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Properties of Concrete Containing Mussel (*Perna viridis*) Shell Ash as Partial Cement Replacement

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Abstract: This paper present data on physical, chemical and engineering properties of concrete such as compressive and spilt tensile strength using mussel shell ash (MSA) (*Perna viridis*) as partial cement replacement. Samples of concrete were mixed with different proportions of 0%, 3%, 5% and 7% (S0, S3, S5 & S7) MSA with target strength of 35 MPa on day 28. The samples were cured in water for 7, 28 and 60 days. The properties of MSA were identified through tests including X-Ray Fluorescence (XRF), setting time, particle size distribution (PSD), specific gravity and scanning electron microscopy (SEM). The results showed that the specific gravity of MSA was lower compared to cement while the XRF test showed that the content of calcium oxide (CaO) in mussel shell ash (71.5%) was higher than that of Portland cement (57.2%). The initial and final setting time for the cement paste became shorter when the percentage of MSA increased. From the observation of the microstructure analysis, the SEM test showed that there was a significant increase in calcium hydroxide (CH) content along with the increment in mussel shell ash content in concrete. The strength tests (compressive strength and tensile strength) were carried out and analyzed in terms of correlation. It was found that mussel shell ash influenced the strength of concrete especially for concrete containing 3% mussel shell ash as a partial cement replacement (S3).

Keywords: mussel shell ash, partial replacement, concrete, calcium oxide, strength

1. Introduction

Concrete is one of material that mostly applied in construction sector and its qualities has gone through many changes accordance to technological progress. The strength and its properties can be defined according to water, cement, fine and coarse aggregates ratio that been used. However, the utilization of waste in concrete had become one of interest among researchers due to its potential in produce high strength and durability of concrete, cost in production materials and also reduce the quantity of waste generated.

Mussel production is one of fisheries sector that contributed in economy income in Malaysia. According to Department of Fisheries Malaysia 2016, it was reported that the total mussel production in year 2014 was 43 thousand

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metric tons and increased to 45 thousand metric tons in year 2015 [1]. However, the increment of this mussel production had drawn to the increments of waste that been generated from its shells. Figure 1 shows the descriptive of mussel shell. Regarding to this situation, this could cause pollution environment and diseases due to dirty and insufficient dumping area. Besides mussel waste, cement production also becoming one of issue that contributed in environment pollution. It was related to emission of carbon dioxide gases from 50% of chemical process and 40% burning fuel [2]. The carbon dioxide emission could cause greenhouse gases due to the increment of carbon dioxide gas that came from burning (1450 °C) of lime in cement production [2]. Hence, it was necessary to highlight this problem regarding to waste generates and air pollution as serious issue that need to be solve in order to protect the environment.

Fig. 1 - Mussel shell

Mussel shell is waste that is potential to be used in construction materials other than cement. It is due to the chemical composition of calcium carbonate ($CaCO_3$) in it were more compared to lime stone that been used in cement production [3]. The crystal structure of mussel shell generates large amount of calcite and aragonite which can enhance the strength and density of concrete compared to limestone ash [4]. This describe that mussel shell is a suitable material to be used as cement partial replacement in concrete since it has cementations properties similar to limes. Figure 2 shows the texture of mussel shell ash after undergo decomposition process.

In previous publications by Othman et al. [4] and Lertwattanaruk et al. [5], it states that mussel shell consists of high calcium carbonate content which had become the major component of oxide compared to other component in it. The addition of calcium carbonate in concrete mixture will enhance the existing calcium oxide to form calcium hydroxide (CH). Calcium hydroxide will increase the durability of concrete due to its properties which can act as filler to reduce porosity in concrete. Theoretically, concrete strength in term of compressive and tensile strength will increase when water absorption rate and porosity concrete were decline. Therefore, further research related to the utilization of mussel shell in term of its strength (compression and tensile) were compared to normal concrete in determination of its potential as partial replacement for cement in concrete mixture and at the same time reduce the quantity of waste generates in mussel production.

Fig. 2 - Mussel shell ash

2. Materials and Methods

2.1 Materials

The raw materials that in use in this study include water, coarse aggregate, fine aggregate, ordinary Portland cement and mussel shell. Ordinary Portland cements (OPC) are complies with specification base on BS EN 197-1: 2000 [6] with specific gravity of 3.11g/cm3 while 2.82 g/cm3 for mussel shell ash (10g sample of OPC and MSA). The production MSA were obtained by thermal decomposition process of mussel shell under 1000ºC for 1 hour. Then it were stored in room temperature for 24 hour. The physical properties (specific gravity, setting time, PSD) and chemical composition (XRF) of MSA ash were define in Table 1. The chemical composition of mussel shell were detected using X-ray Fluorescence (XRF) with specification based on ASTM E1621-13 model Bruker AXS S4 [7].

Table 1 - Chemical and physical properties of OPC and MSA as replacement for cementious materials

	Chemical compound	**OPC (%)**	**MSA (%)**	**Percentage total replacement (%)**		
				S3	**S5**	**S7**
Chemical compositi on	Na_2O	-	0	-	-	-
	Al_2O_3	3	-	3	3	3
	SiO_2	14.3	-	13	13	13
	SO3	3	-	4	3	3
	K2O	-	-	0	0	0
	CaO	57.2	71.5	58	57	57
	Fe_2O_3	2	0.14	3	2	2
	MgO	1	-	1	1	1
	SrO	-	0	-	-	-
	C	0	0	0	0	0
Physical propertie s	Specific gravity	3.11	2.82	-	-	-
	PSD:					
	10%	3.44	9.14	4.07	4.08	3.97
	50%	17.42	28.26	17.94	18.56	
	90%	35.86	43.97		18.14	
	Setting time (mins.):			34.36	37.7 37.61	
	Initial	9	8			
	Final	190	2	70	55	

- = not determined

According to Neville and Brooks, the specific gravity value of cement will affect the properties of concrete included it setting time [8]. Thus, this analysis was obtained in this research according to BS EN 196-3: 2005 [9] for ordinary Portland cement and mussel shell ash to identify the condition of mussel shell concrete on initial and final setting time (400g of OPC with S3, S5 and S7 partial cement replacement, 205mL water). The result show that early setting time for MSA paste (S3, S5 and S7) is faster (80 minutes) compared to ordinary Portland cement (90 minutes). It is due to the properties of mussel that consist higher rates of hydration to absorb water [10]. However, opposite situation occurs in final setting time which for MSA paste, it was 210 minutes which is slower compared to ordinary Portland cement (190 minutes). According to Yeonung et al. [11], the addition of calcium oxide will promote slower final setting time due to its performance developing in it' s ultimate strength.

The particle size distribution (PSD) of MSA according to BS ISO 13320:2009 using CILAS 1180 Liquid (0.04µm-2500µm). The rates of hydration are depending on its fineness which help in accelerates strength of concrete [13]. Thus, the higher fines of particle are needed to develop in accelerates strength of concrete. According to the Fig. 3, Portland cement has smaller size compared to mussel shell ash. At cumulative 10%, the size of Portland cement was 3.44 µm while for mussel shell ash were 9.14 µm. Based on 50% cumulative, Portland cements are 17.42 µm and MSA are 28.26 µm. While for 90%, it was 35.86 µm for Portland cement and 43.97 µm for mussel shell ash.

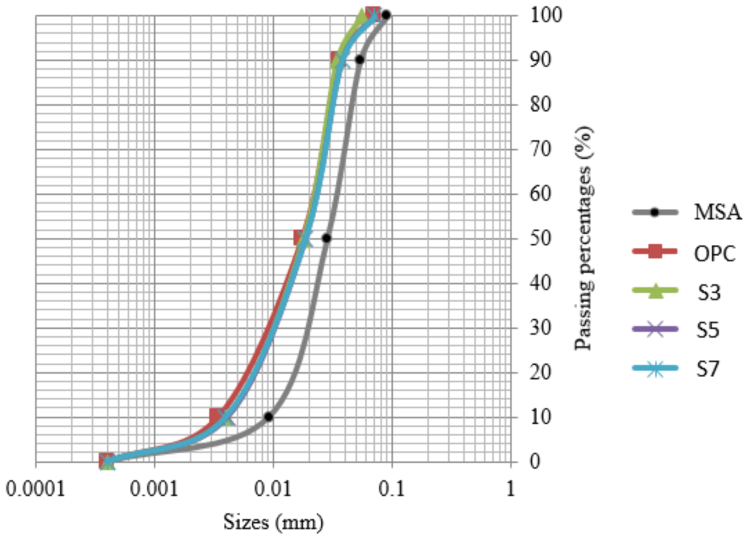
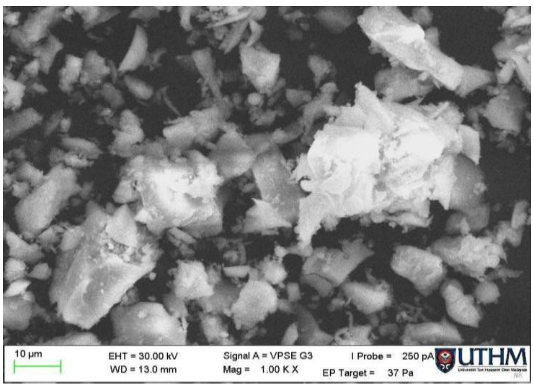
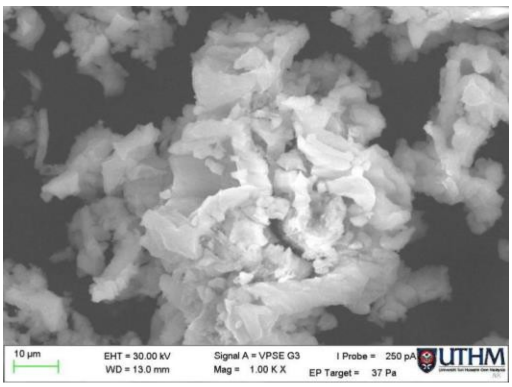


Fig. 3 - Particle size distribution of MSA, S3, S5, S7 and OPC

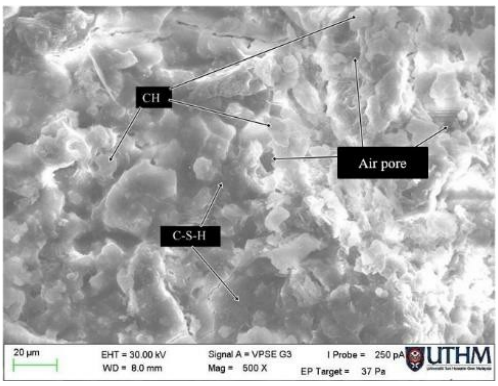
Microstructural observations such as SEM were performed on the MSA concrete specimens. It is to identify the potential influences of $CaCO_3$ particle as a filler and its formation in concrete specimens. The microstructure of the MSA samples was determined using an ASTM E2090-12 [14] method with magnification of 5 kX, 10 kX, 20 kX and 50 kX and electrical power of 3 kV and 5 kV [15]. The SEM sample of OPC, MSA and MSA concrete S0, S3, S5 and S7 were obtained base on 28 and 60 days curing. The demonstration of microstructural texture mussel shell concrete were obtained as in Fig. 4. According to SEM microstructural image obtained, there were similarity in shapes of OPC and MSA which both materials consist of cuboid and square forms. It is because of the high content of CaO in both substance. Nevertheless, there was a difference marked towards MSA and OPC position which the particle of OPC is usually not fully compacted between it spaces compared to MSA. These due to different mineral composition that contained in it. OPC consist of major component of is *alite*, *belite*, *aluminate* and *ferrite* produced from reaction between silica and calcium oxide while major mineral component for MSA is only calcium oxide which were produced from calcium carbonate reaction [15].



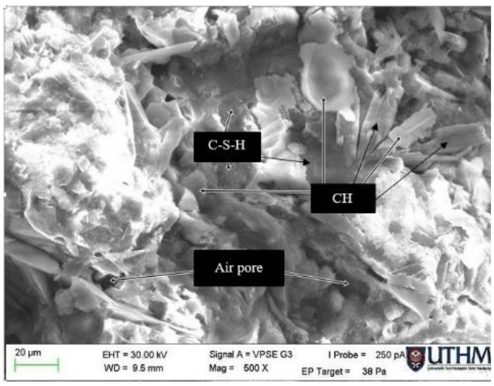
(a) SEM image of OPC



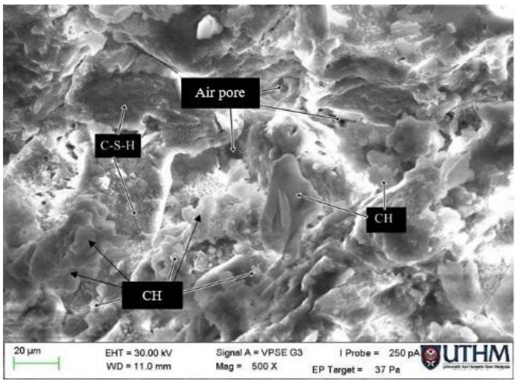
(b) SEM image of MSA



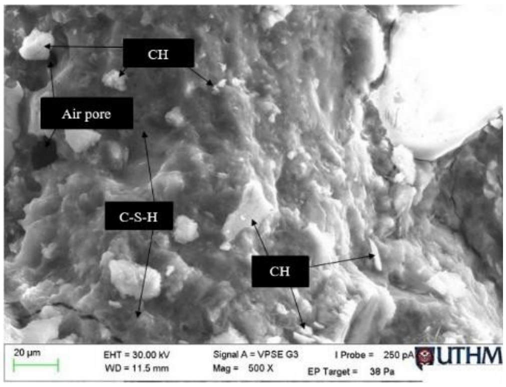
(c) S0 in 28 days curing



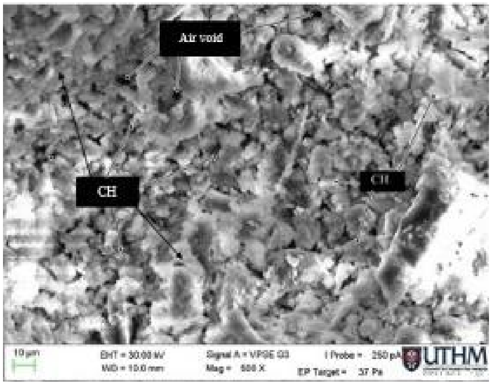
(d) S0 in 60 days curing



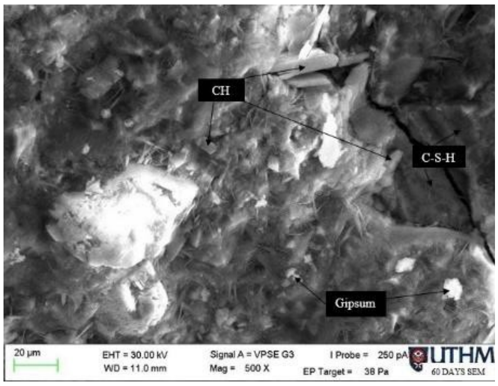
(e) S3 in 28 days curing



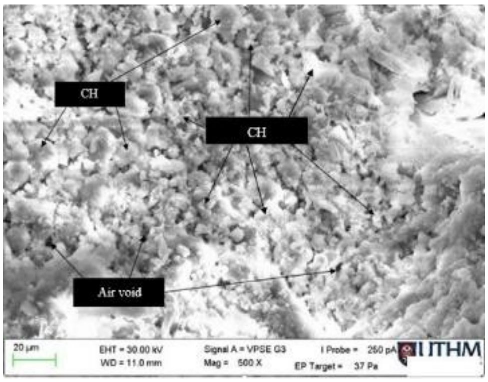
(f) S3 in 60 days curing



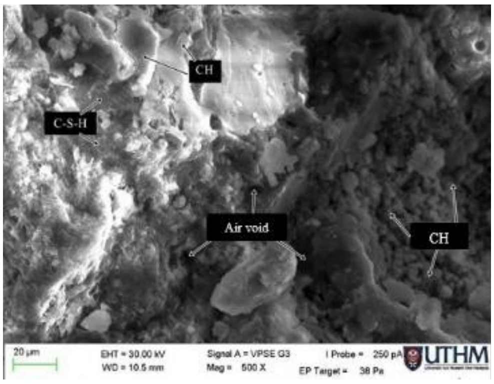
(g) S5 in 28 days curing



(h) S5 in 60 days curing



(i) S7 in 28 days curing



(j) S7 in 60 days curing

Fig. 4 - SEM analysis of S0, S3, S5 and S7 of MSA concrete base on 28 and 60 days curing

The different minerals presence in OPC cause the formation of various sizes and form particle. According to Hawlett [16], alite and belite average volume is about 15-20 µm similar to calcium oxide (CaO). However, alite crystal of OPC is square shaped and irregular form while belite consist of striped, perforated long and round in shape compared to MSA which ash is not too harsh, shaped sub-rounded, less porosity and average volume is 2.87 µm [5] [15]. Based on specimens SEM images, it's showed there were formation of calcium hydroxide (CH) and calcium silicate dehydrated (CSH) from chemical reaction of cements and water. There were also precipitation of air pore in concrete specimens in S0 in 28 days curing. In S0 for 60 days curing, its shows air pore had increased and formed in bigger sizes. It also detected formation of C-S-H and CH in it. However, for S3 in 28 and 60 days curing, the CH formation is increasing while air pore for the concrete specimens are decreasing.

Specimens for S5 in 28 days and 60 shows the air void is getting smaller and increments in CH value compared to S3. While for S7 in 28 and 60 days, the concrete specimens are reducing in porosity and increasing in CH value from S3 and S5. Specimens with MSA will increase the CH content which reduce the porosity in concrete. It is due to high CaO content in MSA which react in hydration process of minerals in cements to form CH. This coincide with Hargis, that additional calcium oxide in concrete will increased formation of CH which can enhance the durability concrete [17].

2.2 Mix Design

The test specimens were mixed from 4 separate (S0, S3, S5 and S7 MSA) batch of concrete. It was calculated and designed according to Design of Engineering (DoE) method with (w/c) 0.51 [18]. The weight of materials used for each specimens of water, fine and course aggregates were 0.21kg, 0.926kg and 0.822kg for cube and 0.41kg, 1.852kg and 1.644kg for cylinder. Whereas, replacement value of cement to ash materials (S3, S5, S7) 0.012kg, 0.02kg, 0.028kg with weight (OPC+MSA) of 0.402kg for 0.001m^3 mould and (S3, S5, S7) 0.024kg, 0.040kg, 0.056kg with weight (OPC+MSA) of 0.804kg for 0.002m^3 mould. Table 2 shows the details of the OPC and MSA mixtures used in the tests. According to DoE calculation, the target slump for S0 were follow the criteria between 60 - 180 mm (experimental value 86mm). However for S3, S5 and S7, the slump value were 75mm, 62mm and 51mm which define as less workability compared to S0. It happen due to high hydration rates of MSA in concrete which makes it's less workability [10]. The following describes briefly the procedure used for mixing included equipment used:

(i) All material (OPC, MSA, fine and course aggregates) weighed and wrapped to prevent from any moisture. Equipment like former mould and cone for slump test purposed were prepared and cleared before process mix concrete started.
(ii) Mould were cleaned (washed, brushed and dried) to prevent from any debris or other unwanted materials on it. Then, the mould were wiped with oil in thin layer before concrete mix poured to prevent the development of bond between specimens and mould.
(iii) OPC, MSA, fine and coarse aggregate were placed into the mixing drum and then add water accordingly to value from DoE method.
(iv) Concrete sample then carried out with workability test. Then, sample were poured in mould that been prepared before and imposed to impact of 25 times in each of 3 layer using rode.
(v) Samples were hardening in mould for 24 hours. Mortar and paste specimens were demoulded 24 hour after casting then placed in container filled with water for curing purposes according to 7, 28 and 60 days.

2.3 Compressive Strength Test

Compression test carried out to identify concrete strength with consistent rates of stress until it is failed. The tests were carried out on 100mm × 100mm × 100mm cube for concrete consist of S0, S3 and S7 MSA specimens, and performed after 7, 28 and 60 days curing. Compression test carried out refer on EN BS 12390-3:2009 [19]. Concrete strength was determined by distributing of maximum load imposed to cross sectional area of sample. The strength loss was also investigated by comparing with the compressive strength of MSA concrete specimens cured in different period with different percentage of MSA. Each data were collected and compared in histogram graph to differentiate the performance and its ability in gain straight of each specimens value.

2.4 Split Tensile Strength Test

Concrete is normally not specifically design to resist tension stress which were relates to concrete cracking. However, the absence of cracking is really necessary and very important to prevent corrosion of reinforcement or steel [20]. Thus, this test were applied in this paper to identify the development its strength in tensile by using MSA concrete (S0, S3, S5 and S7). This test was carried out according to EN BS 12390-6:2009 [21]. Split tensile tests were carried out using cylindrical shape with diametrical 100 mm and with height 200mm sizes. Some alertness should be considered during splitting test which the platens of machine should be fixed in a plane perpendicular to axis and movement on vertical plane also should be permitted. It is to prevent of some non-parallelism of generatrices cylinder specimen that can affect the accuracy of data collection.

Table 2 - Details of cement replacement in mixtures

Code	Percentage replacement	w/cm
MSA (S0)	100% cement	0.51
MSA (S3)	97% cement + MSA 3%	0.51
MSA (S5)	95% cement + MSA 5%	0.51
MSA (S7)	93% cement + MSA 7%	0.51

2.5 Correlation Analysis

Correlation test in this study was applied according to Excel Microsoft programmer 2016. Correlation is a statistical method used to assess parallel equation between two continuous variables [22]. Correlation may be eliminated definition as reciprocal relationship between two or more different case and it in between range -1 (negative paragon correlation) through 0 (no correlation) until +1 (perfect positive correlation). In statistics term, correlation is method evaluate possibility bilateral linear equation between two continuous variables [23]. The correlation was measured through statistical method named correlation coefficient, where its represents linear equation strength between variable. The closer the correlation coefficient towards ±1, the stronger correlation value. If correlation coefficient value are positives, all variables is directly related. On the other hand, if the correlation coefficient value are negatives, all variables are inversely related [24]. For the correlation coefficient value, it can be categorize according to Table 3 which will be refer as guideline to define its value, r [25].

Table 3 - Strength of correlation coefficient value

Correlation Coefficient Size (r)	Define
0.9 to 1.00 or -0.9 to -1.0	Very strong
0.7 to 0.9 or -0.7 to -0.9	Strong
0.5 to 0.7 or -0.5 to -0.7	Moderate
0.3 to 0.5 or -0.3 to -0.5	Weak
0.01 to 0.3 or -0.01 to -0.3	Very weak
0.00	No

3. Results and Discussion

3.1 Compressive Strength

Compressive strength is one of measurement that determined concrete ability in withstand loading that will reflect on its quality. The specimens treated in a standard manner which includes full compaction and wet curing for specific period of time which representing its potential quality [20]. The results for compressive strength development of 0.001m3 cube specimens that cured in tap water at the ages of 7, 28 and 60 days that are summarized according in Table 4.

Table 4 - Compressive strength value with different MSA percentage specimens and curing duration.

Percentage of partial replacement (%)	Compressive strength (MPa)		
	Curing duration (Days)		
	7	28	60
S	35.2	43	46
S	29.9	38	42
S	31.0	39	39
S	30.4	35	36

According to data obtained, these clearly shows that the decreases of strength values as the replacement level of MSA filler increased. The compressive strength value in 28 days for S0 indicates the highest value 43.5MPa compared to S3 (38.8 MPa), S5 (39.1 MPa) and S7 (35.9 MPa). Nevertheless, all MSA concrete specimens had achieved beyond the target strength according to DoE (35 MPa) in 28 days. Based on MSA concrete specimens, the higher strength value goes to S5 (39.1 MPa) and for 60 days curing was pointed to S3 replacements (42.2MPa). Fig. 5 shows the pattern of graph plotted obtained from compression strength against curing duration. From the graph, concrete with MSA mixture recorded decline compared to plain concrete (S0) in preservation age of 60 days. In short, this case happened due to the formation of 4 special minerals of chemical composition in cement namely *alite*, *belite*, *tricalcium aluminate* and *tetracalcium aluminoferrite*. When cement replaced with material like MSA, it will reduced the amount of *alite* and belite in concrete which resultant least formation of CSH in it. This cause declination of concrete strength. Thus, it shows that MSA should be applied in small quantity in order to produce an achieving goals in term of its durability and its strength.

3.2 Split tensile strength

Tensile strength one of major parameter in analyses concrete strength in this research towards cracking value. Base on Table 5, data obtained for concrete with MSA in tensile strength indicates higher value compared normal concrete (S0). In 7 days curing, it's showed that S5 records the highest tensile strength opposed S0, S3 and S7 which is 2.52 MPa. Meanwhile, for concrete with 28 and 60 days curing, S7 and S3 indicates the higher tensile strength of 3.02 MPa and 3.19 MPa which proved that MSA can improved split tensile strength (cracking) of specimens. According to

American Concrete Institute [26], tensile strength are 10% of compressive strength. Similar to compressive strength, the optimum percentage of MSA must not exceed 5% because excessive MSA could decrease the compressive strength which could effect on tensile strength of specimens. Thus, it proved that the percentages of MSA should be limit so that it could enhance its tensile strength.

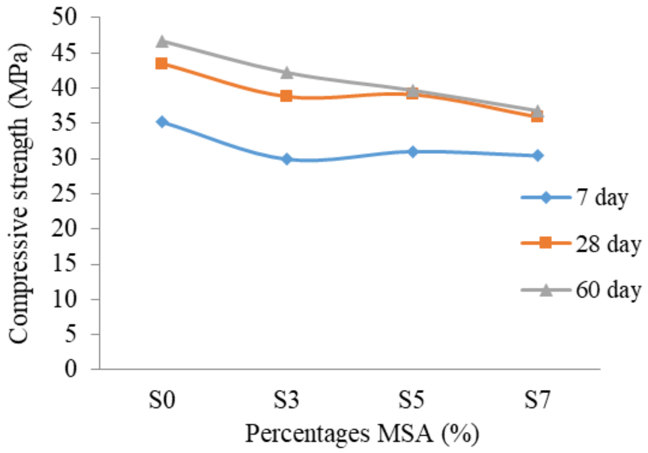


Fig. 5 - Numerical data showing the compressive strength of MSA concrete with different percentage value plotted against curing time.

Table 5 - Split tensile strength value of MSA percentage with different curing times

Percentage of partial replacement (%)	Tensile strength (MPa)		
	Curing duration (Days)		
	7	28	60
S0	2.46	2.57	2.90
S3	2.48	2.86	3.19
S5	2.52	2.44	3.02
S7	2.11	3.02	2.76

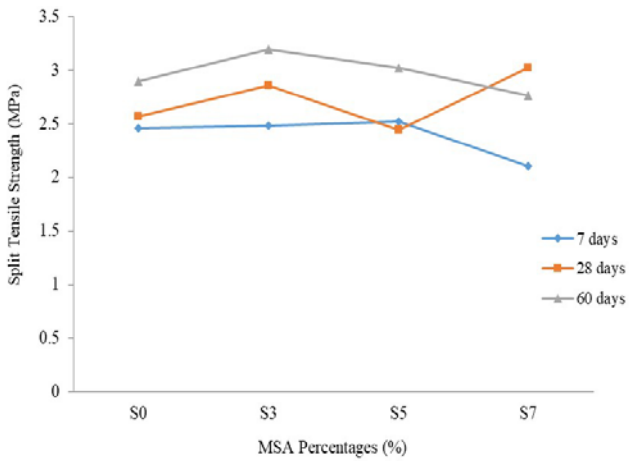


Fig. 6 - Split tensile strength of MSA concrete with different curing time

3.3 Correlation Analysis

The optimum percentage of MSA are obtained to identify the strength of MSA concrete according to data collection from mechanical characteristic recorded. The data used in correlation analysis are to determine coefficient value between two different variables. This correlation are focused on the relationship between compressive strength and split tensile strength of MSA concrete. Fig. 7 indicates the relationship between compressive and split tensile strength of 7, 28 and 60 days of curing.

The graph shows that the compressive and split tensile directly proportional to the curing duration similar to increases of split tensile strength to the compression strength value which support the statements of previous research [4]. Table 6 indicates correlation coefficient between compressive and split tensile strength with different MSA on 7, 28 and 60 days curing. The correlation coefficient value of S0 is 0.75 whereas for S3 is 0.96. MSA of S5 and S7 are 0.74 and also 0.84. According to correlation coefficient data's of MSA, S3 represents as a very strong correlation. While for S0, S5 and S7 were in strong category ($0.70 \leq r \leq 0.90$ or $-0.70 \leq r \leq -0.90$). According to the mechanical properties of concrete specimen's determination, it was identified that MSA can contributed in generates strength and durability of concrete especially with S3 replacements. The correlation value for S3 given a very strong and consistent correlation compared to S0, S5 and S7 replacements. All results research showed that concrete with mussel shell ash mixture produce better result compared to controlling concrete in preservation age at the end of 60 days.

Table 6 - Correlation coefficient between compressive and split tensile strength

MSA	Independent nt variable, x	Depende	Consta		r
			M	c	
S0			22.3	-17.3	0.75
S3	Compressiv strength	Split tensile strength	17.5	-12.7	0.96
S5			9	9	0.74
S7			6	16.6	0.84

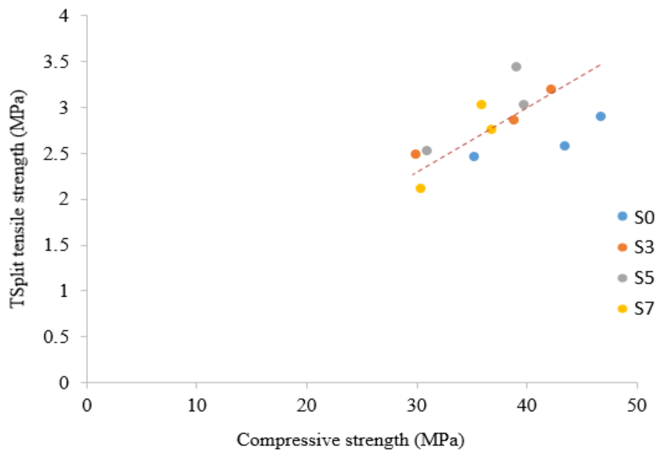


Fig. 7 - Relationship between compressive strength and split tensile strength on 7, 28 and 60 days curing

4. Summary

MSA is one of materials that containing higher calcium oxide (71.5%) compared to OPC (57.2%). It played an important roles in hydration reaction process of hardening concrete which generates the formation of CH that can reduce porosity (more compacted concrete) and increased the strength, durability of concrete specimens. The specific gravity of MSA was 2.82 g/cm3 which is smaller than OPC. Based on its sizes, MSA can be categorizes similar to cements sizes which was 9 µm until 43 µm. Thus, it was potentially can be applied in concrete mixture since consist similar characteristic as cements.

According to the data obtained, the increments percentages of MSA replacements with cements could decrease the compressive strength of specimen due to loses of some minerals in cement [17]. However, the total specimens had achieved the target strength which was 35MPa on 28 days curing. Meanwhile, split tensile strength with S3 MSA was giving positive feedback which increase abruptly compared to S0. At the 60 days curing, S3 also indicates the highest value compared to S0, S5 and S7. Based on correlation analysis, it showed that S3 indicates very strong correlation coefficients ($r > 0.9$) between the compressive and split tensile strength. Thus, it can be conclude that the utilization of 3% MSA as cement partial replacement is optimum which can enhance in tensile strength compared to normal concrete.

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