7 *Bulk density of cementitious composite containing DEW hydrated up to 90 days*

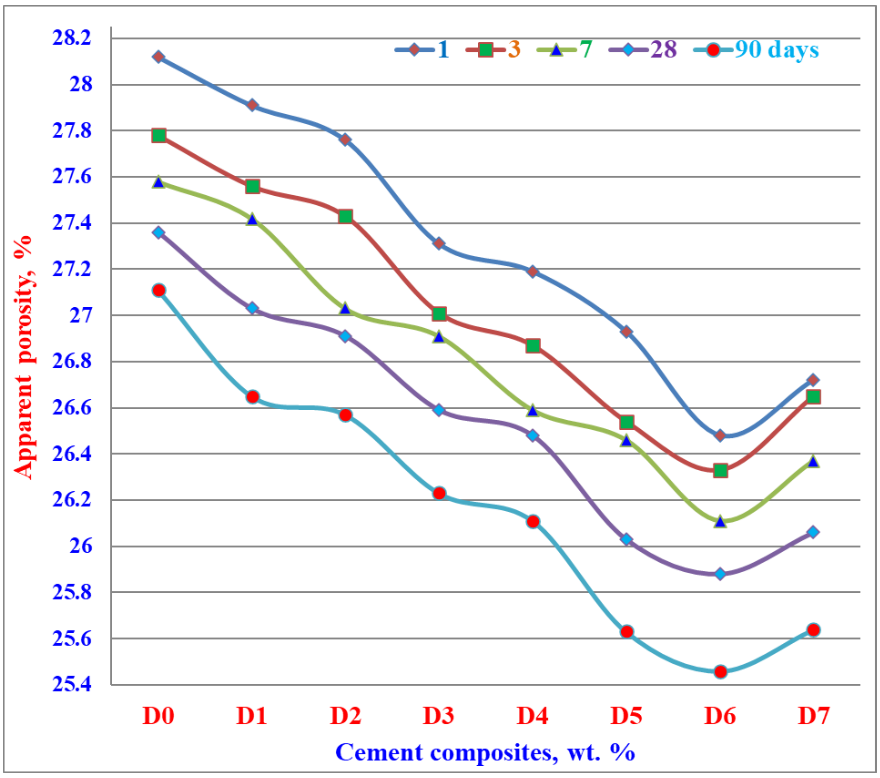

Fig. 8 *Apparent porosity of cementitious composite containing DEW hydrated up to 90 days*

Flexural strength (FS) of a cementitious composite from OPC, PFa and GbfS (D0) mixed with various ratios of DEW (D1-D7) hydrated till 90 days are demonstrated in Fig. 9. As shown from, the FS gradually increased as the hydration ages increased till 90 days. The increase of FS is mainly due to the newly formed CSH products as a result of hydration, which improved FS of hardened cement pastes [59,71]. The FS was further improved and enhanced by the addition of DEW till 24 wt. % (D6), and then negatively influenced by any other addition of DEW (D7). Increased FS results with DEW addition are often attributed to its pozzolanic reactivity of DEW with the formed portlandite due to the hydration of di- and tricalcium silicate phases (C_3S and β-C_2S) of the cement. Furthermore, unreacted PFa, GbfS and even DEW helped to act as a filler that closed the pore structure of samples, i.e. the AP decreased and the BD increased. Therefore, the FS improved and enhanced [63,71,73]. The adverse effect with the higher addition of DEW (D7) is principally due to that the high quantities of the additive material hindered and may be ceased the hydration of cement phases and helped to open the pore structure to a large extent [60,63,70,72]. Therefore, the optimum addition of DEW is 24 wt. %, whereas the higher amounts of the DEW must be removed.

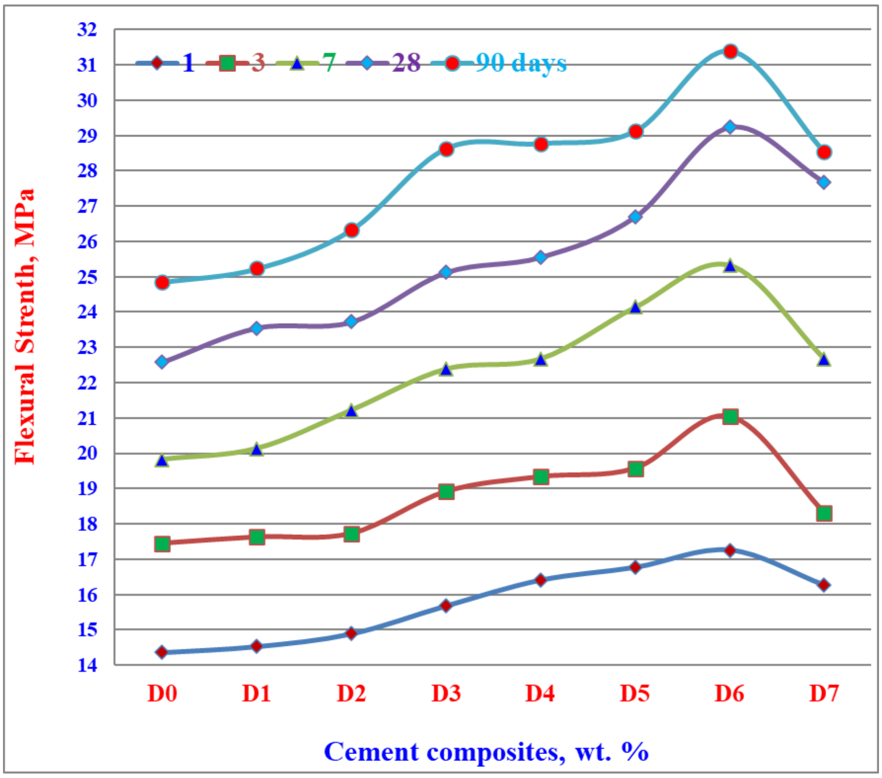

Fig. 9 *Flexural strength of cementitious composite containing DEW hydrated up to 90 days*

Compressive strength (CS) of a cementitious composite incorporated OPC, PFa and GbfS (D0) mixed with different proportions of DEW byproduct (D1-D7) hydrated up to 90 days is illustrated in Fig, 10. CS of all hardened cement composites enhanced with the hydration age till 90 days. It is mainly due to the newly resulting CSH, which are precipitated into the pore volume, i.e. this decreased the AP and increased the BD. So, this could be inverted well on the CS of the cement composites [58,60,63,72]. Also, crystal growth of the formed CSH was gradually improved and enhanced. The improvements of CS often enhanced with DEW addition, but merely till 24 wt. % (D6), and then adversely affected by any other addition of DEW (D7). Improved CS values with DEW addition are often due to the pozzolanic reactivity of DEW with the resulting portlandite from the normal hydration of di- and tricalcium silicates (β-C_2S and C_3S) of OPC cement, Furthermore, the unreacted PFa, GbfS and DEW improved the compaction of the constituents together, i.e. the pore structure was partially or completely closed, i.e. unreacted particles of the wastes were acted as a filler [74-76]. The adverse results with the higher content of DEW (D7) were principally contributed to that the higher addition of these materials were largely stopped the hydration of cement phases. This helped to open more-pore structure to a large extent [58,60,67,70,72]. Therefore, the optimum content of DEW is 24 wt. % and the higher amounts of DEW must be illuminated.

Portlandite or free lime contents (Fln) of a cementitious composite containing OPC, PFa and DEW wastes (D0) blended with various ratios of DEW (D1-D7) hydrated till 90 days are shown in Fig, 11. PC of the blank (D0) was enhanced little by little as the hydration period was going on till 7 days. It is essentially contributed to the hydration of the main silicate phases of the cement (C_3S and β-C_2S) and other phases [58,60,70,71,73]. But after 7 days, PC stated to decrease onwards due to the pozzolanicity of both PFa and GbfS [62,63,80]. On the other side, PC of other cement composites incorporated different ratios of DEW only increased till 3 days of hydration, but then it began to gradually decrease onwards. It is fundamentally contributed to the strong pozzolanic phenomenon of DEW, which could be occurred among the resulting PC and DEW particles [71,73-77].

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Consumption of PC by its reaction with the constituents of DEW led to the formation of extra C-S-H gel-phases. These hydration products were passed and precipitated into the pore system of hard cement composites samples. This in turn improved and supported physical, chemical and mechanical properties. This was contributed to the improvements of the densification parameters due to the reduction of voids, porosity, and water permeability [58,61,64,78]. PC of cement composites incorporated DEW › 24 wt. % (D7) began to little increase or be stable. It is due to fact that the high addition of DEW at the expense of the OPC hindered its hydration so that the excess of DEW did not find of PC to react with, i.e. the hydration process was completely ceased.

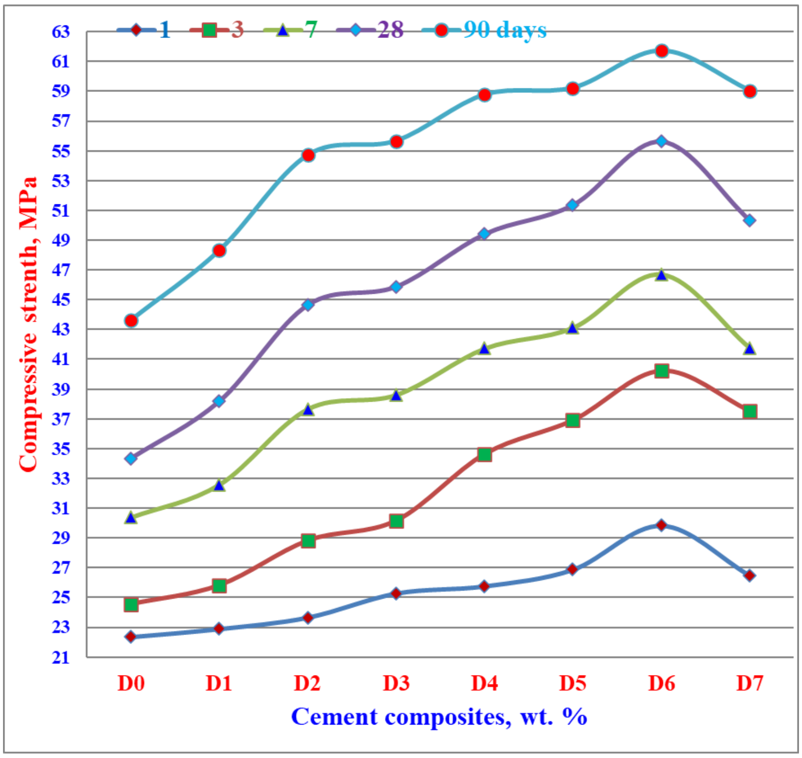

Fig. 10 *Compressive strength of cementitious composite containing DEW hydrated up to 90 days*

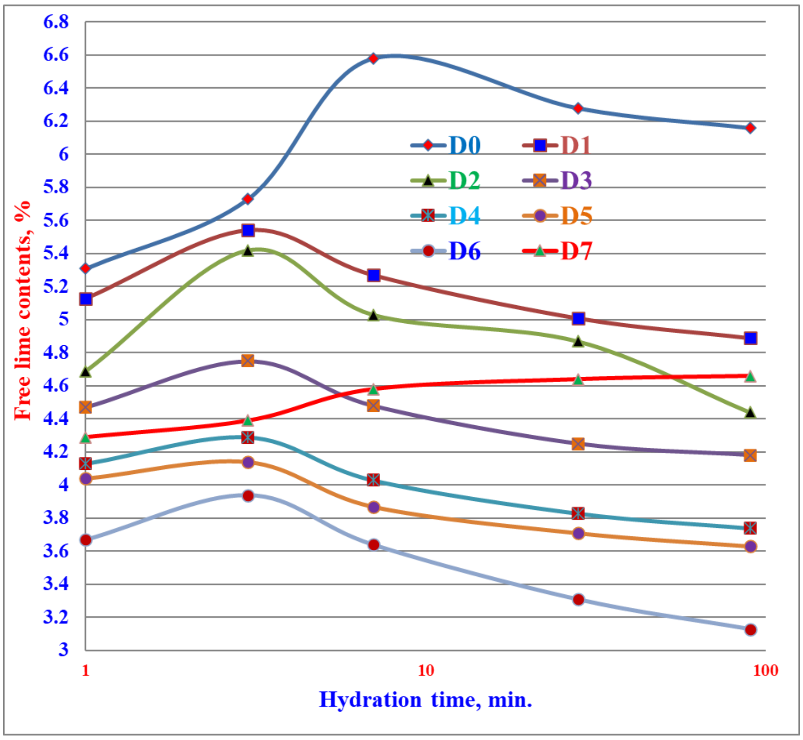

Fig. 11 *Portlandite content of cementitious composite containing DEW hydrated up to 90 days*

Fig. 12 shows the heat of hydration (HH) of a cementitious composite (D0) blended with different ratios of DEW (D1-D7) hydrated till 90 days. When water was poured on cement powder, heat due to hydration starts to generate at once. HH of cement pastes was generally increased with the hydration age till 28 days, but at later ages it was so slightly that it seemed to be stable. The behavior was done by all cement composite samples incorporated different ratios of DEW [70-74]. This is principally due to increasing the rate of hydration of cement phases that was coming from the pozzolanic reactions of DEW with the formed PC. This was followed by a gradual generation of heat [76,77]. The rate of the released HH was greatly enhanced at early stages from 3 to 7 days. It is due to the initiation action on the hydration mechanism of C_3S by the finer particles of DEW. On older stages (28-90 days), the rate of hydration and the released HH increased so little that it seemed to be unchanged or stable. This may be attributed to the reduction of hydration process and/or the non-activation action of C_3S and the slight activation action mechanism of β-C_2S at later ages. Thus, the HH enhanced little as the DEW addition enhanced, but only till 24 % (D6). Increased values of HH are mainly contributed to two hydration mechanisms. The first is the hydration of cement phases and the second is the pozzolanic reactions of DEW with the formed PC from the first process [72-78]. The HH was then unchanged with any further increase of DEW addition (D7). It is principally due to the dilution action of DEW on cementitious compounds and high SiO_2 content of DEW. So, the high DEW content must be prevented due to its adverse action [71-74,78].

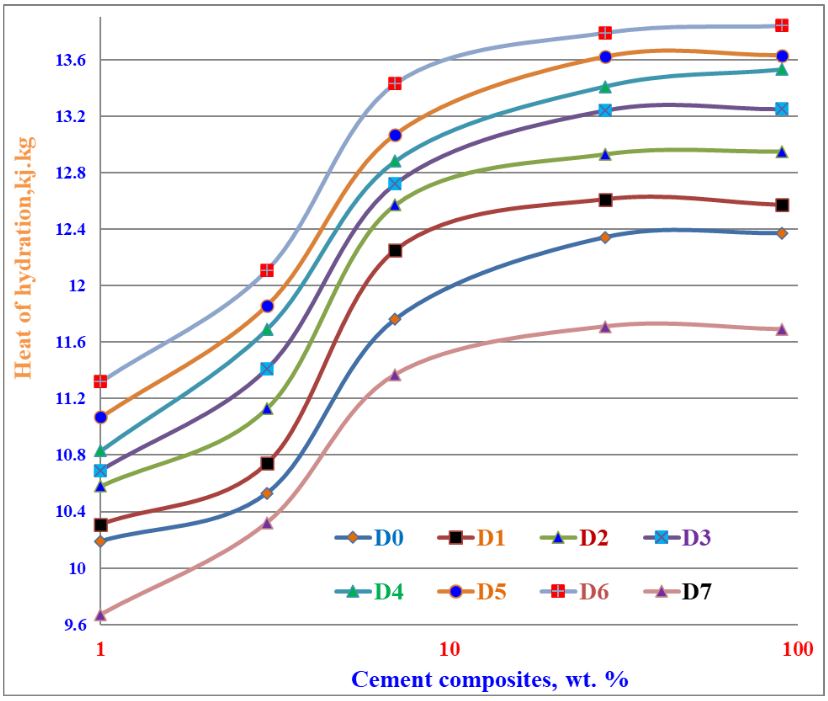

Fig. 12 *Heat of hydration of a cementitious composite containing DEW hydrated up to 90 days*

4. Conclusion

Chemical and physical properties as well as pozzolanic activity of the nanograin sized particles of DEW byproduct blended with cement composite of 94 % OPC and 3 wt. % from both PFa and GbfS were assessed. As DEW contains high ratios of SiO_2 and Al_2O_3, it could be classified as a pozzolanic material. Electrical conductivity of raw materials showed that DEW is the finest, while OPC is the lowest finer. DEW needs more water to form good cement pastes. Both w/c- ratio and setting times (Initial and final) were increased. Free lime in cement composite with 0% DEW due to hydration on 90 days was approximately 6.16 %. This value was reduced with increasing curing time and DEW content. So, DEW is a strong pozzolanic material. But, added DEW › 24 wt%, the CH content starts to increase. Physical properties of cement composites contained DEW byproduct were improved and enhanced with curing time and DEW content, but only up to 24 wt%. Flexural and compressive strengths of the cement composite improved and enhanced with the increase of DEW content till 24 %. Any further increase of DEW content › 24 wt. %, both physical and mechanical properties would be diminished. So, the optimum DEW addition to cement composites without adverse effects on its characteristics is 24 wt. %. Electrical conductivity and free lime tests showed that DEW has the highest pozzolanic reactivity due to its amorphous silica content, which indicates that DEW can react with the evolved Ca $(OH)_2$ from the hydration to produce more C-S–H. Moreover, its high specific surface area enhances its filler effect creating nucleation sites to form additional CSHs. So, DEW could be used as a SCM due to its high pozzolanic character.

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Conflict of Interest

The author declares that there are no financial or competing conflicts of interest in this work.

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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