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EXPERIMENTAL INVESTIGATION ON PROPERTIES OF CEMENT MORTAR INCORPORATING EGGSHELL POWDER

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Abstract: The use of waste materials in buildings is an innovative approach towards sustainability. The objective of this paper is to study the properties and behaviour of cement mortar using eggshells wastes. Various low concentrations of eggshell powder (ESP) (0,1,3 ,5, 7 and 10 wt%) were used as partial replacement of cement in mortar of (1:2) mixing ratio by weight. The maximum particle size of ESP is 75 µm. Soundness of cement in ESP-cement pastes was evaluated using Le Chatelier method. The influence of ESP on strengths of cement mortar was investigated by means of compression and flexural tests at various curing periods. It was found that, there was no negative impact of ESP on soundness of cement. A significant enhancement in compressive strength was recorded for all ESP-cement mortars at 7 days and 28 days. The highest improvements, (31.63%) and (11.65%), were observed for samples with 10wt% and 5 wt% of ESP respectively. At moderate periods of curing, samples with 1 wt% of ESP have the highest enhancements in compressive strength of about (8.93%) and (1.75%) for 42 days and 56 days respectively. A reduction in compressive strength was observed for all samples at late ages (70 days and 90 days). In terms of flexural strength, the addition of ESP (up to 5 wt%) results in improvement in the flexural strength when compared with the control samples. The highest enhancement (21.20%) was recorded when cement was replaced with only 1 wt% at 7 days.

Keywords: *eggshell powder (ESP), cement mortar, waste materials, sustainability.*

التحري العملي لخواص مونة الاسمنت الحاوية على مسحوق قشور البيض

الخلاصة: ان استخدام مواد المخلفات في البناء هو نهج مبتكر نحو الاستدامة.ان الهدف من هذا البحث هو دراسة خواص و سلوك مونة الاسمنت باستخدام مخلفات قشور البيض. استخدمت نسب اضافة قليلة و مختلفة من مسحوق قشور البيض (ESP) (٠ % ، ١ % ، ٣% ، ٥%، ٧% و ١٠% وزنا) كبديل جزئي عن الاسمنت في مونة ذات نسبة خلط وزنية (١:٢). ان الحجم الحبيبي الاقصى لمسحوق قشور البيض كان ٧٥ مايكرون . تم التحري عن ثبات عجينة الاسمنت الحاوية على ESP باستخدام طريقة لي شاتليه. ان تأثير ESP على مقاومة مونة الاسمنت تم التحري عنه بواسطة فحصي الانضغاط و الانثناء و لاعمار انضاج مختلفة. لقد وجد بانه لا يوجد تاثير سلبي ل ESP على ثبات الاسمنت. فيما يخص مقاومة اللانضغاط ، فقد زادت زيادة ملحوظة عند استخدام نسب مختلفة من ESP و لاعمار انضاج ٧ أيام و ٢٨ يوما. أعلى زيادة سجلت كانت بحدود (٣١,٦٣%) و (١١,٦٥%) و لنماذج مونة الاسمنت الحاوية على ٥% و ١٠% وزنا من ESP على التوالي. اما في الفترات المعتدلة من المعالجة ،فقد وجد ان النماذج الحاوية على ١% وزنا من ESP اظهرت أعلى زيادة في مقاومة الانضغاط و كانت حوالي (٨,٩٣%) و (١,٧٥%) و لاعمار انضاج ٤٢ يوما و ٥٦ يوما على التوالي. ولوحظ وجود انخفاض في مقاومة الانضغاط و لجميع العينات في اعمار انضاج متاخرة (٧٠ يوما و ٩٠ يوما). فيما يخص مقاومة الانثناء، فقد وجد بان إضافة ESP (إلى حد ٥% بالوزن) يؤدي الى تحسن مقاومة الانثناء عند مقارنتها مع النماذج المرجعية. وسجلت أعلى زيادة (٢١,٢٠%) عندما تم استبدال الأسمنت فقط مع ١% بالوزن و لعمر انضاج ٧ أيام.

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1. Introduction

Cement is one of the most commonly used building materials with adhesive and cohesive properties[1]. Cement manufacture, however, is resource and energy intensive[2]. The negative impact of the production of cement may fall into two category: *(a)* use of energy (fuel, electricity) and emissions associated with this (CO_2, SO_2 and dust); *(b)* use of natural raw materials (mainly limestone)[3]. In terms of carbon dioxide (CO_2) emission, for every tonne of Portland cement produced approximately one tonne of CO_2 is released into the atmosphere[4]. The global cement industry contributes about 7% of greenhouse gas emission to the earth's atmosphere and among the greenhouse gases; CO_2 contributes about 65% of global warming. It is essential, therefore, to develop alternative binders to make concrete and taking into consideration the environmental effects associated with cement manufacturing.

Utilizing different types of wastes such as: industrial, by products and domestic wastes in cement can significantly contribute towards sustainable development and construction. Because of the environmental and economical reasons, there has been a growing trend in using wastes or by-products as a supplementary material (SM) in the production of the concrete[5]. Due to rising civilization and change in lifestyle and food habits, the amount of solid waste has been increasing rapidly[6]. The accumulation of solid wastes in the plants represents two major problems: solid waste disposal and a negative impact on the environment. Utilization of post-consumer waste materials such as food waste is one of the effective solutions to environmental problems. The key advantages of using such waste materials as alternatives to construction materials are: cost reduction of cement-based materials, cost reduction for landfill, saving in energy, and protecting the environment form possible pollution effects[7].

Accumulating quantities of eggshell wastes are generated in the form of post-consumer commercial and household waste such as poultries, hatcheries, homes and fast food centers[8-9]. Eggshell has a cellulosic structure and contains amino acids; therefore, it is expected to be a good bio-sorbent[10]. It was reported that large amounts of eggshells are produced in many countries as waste products and disposed in landfills annually. The main component of eggshells is calcium carbonate ($CaCO_3$). It is around 95% of the shell while other 5% of the eggshell contains: Magnesium, Aluminum, Phosphorous, Sodium, Potassium, Zinc, Iron, Copper, Ironic acid and Silica acid[11]. Since eggshells wastes (ESW) is a source of $CaCO_3$, many attempts were made by researchers to employ ESW in various applications. These wastes were included in polymeric matrices [12-14], soil as stabilization agent [13-18], lime-based mortar for restoration purposes[19] as well as in the production of ceramic tiles[20].

In terms of concrete production, several studies were carried out to investigate the combined effect of eggshell wastes with other wastes. Shanker and Blaji[21] investigated the suitability to use both eggshell powder (ESP) and fly ash (FA) for partial replacement of sand in concrete. An equal rate of proportion of both FA and ESP was employed. Various percentages of replacements (0%, 7%, 14%, 21% ,28% and 35%) were used of FA and ESP to replace the fine aggregate. The mechanical properties of hardened (FA and ESP concrete) were explored by means of compression, splitting strength and flexural strength tests. It was concluded that FA and ESP could be used in structural concrete.

Sivakumar and Mahendran[22] studied the strength and permeability properties of concrete using Fly ash (FA) and Rice husk ash (RHA) with Synthesis Egg shell powder (ESP) as partial replacement of cement in concrete. In their work, FA and RHA addition were (5%, 10%, 20%, 30%, and 40%) by weight of cement. The addition of ESP was 5% for all mixes. It was found that strength and permeability properties of concrete enhanced by 30% when combined FA (15%), RHA (15%) with additive ESP (5%) was used. In another study[23] compressive and flexural strength of concrete was evaluated using a combination of RHA and ESP as partial replacement of cement. The percentage of ESP was kept the same for all mixes

(5%) while RHA varies (0, 5, 7.5, 10,15 and 20%) by weight of cement. It was reported that the strength of concrete is higher than control concrete for different replacement levels and for various periods of curing (7, 14 and 28 days).

Gowsika *et al* [24] carried out an experimental investigation to study the effect of eggshell powder on 28 days compressive strength of cement mortar in addition to concrete properties (compressive strength, split tensile strength, and flexural strength). It their work, cement mortar of mix proportion 1:3 was used in which cement was partially replaced with eggshell powder. The latter was obtained from industrial wastes and added in various ratios for cement replacement (5%,10%,15%,20%,25% and 30 wt%). A sharp reduction in 28 days compressive strength of cement was recorded when eggshell powder exceeded 5%. In terms of concrete properties, the 5% eggshell powder was used in concrete mixes with the use of various admixtures (saw dust ash, fly ash and micro silica) to enhance the strength of the concrete. It was concluded that replacement of 5% eggshell powder with 20% microsilica will not lead to any reduction in compressive strength properties of conventional samples. Also, the flexural strength of concrete samples that contain 5% eggshell powder with 10% microsilica replacement in cement showed similar flexural strength as in conventional concrete. Higher split tensile strength was conducted for samples with 5% eggshell powder and 10% microsilica replacement in cement when compared with conventional samples.

Another study[25] was carried out to develop concrete production using eggshell powder (ESP). Various concentrations of ESP were utilized to replace 5-15% of cement weight in concrete. The ESP was sieved through 90 micron sieve. The performance of ESP concretes was studied through strength properties like compressive strength and splitting tensile strength and transport properties like water absorption and sorption. Higher compressive strength was conducted for concrete samples with 5% ESP when compared with control samples for 7 and 28 days of curing. ESP replacements greater than 10 % had lower strength than control concrete. The compressive strength of ESP concrete was enhanced with the addition of fly ash. In terms of spilt strength, it was concluded that concrete with 15 % ESP had lower split tensile strength than control concrete.

In the present study, the possibility of using local eggshell wastes in the production of cement mortar without using admixtures was addressed. The effect of adding low percentages of eggshell powder (ESP) (up to 10 wt%) on soundness of cement was investigated. Additionally, compressive and flexural strengths of ESP-cement mortars were explored. The outcomes will provide evidence on the possibility of using alternative supplementary materials that based on local solid wastes. This helps in reduce the negative impact of such wastes on the environment throughout the transformation of wastes into useful materials.

2. Materials

2.1. Cement

Ordinary Portland Cement (OPC) – Type I - is used in this work. The chemical composition of this cement is shown in Table (1). The chemical test was carried out by Iraqi National Center for Construction Laboratories and Researches (NCCLR).

Table 1. Chemical composition of cement

Oxide composition	Abbreviation	Content (%)
Lime	CaO	64.25
Silica	SiO_2	19.35

Alumina	Al_2O_3	4.61
Iron oxide	Fe_2O_3	3.19
Sulfate	SO_3	2.85
Magnesia	MgO	2.03
Loss On Ignation	L.O.I	4.00
Insoluble Redsidue	I.R	1.50
Lime Saturation Factor	L.S.F	1.01
Tricalcium Aluminates	C_3A	6.83

2.2. Fine Aggregate

Al-Ekhaider natural sand is used in this study as fine aggregate of 4.75 mm maximum size. Table (2) shows the grading of this sand which meets the requirements of Iraqi Specification No.45/1984, zone 3[26].

Table 2. Grading of fine aggregate

Sieve size (mm)	Passing (%)	Limits of Iraqi Specifications No. 45/1984 , zone 3
4.75	99.21	90-100
2.36	89.65	85-100
1.18	76.99	75-100
0.6	61.58	60-79
0.3	17.28	12-40
0.15	8.99	0-10

2.3. Eggshell Powder

The eggshells (ES) were collected from domestic sources. The ES was washed manually and then left to dry at ambient conditions. In order to satisfy the physical requirement for fineness, ES was ground fine enough to pass through a 75 µm sieve. This is accomplished by crushing and grinding the eggshells manually and then sieving the ground ESP to the desired particle size. The chemical compositions of ESP are shown in Table (3)[27].

Table 3. Chemical analysis of ESP[27]

CaO	50.70
SiO_2	0.09
Al_2O_3	0.03
Fe_2O_3	0.02
SO_3	0.57
MgO	0.01
Na_2O	0.19
P_2O_5	0.24
NiO	0.0001
SrO	0.13
Cl	0.219

2.4. Water

Tap water is used in this work for all tests.

3. Preparation of ESP- Cement Pastes

A total of six cement pastes with and without ESP were prepared to be used for soundness test. Two of them are control paste (without ESP) and others containing ESP with various concentrations (1,3,5,7 and 10wt%). The (water/cement) ratio was kept constant for all mixes for comparison purposes. This ratio is ٠.4 which was conducted from the standard consistency test of cement. The latter was carried out in accordance to ASTM C187 [28].

4. Fabrication of ESP-Cement Mortar Specimens

In total, one hundred and sixty two (162) specimens of cement mortar were constructed. One hundred and eight (108) of these samples were for compression test of 50×50×50 mm dimensions. Other fifty four (54) specimens were for flexural test of 40×40×160 mm dimensions. The mixing procedure involved manual mixing of cement, sand and water. The (cement: sand) ratio was (1:2) by weight and (water/cement) ratio was 0.4 by weight which was used for all mortars (with and without ESP). Various concentrations of ESP (0,1, 3,5,7 and 10 wt%) were used for partial replacement of cement. For each percentage of ESP, three specimens were cast.

The procedure of casting cubic and prismatic cement mortars was carried out in accordance to ASTM C109[29] and ASTM C348[30] respectively. Prior to casting, moulds were cleaned and lubricated with a thin layer of oil to facilitate demoulding procedure. Moulds were filled with two layers of cement mortar (with and without ESP). Each layer was about (25 mm) and (20 mm) for compression and flexural samples respectively. Each layer was compacted manually for 32 times and 12 stocks for cubes and prisms respectively. After 24 hours at ambient conditions, specimens were then demoulded carefully and marked prior to placing in curing tanks for different ages namely 7, 28, 42, 56, 70 and 90 days for compression specimens; and for 7, 28 and 56 days for flexural specimens. For each age of curing, three specimens were used.

5. Tests

5.1. Soundness of cement incorporated ESP

Various compounds in cement could cause unsoundness in cement; namely free CaO, free MgO and calcium sulphate[1]. In the present study, Le Chatelier method was adopted to detect unsoundness of cement. This method is more sensitive to detect the unsoundness that caused by free CaO. This method is an accelerated test and it was carried out in accordance to BS EN 196-3[31].

5.2. Strength of ESP-Cement Mortar

5.2.1. Compression Strength Test

In total, one hundred and eight of cubic cement mortars were tested in compression. The samples were placed between bearing plates of universal testing machine (Figure 1). The load was applied gradually with a displacement rate of 3.0 mm/min, using a load cell capacity of 300 kN until failure.

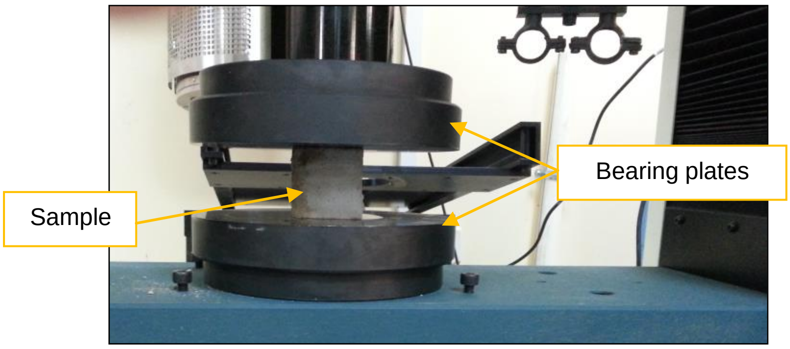

Figure 1. Compression test arrangements of cement mortar

5.2.2. Flexural Strength Test

A total of fifty-four prismatic cement mortars were tested in flexural. Its arrangements are shown in Figure (2 left). A prism was placed in testing machine with one side on the supporting rollers and its longitudinal axis normal to the supports (Figure 2 right). The load was applied vertically by means of loading roller to the opposite side face of the prism. The application of load was carried out with a constant displacement of 1.5 mm/ min, using a load cell capacity of 100 kN until failure. The flexural strength was calculated using the following equation[32]:

$$R_f \quad \frac{1.5 \times F_f \times L}{b^3} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where:

R_f : flexural strength, (MPa);
b: side of the square section of the prism, (mm);
F_f : load applied to the middle of the prism at fracture, (N);
L : distance between the supports, (mm).

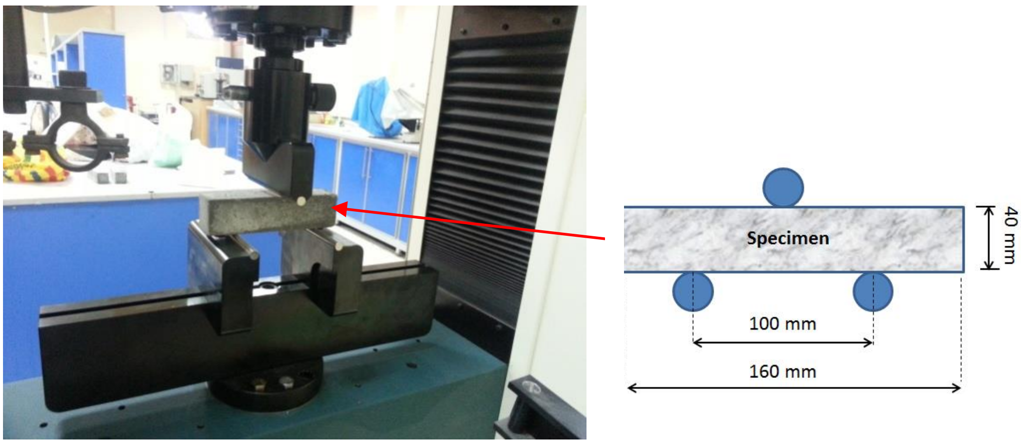

Figure 2. Flexural test arrangements of cement mortar

6. Results and Discussion

6.1. Effect of ESP on Soundness

The obtained results from soundness test are listed in Table (4). These results present an average of two cement pastes with various ESP concentrations. No change in volume expansion of cement pastes was achieved with the inclusion of various ESP in cement when compared with control samples (without ESP). The recorded expansion (0.5 mm) was the same for all mix. This means that the ESP has no influence in causing significant variations in volume of cement paste after it has fixed itself firmly.

Table 4. Results of soundness test of ESP-cement pastes

ESP (wt %)	No. of tested samples	Expansion (mm)
0	2	0.5
1	2	0.5
3	2	0.5
5	2	0.5
7	2	0.5
10	2	0.5

6.2 Effect of ESP on Compressive Strength

The results of compression test are listed in Table (5). Each value presents an average of testing three cubic samples. An increment in compressive strength was observed as the age of curing increases for all samples (with and without ESP) due to increase in hydration process with time which leads to increase the hydration products[1]. The replacement of cement with ESP leads to a significant enhancement in the compressive strength for specific concentrations of ESP and for particular ages of curing. As illustrated in Figure(3), enhancement in compressive strength was achieved with the inclusion of various percentages of ESP and for curing ages up to 56 days when compared with control samples (without ESP). The strength at early age (7 days) increases with the increase in ESP. As shown in Table (6), the highest enhancement in the strength (31.63%) is recorded for cement mortar with the highest ESP concentration (10 wt%). This may attributed to the role of calcium carbonate ($CaCO_3$) (the main component of ESP) on accelerating the hydration of C_3S of cement[32-35]. Additionally, the $CaCO_3$ from ESP reacts with alumina phase of the cement to produce carboaluminates. The formation of the latter prevents or postpones the transformation of ettringite to sulphoaluminates. This will leads to an increment in the total volume of the hydration products[36].

As the hydration of cement increases (up to 28 days), the strength increases with the incorporation of very low concentration of ESP (1, 3 and 5 wt%). The highest value (11.65%) was achieved with samples of 5 wt% of ESP. In terms of moderate curing ages (42 days and 56 days), the strength developed significantly with the addition of ESP concentrations up to 5 wt% for 42 days and the highest was detected for samples with 1 wt% of ESP compared with control mortar. The 1 wt % of ESP seems to be the only ESP % which positively enhanced the strength at 56 days and further increment in ESP (beyond 1 wt%) reduces the compressive strength. Regarding strengths at late ages (70 days and 90 days), a reduction was observed with the addition of ESP and the lowest strengths (-16.52%) and (-24.59%) were obtained for samples with highest ESP for 70 days and 90 days respectively (Table 6). This may related to the diluent effect[37] of unreacted $CaCO_3$ from ESP.

Table 5. Results of compression test of ESP- cement mortars at different curing ages

ESP (wt %)	Compressive Strength (MPa)					
	7 days	*28 days*	*42 days*	*56 days*	*70 days*	*90 days*
0	26.97	35.38	41.42	46.88	48.60	59.04
1	30.85	36.92	45.12	47.70	47.89	59.01
3	32.39	36.80	44.95	46.03	47.00	56.43
5	34.53	39.50	42.83	45.63	47.47	50.17
7	34.08	35.98	38.57	42.60	45.50	48.10
10	35.50	36.16	38.67	39.93	40.57	44.53

Table 6. Variations of compressive strength of ESP- cement mortars at different curing ages

ESP (wt %)	Variations of compressive strength (%)					
	7 days	*28 days*	*42 days*	*56 days*	*70 days*	*90 days*
0	___	___	___	___	___	___
1	14.39	4.35	8.93	1.75	- 1.46	- 0.06
3	20.10	4.01	8.52	- 1.81	- 3.29	- 4.43
5	28.03	11.65	3.40	- 2.67	- 2.33	- 15.04
7	26.36	1.70	- 6.88	- 9.13	- 6.38	- 18.54
10	31.63	2.20	- 6.63	-14.82	-16.52	- 24.59

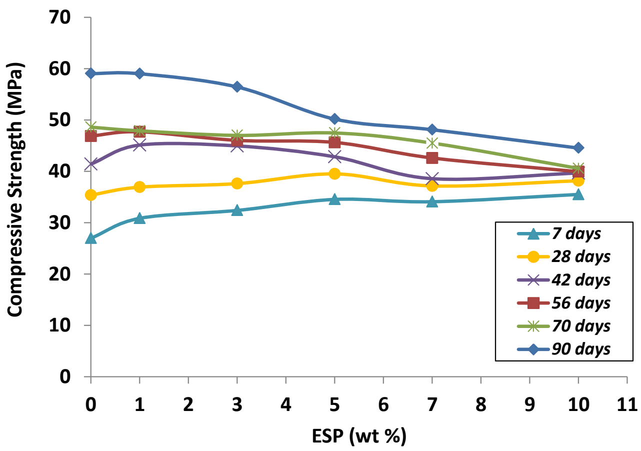

Figure 3. Relationship between ESP(%) and compressive strength of cement mortar at various curing ages

6.3 Effect of ESP on Flexural Strength

Table (7) summarizes the results of flexural strength of ESP-cement mortar and for different periods of curing. Each value presents an average of testing three prismatic samples. As shown in Figure (4), replacement of cement with percentages of ESP up to 5 wt% increases flexural strength of mortar at early age (7 days) and the highest enhancement (0bserved for samples containing 1, 3 and 5 wt% of ESP. The highest increment (9.12% and 6.38%) were recorded when cement was replaced with 5 wt% and 1 wt% of ESP respectively and at 28 days and 56 days respectively. Further increment in ESP (beyond 5 wt%) results in reduction in the strength as compared with control specimens.

Since the behaviour of ESP-cement mortar in flexural is not clear enough from the present results, further investigation is required to explore the influence of ESP on flexural strength of cement mortar for ages between 28 days and 56 days, and ages beyond 56 days and up to 90 days.

Table 7. Results of flexural test of ESP-cement mortars at different curing ages

ESP (wt%)	Flexural Strength (MPa)		
	7 days	*28 days*	*56 days*
0	5.66	7.13	8.00
1	6.86	7.64	8.51
3	5.81	7.72	8.14
5	6.3	7.78	8.44
7	5.09	6.82	7.94
10	4.75	6.75	7.50

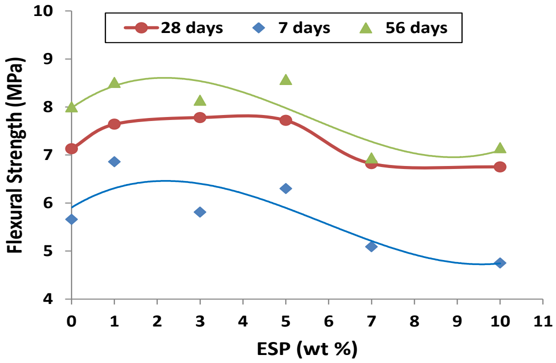

Figure 4. Relationship between ESP(%) and flexural strength of cement mortar at various curing ages

7. Conclusions

The possibility of utilize eggshell wastes as partial replacement of cement in mortars, and without using admixtures, was explored. The effect of inclusion low concentrations of ESP on properties and behaviour of cement mortar was investigated. The following could be drawn:

1. Partial replacement of cement with various concentrations of ESP causes the same expansion (0.5 mm) of cement paste when compared with control specimens.
2. A significant enhancement in compressive strength was achieved at early age (7 days) and the highest improvement (31.63%) was obtained for samples with 10 wt% of ESP.
3. The compressive strength at 28 days enhanced with the addition of ESP and the highest improvement (11.65%) was obtained for samples with 5 wt% of ESP.
4. The addition of 1 wt% of ESP results in highest improvement in compressive strength at moderate periods of curing (42 days and 56 days). The rates of enhancement are (8.93%) and (1.75%) at 42 days and 56 days respectively.
5. Reduction in late compressive strength was obtained when ESP was used to replace the cement in the mortar. The highest reduction was recorded for samples with 10wt% of ESP.
6. Flexural strength of ESP-cement mortar was higher than control samples for all curing ages (7, 28 and 56 days) and for specimens that incorporated up to 5 wt% of ESP. These enhancements are about 11.31%, 9.12% and 5.50% at 7, 28 and 56 days respectively. However, highest improvement in flexural strength (21.20%) was recorded for samples with 1 wt% at 7 days. Further investigation is required to explain the behaviour of ESP-cement mortar at ages between 28 days and 56 days, and for ages beyond 56 days and up to 90 days. Such investigation will help to draw conclusions about the optimum percentage of ESP which have a significant influence of flexural strength of cement mortar.

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