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Effect of Date Palm Seed Pod Ash and Eggshell Powder on the Physico-Mechanical Properties of Cement Blends

Iliya Bila Auta[1], Ibrahim Ikara Abdulkarim[2], Olubajo Olumide Olu[1, *]

[1]Department of Chemical Engineering, Abubakar Tafawa Balewa University, Bauchi, Nigeria
[2]Department of Civil Engineering, Abubakar Tafawa Balewa University, Bauchi, Nigeria

Email address:

ooolubajo@atbu.edu.ng (Olubajo Olumide Olu)
[*]Corresponding author

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Abstract: The aim of this research was to investigate the effect of replacing eggshell powder (ESP) with date palm seed pod ash (DPSA), curing age and cement replacement on the properties of cement blended with ESP and/or DPSA on the water consistence, setting times and mortar compressive strengths according to ASTM standards. DPSA was produced by calcining date palm seed pod at 590°C for 8 hours followed by 630°C for 3 hours and the resultant ash was ground and sieve with a 90-micron sieve. Portland limestone cement CEM II 42.5R was employed and replaced by eggshell powder and DPSA at various proportions between 0 – 12.5 wt.% at interval of 2.5 wt.% for consistence and setting times whereas cement replacement was varied between 0 -8 wt.% at interval of 2 wt.% for the mortar compressive strength by using 50 mm cubes with a mixing ratio 1:3:5 (water, binder and sand). DPSA revealed high silica content of 42.75 wt.% with SiO_2+Al_2O_3+Fe_2O_3 < 70% (45.38 wt.%) and hence may not be considered as a good pozzolana whereas ESP revealed a high lime content of 55.45 wt.% and considered a filler respectively using X-ray fluorescence spectrometer. Results indicated an increase in the water consistence for DPSA cement blend in comparison with control which related to either presence of unburnt carbon, clinker diminution or formation of magnesium hydroxide as a protective layer. Most of the cement blends experienced a diminution in the setting time compared to control except for cement blended with higher DPSA content. The accelerated and retarded setting times could possibly be due to available lime which favors ettringite instead of monosulfate and unburnt carbon present resulting in high water demand. The compressive strengths of both the control and cement experienced increments as curing age progressed with most of the blends exhibiting enhanced strength especially at the later stage at 28 and 60 days in comparison with control PLC. The reason for the enhanced strength at the later stage despite clinker diminution could be attributed to pozzolanic reaction between silica present in DPSA coupled with the available lime present in ESP. The optimal cement replacement of 4 wt.% was observed beyond which cement blends produced slightly lower strength in comparison with control owing to clinker diminution effect and higher water demand due to unburnt carbon present.

Keywords: Date Palm Seed Pod Ash, Eggshell Powder, Consistence, Setting Times, Compressive Strength

1. Introduction

The progress of nation's economic growth is dependent on the level of infrastructural development which is tied to the construction sector and an increase in the demand for cement globally could be linked with man's necessity for housing and shelter as well as infrastructural development [1]. The detrimental effects of the release of enormous carbon dioxide (CO_2) emissions via the cement production, various techniques have been considered to reduce CO_2, one of such, is the use of SCM derived from agro-industrial waste employed as a partial cement replacement. Others include development of geopolymeric concrete, carbon capturing and the use of other fuel alternatives or clinker production process. Amongst the above option, one of the most effective ways of reducing CO_2 emission is the use of SCMs which could enhance the cement properties. Similarly, the drive for sustainability in the construction sectors has driven researchers to sort to develop new technologies through management of waste materials which is enormous [2, 3]. Date palm seed (DPS) is seen to be one of these waste materials which possesses the potential to be employed in the construction sector due to its abundance in the tropical regions including Nigeria with millions of industrial wastes annually generated and are underutilized which can cause environmental issues as a result of storage and pollution [4]. The use of these innovative materials has become relevant as a result of its high calorific value thus, can be employed as a source of energy and its ash could be employed to reduce cost and improve properties due to the high production cost of cement. Researches have proven that the use of these agro-industrial wastes can produce close to or even better strength characteristics compared to OPC [5, 6]. Date palm belongs to the palm family (*Phoenix Dactyleifera*) comprising of an edible fruit and seed which are grown in temperate and arid regions. The growing demand of dates enhanced their production which exceeded 7.5 million tons in 2010 [7]. Date seeds have many uses ranging from complementary feed materials for animals and poultry, conventional soil fertilizer [8], for extracting oil for cosmetic and pharmaceutical purposes [9], as an antimicrobial agent [10], as water filter medium [11, 12], as an alternative to coffee drink [13] and as an animal / poultry feed [8, 14], as source of dietary fibres [15] and as an edible oil [16]. Limited research has been conducted on the utilization of calcined date seed pod and eggshell powder as cement replacement in a ternary blend. This study focuses on the use of DPSA and ESP as a cement replacement material and to investigate their effects on the physicomechnical properties of ternary cement blends.

Biogenic wastes used as a pozzolans is increasing finding application in the cement industry and their presence as a cement replacement material can possibly improve the physical and mechanical properties of cement. These biogenic wastes include rice husk ash, groundnut shell ash, corn cob ash, date palm seed pod ash, saw dust ash, etc. which is mainly composed of silica obtained from the calcination at controlled temperatures. Biogenic wastes pozzolans are artificial pozzolans which are ashes of agricultural plant residue. Most biogenic materials used as pozzolans undergo processing such as grinding and calcination to improve their reactivity (pozzolanic reactivity). The pozzolanic activity cannot be effectively determined by the oxide content of the main components but mainly by the amorphous nature of the materials. Some of the advantages of employing these pozzolan include resistance to sulfate and chloride attack [17], reduction in the heat of hydration in comparison with PC. Massazza and Costa [17], reduction in the alkali silica reaction [18] and reduction in the cost of cement production [6]. The formation of soluble CH which occupies about 25 wt.% if the hydrated cement paste leading to an increase in the mortar porosity and making it susceptible to sulfate attack, thus the use of these pozzolan can provide reactive silicates during hydration to react with the CH available to form additional CSH which are superior forming denser microstructure resulting in strength improvement [19, 20]. The economy and green potentials of these wastes seem to globally stimulate and generate research interests [21]. Utilization of these biogenic wastes dumped as landfills and constituting environmental issues can be overemphasized and its blends durability can be influenced by density, porosity, water cement ratio, hydration degree, supplementary cementitious materials content, aggregates and filter content [22]. According to Balendian and Martin [23], eggshell is composed of 95% $CaCO_3$ and 5% Mg, Al, P, Na, K, Zn Fe, Cu, ironic acid and silica acid. The use of recycled materials as aggregates partial replacement material produced similar concrete compressive strength with those of virgin concrete such as date palm seed [24], coconut shell [25], palm kernel seed [26] with its concrete compressive strength diminishing as coarse aggregate was gradually replaced with date palm seed. Gunarani & Chakkravarthy [27] investigated the impact of cement replacement with date seed ash (obtained by oven drying and open air burning until the seed internal moisture is lost and then ground and sieved to below 90 μm) on the mortar compressive strength from 0-10% at 2% interval and experienced an increase with an optimal strength at 4% cement replacement beyond which resulted in the diminution in the mortar compressive strength especially at 14 and 28 days. It was also seen that the mortars blended with 4 % date palm seed ash exhibited lower water absorption index compared with other replacement which were relatively high.

Gunarani & Chakkravarthy [27] drive to employ date palm seed ash as a cement replacement material comes from the backdrop after attempting the use of date palm seed as a coarse aggregate replacement material. Al-Kutti *et al*. [4] and Nasir and Al-Kutti [1] studied the effect of replacing cement with Date palm ash (DPA) obtained by burning local palm frond in an oven for 7 hours, passed through 425µm with specific gravity of 2.43 on the mechanical, durability and microstructural performances from 10-30% at 10% interval and the optimal cement replacement of 10% with DPA produced a significantly better strength compared to OPC after 360 days. DPA constituted major oxides of Si, Ca, Mg, and K totaling above 80% and minor traces of Fe and Al and by partially replacing cement with DPA as binders, Si, Mg, and K content increased while Ca content experienced a decrease. Cement blended with 10% PA produced lower 3 days compressive strength compared with other replacements while beyond 28 days, the compressive strength was enhanced in comparison with OPC and other blends which could be attributed to the formation of CSH gel at the latter ages [6, 28]. Zain *et al*. [29] suggested that the high alkaline oxides like K_2O content (above 7.40 wt.%) could be attributed to the type and quantity of fertilizers employed during the growth of the date palm trees. Adefemi *et al*. [24] investigated the effect of replacing crushed granite with date seeds up to 100% at interval of 25% and observed that the 28 days concrete strength diminished by 37.98% and 46.36% as the crushed granite was replaced with date seeds for 1:2:4 and 1:3:6 respectively. The 28 days strength diminished by

9.15% as crushed granite was replaced by 25% with date seed. Smith *et al*. [30] investigated the effect of DPSA particle size on concrete properties by varying particle size between 600, 300, 150 and 75 microns and observed a decrease in the consistence and setting time of the blend as fineness was enhanced which also produced the best compressive strength but produced consistence and setting time results higher than control. The DPSA was obtained from controlled burning of DPS at maximum of 590°C for 8 hrs followed by cooling for 2 days, then the resultant product was heated at 630°C for 3 hrs and then cooled for 3 days and sieved by 75 microns before XRF analysis was conducted. Almograbi [31] investigated the use of DPS as an aggregate in light weight concrete and concluded that it possesses significantly good durability properties such as water permeability, water absorption and sorptivity tests in comparison with other conventional lightweight concrete. It is almost spherical and elongated shaped seed with specific gravity of 1.13, water absorption of 36% (24 hours) and maximum size of 15 mm. One distinctive property is that DPS is a naturally light weight material collimating in a reduction in the total dead load of a structure.

2. Materials and Method

2.1. Materials

The date palm seed pod employed in this research was sourced from Bauchi metropolis and sun dried for several days to obtain uniform weight. The date palm seed pod were sieved to remove unwanted materials and then calcined with a furnace at 590°C for 8 hours and then heated at 630°C for 3 hours according to Smith *et al*. [30] to obtain date seed pod ash (DPSA). The resultant ash was first cooled and then ground and sieved with 90µm sieve. Portland limestone cement CEM II 42.5R was obtained from Dangote supplier was employed for this research while distilled water was used for the physico-mechanical properties of the cement blended with date palm pod ash and eggshell powder. The standard sand was obtained in Bauchi by sieving sand into various classes according to Indian standards which was used as aggregates in the production of mortars.

(a) (b) (c)

Figure 1. *(a) Date palm seed pod (b) Date palm seed pod ash (c) Eggshell powder.*

2.2. Methods

Characterization of DPSA, PLC and ESP were conducted to determine the chemical compositions via X-ray fluorescence spectrometer (XRF). The standard consistence and initial and final setting time were measured using a Vicat apparatus after blending DPSA and ESP with the PLC together and placed in a mould based on the mix proportion for the standard consistence, setting times and soundness presented in Table 3. The water for standard consistence was determined according to ASTM C187 [32], after which the initial setting time was obtained from a series of penetration measurements at interval of 10 minutes conducted until the needle fails to penetrate the cement paste surface to 5 +/- 0.5mm measured from the bottom of the mould. The initial setting time value was then obtained from the average of the initial setting time values and recorded while the final setting time was obtained when only annular needle makes an impression on the top surface of the cement paste and the period between when the water was added to the cement and when the slight impression on the surface of the mould is determined as the final setting time which are in accordance to ASTM C191 [33]. The experimental mix for 1:3:5 (water: cement: aggregate) were conducted. The various proportion of the cement blends as illustrated in Table 1 were mixed properly and then casted into 50 mm cube moulds which were assembled and lubricated for easy removal. The mortars were prepared from 0 – 8 wt.% of ESP replaced by 0, 25%, 50%, 75% and 100% by DPSA. Two hundred and fifty-two (252) mortar specimen were casted at the experimental matrix and were cured in tap water by total immersion of the specimen and tested total immersion of the specimen and tested for 3, 7, 28 and 60 days.

With a loading ratio of 3KN/s was applied according to BS 1881 [55] to determine the effect of replacing ESP with DPSA on the mortar compressive strength of ternary cement.

Table 1. *Experimental matrix for consistence and setting time of PLC and various cement blends.*

S/No	Blends	DPSA / DPSA ESP	ESP (%)	DPSA (%)	PLC (%)	ESP (g)	DPSA (g)	PLC (g)
1	PLC	0.0	0.0	0.0	100.0	0.0	0.0	300.0
2	2.5ESP	0.0	2.5	0.0	97.5	7.5	0.0	292.5
3	2ESP0.5DPSA	0.2	2.0	0.5	97.5	6.0	1.5	292.5
4	1.5ESP1 DPSA	0.4	1.5	1.0	97.5	4.5	3.0	292.5
5	1.25ESP1.25 DPSA	0.5	1.25	1.25	97.5	3.75	3.75	292.5
6	1ESP1.5 DPSA	0.6	1.0	1.5	97.5	3.0	4.5	292.5
7	0.5ESP2 DPSA	0.8	0.5	2.0	97.5	1.5	6.0	292.5
8	2.5 DPSA	1.0	0.0	2.5	97.5	0.0	7.5	292.5
9	5ESP	0.0	5.0	0.0	95.0	15.0	0.0	285.0
10	4ESP1DPSA	0.2	4.0	1.0	95.0	12.0	3.0	285.0
11	3ESP2 DPSA	0.4	3.0	2.0	95.0	9.0	6.0	285.0
12	2.5ESP2.5 DPSA	0.5	2.5	2.5	95.0	7.5	7.5	285.0
13	2ESP3 DPSA	0.6	2.0	3.0	95.0	6.0	9.0	285.0
14	1ESP4 DPSA	0.8	1.0	4.0	95.0	3.0	12.0	285.0
15	5 DPSA	1.0	0.0	5.0	95.0	0.0	15.0	285.0
16	7.5ESP	0.0	7.5	0.0	92.5	22.5	0.0	277.5
17	6ESP1.5 DPSA	0.2	6.0	1.5	92.5	18.0	4.5	277.5
18	4.5ESP3 DPSA	0.4	4.5	3.0	92.5	13.5	9.0	277.5
19	3.75ESP3.75 DPSA	0.5	3.75	3.75	92.5	11.25	11.25	277.5
20	3ESP4.5 DPSA	0.6	3.0	4.5	92.5	9.0	13.5	277.5
21	1.5ESP6 DPSA	0.8	1.5	6.0	92.5	4.5	18.0	277.5
22	7.5 DPSA	1.0	0.0	7.5	92.5	0.0	22.5	277.5
23	10ESP	0.0	10.0	0.0	90.0	30.0	0.0	270.0
24	8ESP2 DPSA	0.2	8.0	2.0	90.0	24.0	6.0	270.0
25	6ESP4 DPSA	0.4	6.0	4.0	90.0	18.0	12.0	270.0
26	5ESP5 DPSA	0.5	5.0	5.0	90.0	15.0	15.0	270.0
27	4ESP6 DPSA	0.6	4.0	6.0	90.0	12.0	18.0	270.0
28	2ESP8 DPSA	0.8	2.0	8.0	90.0	6.0	24.0	270.0
29	10 DPSA	1.0	0.0	10.0	90.0	0.0	30.0	270.0
30	12.5ESP	0.0	12.5	0.0	87.5	37.5	0.0	262.5
31	10ESP2.5DPSA	0.2	10.0	2.5	87.5	30.0	7.5	262.5
32	7.5ESP5DPSA	0.4	7.5	5.0	87.5	22.5	15.0	262.5
33	6.25ESP6.25DPSA	0.5	6.25	6.25	87.5	18.75	18.75	262.5
34	5ESP7.5DPSA	0.6	5.0	7.5	87.5	15.0	22.5	262.5
35	2.5ESP10DPSA	0.8	2.5	10.0	87.5	7.5	30.0	262.5
36	12.5DPSA	1.0	0.0	12.5	87.5	0.0	37.5	262.5

3. Results and Discussion

Table 2 indicates chemical compositions of several date palm ashes with their authors and date palm seed pod ash, eggshell powder and Portland limestone cement were obtained via Xray fluorescence spectrometer while Figure 1 (a), (b) and (c) depicts date palm seed pods, date palm seed pod ash and eggshell powder respectively. The PLC comprises of similar major constituents such as SiO_2, CaO, Al_2O_3 and minor traces of MgO, Fe_2O_3, SO_3 in comparison with OPC from Nasir and Al-Kutti [1] works whereas DPSA major constituents include SiO_2, CaO, MgO, K_2O, SO_3 which summed up to 79.98 wt.% while the minor traces like Al_2O_3, Fe_2O_3, Na_2O, Cl. ESP indicated mainly CaO with minor traces like SiO_2, CaO, MgO, K_2O, Na_2O, SO_3, Al_2O_3, P_2O_5. The chemical analysis of DPSA indicated a total percentage composition of Aluminium Oxide (Al_2O_3 = 1.99 wt.%), Silicon Oxide (SiO_2 = 42.75%) and Iron oxide (Fe_2O_3 = 0.64 wt.%) totaling 45.38 wt.%, which does not satisfies the 70% minimum requirement specified by ASTM C311/C311M-16, and hence it may not be a good pozzolana. The analysis also showed that DPSA has a moderate calcium oxide content (CaO = 4.76 wt.%) which makes the ash to be self-cementitious. Alkali like K_2O (22.27 wt.%) and Na_2O (1.56 wt.%) whose K_2O content depends on the type and amount of fertilizer employed during planting according to Zain *et al.* [29], which may inhibit the formation of Calcium trisilicates in concrete as reported by Mahmoud, [53]. The DPSA moderate LOI of 5.51wt.% could be dependent on the duration of combustion, duration of air supply and the cooling process [4]. The low specific gravity of PLC of 2.90 indicate the inclusion of limestone which possesses a lower specific gravity compared to Portland limestone cement. Results from the standard consistence, water demand and both setting time tests of the various experimental mix for PLC and 35 cement blend specimens are tabulated in Table 3.

Table 2. *Chemical composition of Portland limestone cement, eggshell powder and various authors date palm ashes.*

Oxides	PLC wt.%	ESP wt. %	DPSA wt.%	Smith *et al.* [30]	Nasir and Al-Kutti [1]	Al-Kutti *et al.* [4]
SiO_2	12.39	0.58	42.75	45.50	42.20	35.97
Al_2O_3	4.20	0.1	1.99	20.75	1.10	0.65
Fe_2O_3	1.95	0.19	0.64	7.25	3.20	0.18
CaO	43.14	55.54	4.76	9.82	19.20	13.04
MgO	0.74	0.53	5.04	0.52	14.10	6.36
K_2O	0.63	0.02	22.27	9.07	5.70	7.04
Na_2O	0.09	0.08	1.56	3.00	0.90	3.60
SO_3	1.03	0.6	5.16	0.12	3.60	
Cl	-	-	2.11	0.45	7.00	
P_2O_5	0.18	0.48	-	-		
Mn_2O_3	0.10	0.01	-	-		
TiO_2	0.19	0.09	-	-		
Impurities		-	8.60	-		
LOI	34.67	44.17	5.12	1.72	3.60	8.41
Total	99.32	99.13	100	-		
Specific gravity	2.99	2.47		-	2.43	2.43

Table 3. *Results of DPSA-ESP cement blends for setting times and consistence.*

S/No	Blends	DPSA / DPSA ESP	Consistence %	Water demand Mm	IST/FST mins
1	PLC	0	33.0	99.0	213/431
2	2.5ESP	0	31.0	93.0	197/307
3	2ESP0.5DPSA	0.2	35.0	105.0	206/309
4	1.5ESP1 DPSA	0.4	30.0	90.0	199/361
5	1.25ESP1.25 DPSA	0.5	31.0	93.0	211/521
6	1ESP1.5 DPSA	0.6	33.0	99.0	201/397
7	0.5ESP2 DPSA	0.8	33.0	99.0	221/411
8	2.5 DPSA	1	32.5	97.5	235/436
9	5ESP	0	30.2	90.6	193/378
10	4ESP1DPSA	0.2	30.3	90.9	197/369
11	3ESP2 DPSA	0.4	31.0	93.0	199/343
12	2.5ESP2.5 DPSA	0.5	31.5	94.5	200/334
13	2ESP3 DPSA	0.6	32.5	97.5	223/389
14	1ESP4 DPSA	0.8	33.7	101.1	232/416
15	5 DPSA	1	33.8	101.4	243/498
16	7.5ESP	0	31.3	93.9	202/316
17	6ESP1.5 DPSA	0.2	32.0	96.0	197/319
18	4.5ESP3 DPSA	0.4	33.4	100.2	213/327
19	3.75ESP3.75 DPSA	0.5	32.2	96.6	209/378
20	3ESP4.5 DPSA	0.6	32.3	96.9	213/403
21	1.5ESP6 DPSA	0.8	34.1	102.3	237/610
22	7.5 DPSA	1	33.7	101.1	248/514
23	10ESP	0	32.0	96.0	201/311
24	8ESP2 DPSA	0.2	31.0	93.0	207/299
25	6ESP4 DPSA	0.4	29.7	89.1	221/362
26	5ESP5 DPSA	0.5	33.5	100.5	219/410
27	4ESP6 DPSA	0.6	33.2	99.6	229/718
28	2ESP8 DPSA	0.8	34.0	102.0	239/831
29	10 DPSA	1	35.6	106.8	246/873
30	12.5ESP	0	29.6	88.8	201/316
31	10ESP2.5DPSA	0.2	30.0	90.0	206/319
32	7.5ESP5DPSA	0.4	31.5	94.5	199/394
33	6.25ESP6.25DPSA	0.5	30.6	91.8	179/387
34	5ESP7.5DPSA	0.6	32.6	97.8	187/318
35	2.5ESP10DPSA	0.8	31.1	93.3	169/249
36	12.5DPSA	1	34.6	103.8	241/864

3.1. Initial Setting Time of Ternary Cement Blends Containing DPSA and ESP

The initial setting times of cement blended with ESP gradually replaced with DPSA at 2.5 wt.% cement replacement experienced an acceleration in comparison with control especially for DPSA/DPSA-ESP ratio below 0.8, beyond which resulted in elongated setting time.

Similar trends of accelerated setting times were also experienced for cement replacements from 5 - 12.5 wt.% at DPSA/DPSA-ESP ratios below 0.6, 0.8. 0.4 and 1.0 respectively. The decrease in the setting time experienced by significant proportion of the cement blends could be attributed to the available lime in ESP which influenced the hydration assembly, thereby favoring ettringite formation, thus accelerating the setting time which is in agreement with works by Olubajo *et al*. [6], De Weerdt *et al*. [34] and Lothenbach *et al*. [35].

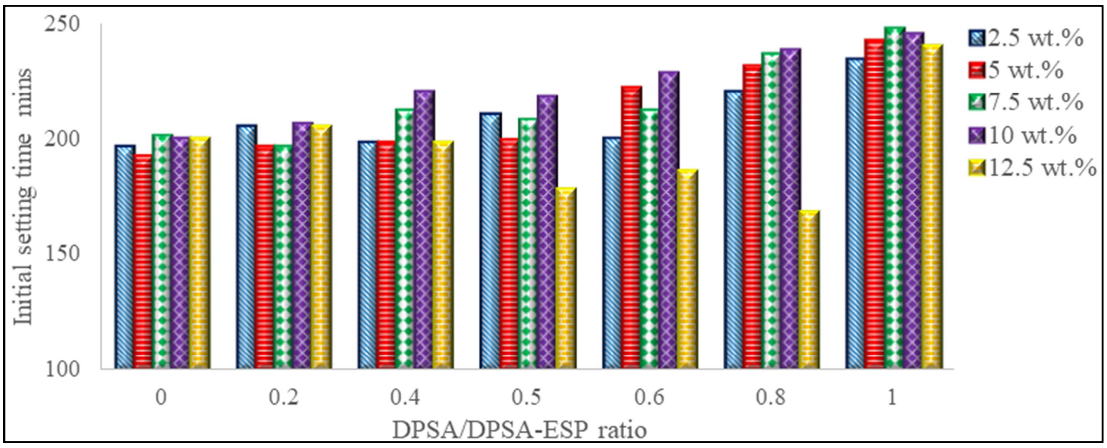

Figure 2. *Variation of DPSA/DPSA-ESP ratio on the Initial setting time of cement blends at various cement replacements.*

Whereas, the retardation in the initial setting time experienced as DPSA content was significantly higher than ESP content could be attributed to the presence of unburnt carbon in the ash as a result of DPSAs' LOI of 5.12 wt.%. Thus, requiring more water and thereby retarding the initial setting time of the cement blends at high DPSA/DPSA-ESP ratios similar to Olubajo *et al*. [36, 37] and Kaya [38]. Similarly owing to the high magnesium content present in the DPSA, according to Olubajo *et al*. [36], Venkateswara *et al.* [39] and Deng [40] could result in the formation of magnesium hydroxide Mg $(OH)_2$ which produces a protective layer which retards the setting time. Likewise, the high potassium content can result in retardation of the setting time of cement replaced with DPSA due to an increase in KCl content. Thus, an elongation in the initial setting time was experienced indirectly as the ESP content was replaced with DPSA content at given cement replacement of 2.5 – 10wt.% with the exception of 12.5 wt.%. It could also be observed that as ESP content was replaced by DPSA content, the initial setting time retarded by an average of approximately 45 minutes with most of the cement blends produced higher setting time results compared to the control between DPSA/DPSA-ESP ratio of 0.8 – 1.0 respectively.

The variation (series of accelerations and a retardation) in the initial setting time was experienced for DPSA-DPSA-ESP ratio of 0, 0.2 and 0.5 as the cement replacement was gradually increased from 2.5 – 12.5 wt.% respectively while for DPSA/DPSA-ESP ratio of 0.4, 0.8 and 1.0 experienced a series of retardations followed by an acceleration in its initial setting time whereas DPSA/DPSA-ESP ratio of 0.6 experienced a series of retardations and accelerations as cement replacement was increased.

3.2. Final Setting Time of Cement Blended with DPSA and ESP

Similar trend of accelerated final setting times of cement blended with ESP gradually replaced with DPSA were experienced for cement replacements from 2.5 - 12.5 wt.% at DPSA/DPSA-ESP ratios below 1.0, 1.0, 0.8. 0.6 and 1.0 respectively. Five cement blends namely 1.5ESP6DPSA, 4ESP6DPSA 2ESP8DPSA 10DPSA 12.5DPSA were all observed to produce final setting times which were above 10 hours and not in accordance to BS 4550-3-3.6 [41].

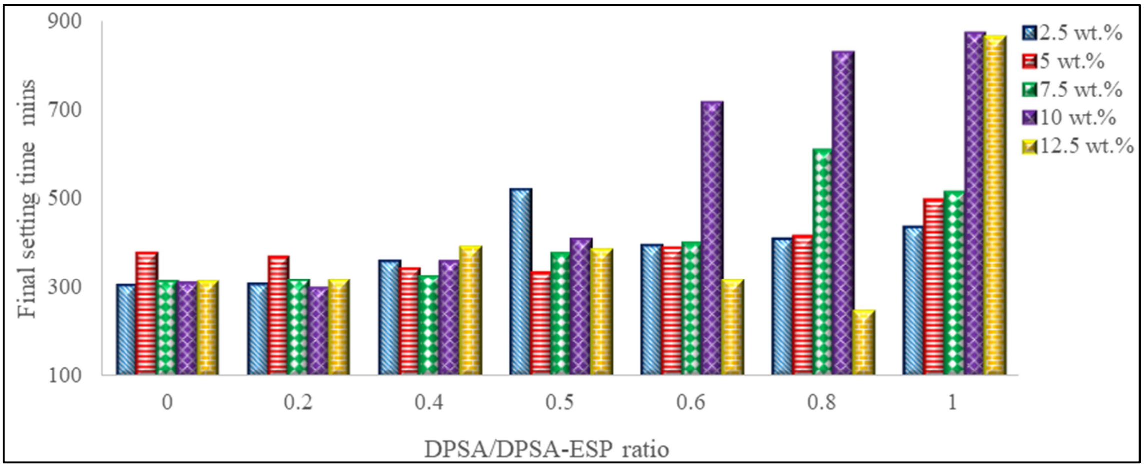

Figure 3. *Variation of DPSA/DPSA-ESP ratio on the Final setting time of cement blends at various cement replacements.*

On the other hand, an increase in the cement replacement resulted in a series of retardations and acceleration, at DPSA/DPSA-ESP ratio of 0 and 0.2, while an acceleration followed by a series of retardation in its final setting time was experienced at DPSA/DPSA-ESP ratio of 0.4. while for DPSA/DPSA-ESP ratio of 0.5 and 0.6 indicated a series of accelerations and retardations while at DPSA/DPSA-ESP ratio of 0.8 and 1.0 observed a series of retardations followed by an acceleration in its final setting time respectively.

3.3. Comparison of Setting Times of DPSA-Cement Blends and ESP-Cement Blends with Portland Limestone Cement

ESP cement blends produced lower setting time results compared to control. The accelerated setting times compared to control could be attributed to the presence of available lime which influences the hydration assembly, thereby favoring formation of ettringite instead of monosulfate, thus enhancing hydration rate. As the cement replacement with ESP content was increased from 2.5 – 12.5 wt.%, the initial and final setting times accelerated between 11- 20 minutes /53 – 124 minutes respectively. Whereas DPSA cement blends produced a higher setting times (experienced a retardation) and the initial and final setting times lengthened between 22- 35 minutes /5 – 442 minutes as the cement replacement with DPSA was increased between 2.5 – 12.5 wt.% respectively. The retardation in the setting times of the DPSA cement blends could be due to the presence of unburnt carbon evident by the relatively moderate LOI of DPSA coupled with MgO content present in DPSA resulting in the formation of $Mg(OH)_2$ which retards hydration rate by provide a protective layer [37, 40].

3.4. Consistence of Ternary Cement Blends Containing DPSA and ESP

For 2.5 wt.% cement replacement, all the consistence of the various cement blends required less water demand compared with control except for cement blend with DPSA/DPSA-ESP ratio of 0.2. Whereas, cement replacement between 5 - 12.5 wt.% produced a lower water consistence in comparison with control except for 5 wt.% (DPSA/DPSA-ESP ratio = 0.8, 1); 7.5 wt.% (DPSA/DPSA-ESP ratio = 0.4, 0.8, 1) 10 wt.% (DPSA/DPSA-ESP ratio = 0.5, 0.6, 0.8, 1) 12.5 wt.% (DPSA/DPSA-ESP ratio = 1) respectively. According to Erdoğdu [42] & Olubajo *et al*. [6] reported that consistence decreased as cement was replaced with eggshell powder while Olubajo [37] observed that eggshell powder which is similar to limestone experienced similar trend and attributed its decrease in consistence to the smooth surface (enhanced surface area) and lower porosity of eggshell powder after grinding.

On the other hand, the inclusion of DPSA resulted in an increase in its consistence which could be linked with the unburnt carbon present in the ash as well as diminution of the clinker content due to cement replacement, thus requiring more water [34, 43, 44]. Another reason for an increase in the water demand could be due to the presence of significant MgO content in DPSA resulting in formation of Mg $(OH)_2$ which retards hydration by producing a protective layer and thus requiring more water to reach consistence [37, 40].

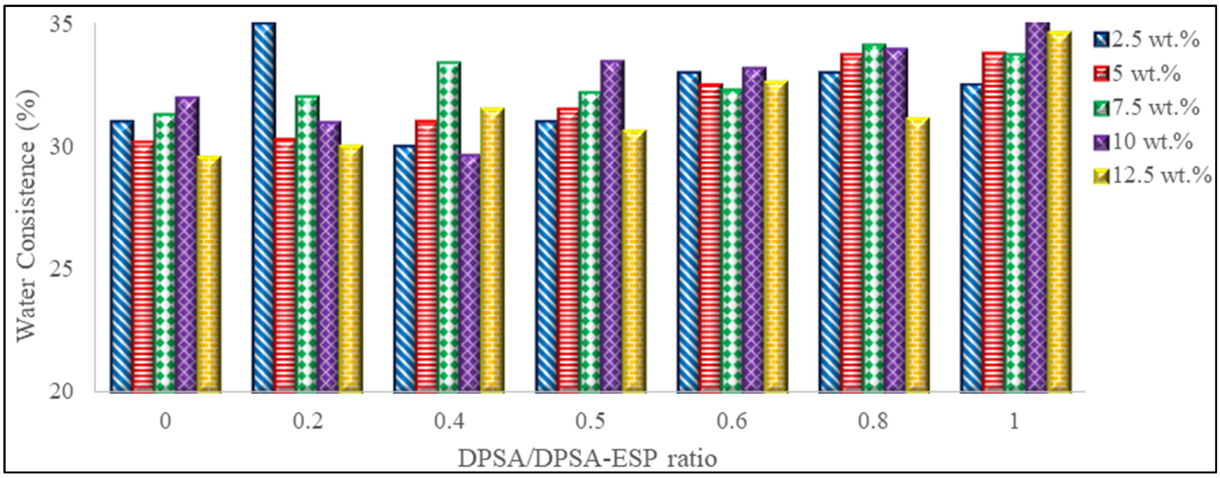

Figure 4. *Variation of DPSA/DPSA-ESP ratio on the water consistence of cement blends at various cement replacements.*

3.5. Mortar Compressive Strength of Cement Blended with DPSA and ESP

The mortar compressive strength of cement blended with ESP and/or DPSA at various DPSA/DPSA-ESP ratios between 0 -1 at interval of 0.25, cement replacements between 2- 8 wt.% at various curing ages of 3, 7, 28 and 60 days were tabulated in Table 4. The mortar compressive strengths of the cement blends were carried out in triplicates using a compression testing machine with uniform load of 3KN/s and average of the three specimen mortar strengths were obtained and tabulated below while Figures 5-8 indicate the influence of DPSA/DPSA-ESP ratios, curing days and cement replacement on the mortar compressive strength of

DPSA- ESP -cement blends.

Table 4. *Compressive Strength Results for DPSA-ESP cement blends at various proportions.*

Cement samples	DPSA /DPSA-ESP ratio	Curing age			
		3 days	7 days	28 days	60 days
PLC	0	24.52	25.58	32.93	35.20
2ESP	0	23.43	33.54	35.65	43.76
1.5ESP0.5DPSA	0.25	19.73	22.94	35.57	39.99
1ESP1DPSA	0.5	22.79	28.67	34.59	36.72
0.5ESP1.5DPSA	0.75	32.67	34.88	37.46	39.02
2DPSA	1	27.41	28.08	30.80	36.57
4ESP	0	20.85	21.17	34.12	41.68
3ESP1DPSA	0.25	24.52	25.51	32.56	37.04
2ESP2DPSA	0.5	28.22	33.41	39.74	40.04
1ESP3DPSA	0.75	19.46	27.33	35.33	36.70
4DPSA	1	18.57	22.17	33.24	34.90
6ESP	0	25.06	26.27	34.25	41.68
4.5ESP1.5DPSA	0.25	16.19	19.55	28.90	38.06
3ESP3DPSA	0.5	32.43	32.85	35.26	37.40
1.5ESP4.5DPSA	0.75	25.73	28.62	29.24	37.38
6DPSA	1	13.66	17.20	26.86	34.32
8ESP	0	26.69	30.93	35.45	37.72
6ESP2DPSA	0.25	18.45	26.93	29.99	39.12
4ESP4DPSA	0.5	15.24	20.04	31.41	37.30
2ESP6DPSA	0.75	17.81	17.89	25.03	33.80
8DPSA	1	16.54	19.39	25.58	33.94

Figure 5 indicates the variation of curing age and DPSA content described by DPSA-DPSA-ESP ratio on the mortar compressive strength of cement blended with ESP and/or DPSA at 2 wt.% cement replacement. It could be observed that at 3 days, cement blended with higher DPSA content produced an enhanced early strength compared with those containing a higher ESP content with a better mortar compressive at DPSA/DPSA-ESP ratio of 0.75 and 1.0. The enhanced early strength could be attributed to presence of lime in DPSA matrix which tend to hasten the rate of hydration via formation of carboaluminate which favors ettringite instead of monosulfate due to interaction of C_3A and the available lime [45]. Another reason is due to the provision of more nucleation sites by formation of calcium hydroxide at the early stage coupled with additional lime from ESP inclusion [45, 46], thus enhancing its early strength [47, 48].

The strength of all the cement blends including the control experienced an increase in their strengths as the curing day progressed as shown in Figures 5-8 respectively. It was also observed that most of cement blends produced better strength compared to control at 60 days. The 60-day strength produced enhanced strength of 43.76, 41.68, 41.68 and 40.04 N/mm^2 as against control of 35.20 N/mm^2 at DPSA/DPSA-ESP ratio of 0.0, 0.0, 0.0 and 0.5 respectively. This enhanced strength is evidence that despite the slight diminution of the clinker content due to the following reasons; the provision of nucleation site as a result of inclusion of available lime in ESP matrix [37, 49-50]; pozzolanic reaction between the available lime after cement hydration and silica present in the various ashes [2, 36-37, 44, 51]. Another reason for the enhanced strength could be due to presence of potassium oxide in DPSA composition increased the strength as suggested by Venkateswara *et al.* [39] resulting in the formation of muscovite thereby increasing its strength [37, 52].

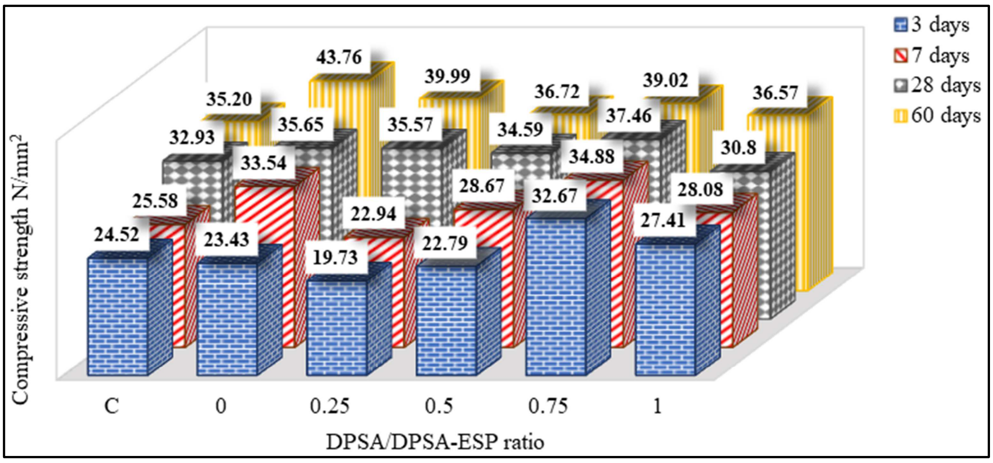

Figure 5. *Variation of curing age on the mortar compressive strength at various DPSA/DPSA-ESP ratio for 2 wt. % cement replacement.*

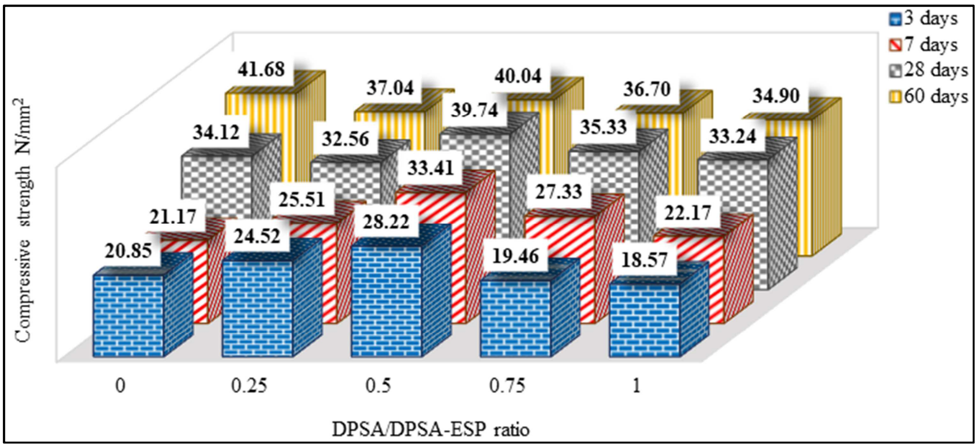

Figure 6. *Variation of curing age on the mortar compressive strength at various DPSA/DPSA-ESP ratio for 4 wt. % cement replacement.*

Figure 6 illustrates the gradual replacement of ESP content with DPSA content at 4 wt.% cement replacement on the mortar compressive strength of various cement blends. The early strength indicated enhanced strengths in comparison with control at DPSA/DPSA-ESP ratio of 0.25 and 0.50 for 3 days while DPSA/DPSA-ESP ratio of 0.50 and 0.75 for 7 days respectively. It was also observed that beyond 7 days, most of the cement blend produced higher strength compared with PLC control despite diminution of clinker by 4 wt.% which could be attributed to either the additional surface area provided by ESP, thus producing more nucleation sites and growth of hydration products resulting in further strength gain in the case of ESP-cement blends and for cement blends containing ESP and DPSA experiencing pozzolanic reactions as well as formation of muscovite thereby, enhancing their later strength [37, 52, 54]. Cement blends with higher ESP content produced better mortar compressive strengths in comparison with cement blends with higher DPSA content. The optimum level for ternary cement blend of ESP content was obtained at 4 wt.% replaced with DPSA up to 2 wt.% (DPSA/DPSA-ESP ratio = 0.5).

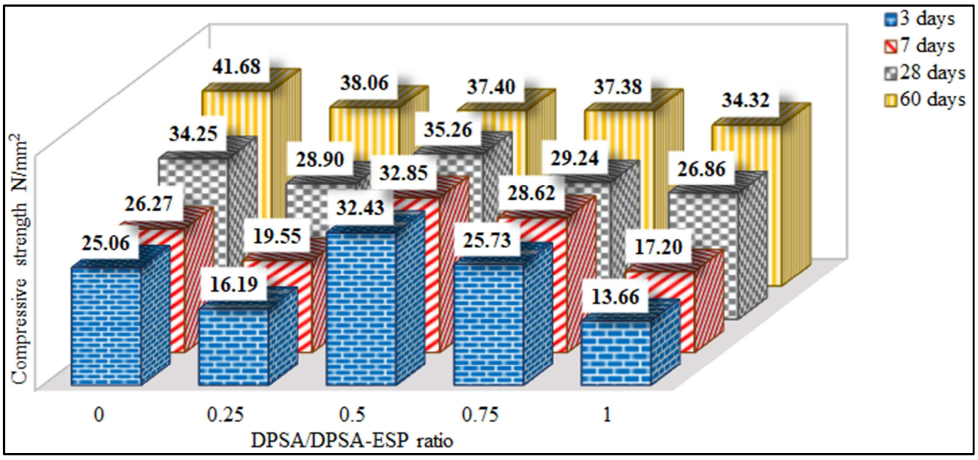

Figure 7. *Variation of curing age on the mortar compressive strength at various DPSA/DPSA-ESP ratio for 6 wt. % cement replacement.*

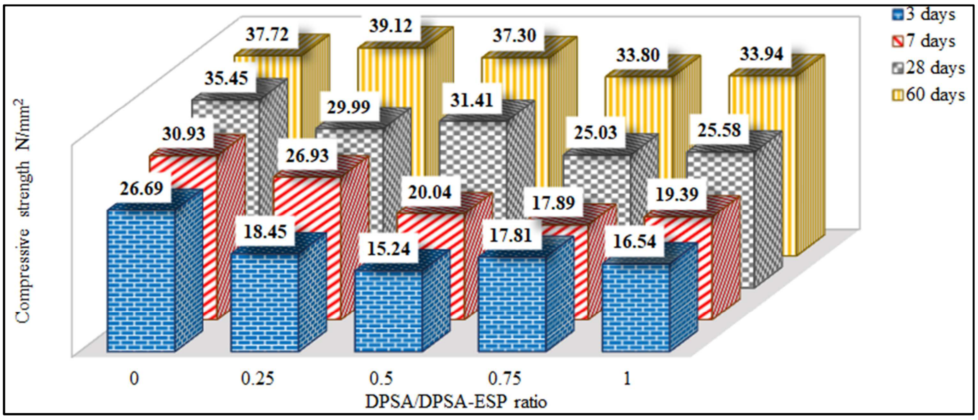

Figure 8. *Variation of curing age on the mortar compressive strength at various DPSA/DPSA-ESP ratio for 8 wt. % cement replacement.*

Figures 7 and 8 indicate the variation of DPSA/DPSA-ESP ratio and curing days as a function of the mortar compressive strength of various cement blends at cement replacement of 6 and 8 wt.% respectively. Similar trend of increase in the strength as the curing day progressed. For cement replacement of 6 wt.% at DPSA/DPSA-ESP ratio of 0.5 produced better mortar compressive strength results for 3, 7 and 28 days in comparison with other cement blends respectively. Whereas for 8 wt.% indicated a diminution in the mortar compressive strength as ESP was gradual replaced with DPSA for various cement blends especially for 3, 7, 28 days which could be attributed to diminution of clinker content while for 60 days, it could be seen that DPSA/DPSA-ESP ratio of 0.25 produced a higher strength of 39.12 N/mm^2 and 0.50 produced slightly lower strength of 37.30 N/mm^2 compared to control. This enhanced strength could be related to either the provision of nucleation sites as result ESP inclusion or pozzolanic reaction between silica and the available lime at 60 days. A significant decline in the mortar compressive strength at high cement replacement was experienced as ESP was replaced with DPSA content for the various curing days.

4. Conclusions

The chemical analysis of DPSA indicated a total percentage composition of Aluminium Oxide (Al_2O_3 = 1.99 wt.%), Silicon Oxide (SiO_2 = 42.75%) and Iron oxide (Fe_2O_3 = 0.64 wt.%) totaling 45.38 wt.%, which does not satisfy the 70% minimum requirement specified by ASTM C311/C311M-16, and hence may not be considered as a good pozzolana. DPSA composition contained a moderate calcium oxide content (CaO = 4.76 wt.%) which makes the ash to be self-cementitious and K_2O content of 22.27 wt.% which is dependent on the type and amount of fertilizer used during cultivation. On the other hand, ESP revealed a high lime content of 55.45 wt.% and considered a filler.

The water demand or water consistence of cement blended with higher DPSA content required more water to attain standard consistence compared to those with higher ESP content. The increase in water demand of cement blended with DPSA and/or ESP content could be linked to the following factors such as unburnt carbon presence, clinker diminution or formation of magnesium hydroxide which retard hydration by provision of protective layer thus requiring more water. Most of the cement blended with DPSA and ESP produced accelerated setting time in comparison with control except for cement blends with higher DPSA content for various cement replacements of DPSA/DPSA-ESP ratio after 0.6, 0.5, 0.6, 0.2 and 0.8 for initial setting time and of DPSA/DPSA-ESP ratio after 0.6, 0.8, 0.6, 0.5 and 0.8 for final setting time respectively. The accelerated setting time could be attributed to the presence of available free lime which influences the hydration assembly thus favoring ettringite formation at the expense of monosulfate while the retarded setting time owing to presence of unburnt carbon leading to higher water requirement. The control and the various cement blends experienced increment in their strength as curing days progressed by 15.32 - 151.2 % of the cement blends 3 days strength. Most cement blended with DPSA and ESP exhibited enhanced strength especially at the later stage at 28 and 60 days in comparison with control PLC. This enhanced strength experienced at the later stage despite the diminution of clinker could be attributed to pozzolanic reaction between silica present in DPSA coupled with the available lime present in ESP. The optimal cement replacement of 4 wt.% was observed beyond which cement blends produced slightly lower strength in comparison with control owing to clinker diminution effect. Other reasons could be linked to more water requirement for cement blended with higher DPSA content due to unburnt carbon present.

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