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Strength and Sorptivity of Eco-Processed Pozzolan Concrete under Chloride and Sulphate Exposure

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Abstract: *Concrete is one of the most frequently used building material an account of its good compressive strength and durability. However, severe aggressive environmental conditions could lead to deterioration of concrete. Therefore, this study focused the impact of Eco-Processed Pozzolan (EPP) on the compressive strength and sorptivity of concrete exposed to chloride and sulphate environment. EPP is a new supplementary cementitious material produced from the extraction of Spent Bleaching Earth (SBE) in the palm oil refineries. In this study, the Ordinary Portland Cement (OPC) was partially replaced with 10%, 20% and 30% of EPP by weight of cement with water to binder ratio of 0.45. The resistance of EPP concretes towards chloride and sulphate attack was investigated by placing concrete cubes in 3.5% Sodium Chloride (NaCl) and 3.0% Sodium Sulphate (Na2SO4) solution and tested for compressive strength and sorptivity of the concretes. The results show that partial replacement of cement with EPP give lower loss of strength of concrete under the chloride and sulphate exposures. The shorter time taken for the capillary suction resulted in higher sorptivity value in comparison with the EPP concrete. The present investigations revealed that incorporation EPP has significantly improved the strength and sorptivity characteristics of concrete.*

Keywords: Eco-processed pozzolan, Supplementary cementitious material, Compressive strength, Sorptivity, Chloride attack, Sulphate attack

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1. Introduction

Concrete structures constructed with Ordinary Portland Cement (OPC) tend to degrade much faster when exposed to the aggressive environmental conditions such as marine structure, underground structure, and wastewater treatment plants structures (Marangu, 2013). Therefore, many industrial waste materials like Fly ash (Vinod et al., 2016; M.S Krishna, Siva & KJB Chari, 2017), Ground granulated blast furnace (Rathod et al., 2018; Parron et al., 2019) and Palm oil fuel ash (Jonida et al., 2018; Liyana et al., 2015) have been used widely as cement replacement. These supplementary cement materials (SCMs) possess pozzolanic properties which mainly consist of high amount of silica which play a crucial role when incorporated in the OPC at a certain proportion.

The use of SCMs in concrete manages to improve the compressive strength (Mallikarjun, Kashinath & Prakash, 2015) and durability of concrete (V. Gopi & K. Shyam, 2019). The SCMs are able to diminish the permeability of concrete. Hence, a substantial resistance against the reinforcement corrosion, sulphate attack and acid attack can be seen (M.A. Adole et al., 2012). The sorptivity test would be more appropriate to examine the durability characteristics of concrete. In addition, the pozzolanic activities reduce the porosity and increase the density. As a consequence, the chemical durability of concrete to aggressive environment increases.

EPP material is currently used in producing blended cement. It is a solid waste material extracted from the waste product of crude palm oil degumming and bleaching process from refinery plants (Raihana et al., 2019). However, there is limited information on the effect of EPP in improving the durability of concrete and the strength-permeability relationship of EPP concretes when exposed to chloride and sulphate exposure. Based on the laboratory test conducted, replacement of EPP that less than 30% show a better performance properties compared to control foamed concrete (Nazrin, 2016). Meanwhile, the addition of EPP in concrete was absorbed to increase the weight of the concrete while immersed in chloride and sulphate solution. The control concrete shows highest weight gained due to solution absorption and expansion occurred by gypsum formation (Elffie Y. et al., 2020).

The aims of this research are to investigate the compressive strength and examine the porosity of the concrete containing EPP as SCMs under chloride and sulphate exposure. Therefore, in this study 10%, 20% and 30% of EPP were used to partially replace cement. In addition, concretes were immersed under 3.5% NaCl and 3.0% Na_2SO_4 solutions for an exposure period up to 28 days.

2. Materials and Methodology

Materials

The type of cement used in this research was the Ordinary Portland Cement (OPC), CEM 1 442.5N obtained from cement industries (SABAH) Sdn Bhd, which meet requirement of Malaysia Standard MS 522 and BS EN 197-1. The Eco-processed pozzolan (EPP) was collected from the Eco Oils Sdn Bhd plant in Lahad Datu, Sabah, Malaysia.

The original EPP was observed to have coarser and porous particles as shown in Figure 1. The morphology of EPP was further analyzed by using Scanning electron microscope (SEM). The SEM image shows that EPP has irregular, sharp, rough and porous structure Figure 2.

Figure 1: Eco-processed pozzolan

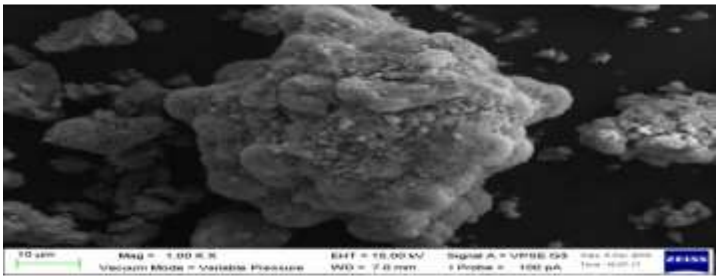

Figure 2: SEM of Eco Process Pozzolan

The chemical composition of EPP and OPC are shown in Tables 1. The main component of EPP is SiO2 at 51.05% and the total combined amount of SiO2, Al2O3 and Fe2O3 is 69%. Hence, the EPP can be classified as Class C pozzolan (ASTM C618). According to ASTM C618, Class C pozzolan commonly produced from lignite or subbituminous coal and is having pozzolanic and some cementitious properties.

Table 1: Chemical Composition of OPC and EPP

Oxides (%)	OPC (Hidayati et al., 2017)	EPP (Elffie et al., 2020)
SiO2	13.8	51.05
Al2O3	3.34	12.32
Fe2O3	3.83	5.86
CaO	56.89	13.32
MgO	1.88	5.70
SO3	3.51	3.65
LOI	0.44	2.7

Mix Proportions

Four (4) types of concrete mixes were prepared, which were the mix with 100% OPC (E0), 90% OPC and 10% EPP (E10), mix with 80% OPC and 20% EPP (E20) and mix with 70% OPC and 30% EPP (E30). The fine aggregate was sieved using 4.7 mm sieve, meanwhile the coarse aggregate with a maximum size 20 mm was used. The mix was designed using the JKR method (Ministry of Works, 2005) and detail mixes are provided in Table 2.

Table 2: Concrete Mix Design (kg/m^3)

Description	Notation	Replacement (%)	Cement	EPP	Fine Aggregate	Coarse Aggregate	Water
Control mix (E0)	E0	-	430	-	516	1650	193.5
Concrete mix with 10% EPP	E10	10	387	43	516	1650	193.5
Concrete mix with 20% EPP	E20	20	344	86	516	1650	193.5
Concrete mix 30% EPP	E30	30	301	129	516	1650	193.5

Casting and Curing

Concrete cubes size of 100 x 100 x 100 mm were casted and cured in a water curing tank for 28 days to gain the design strength. After that, the cubes were shifted in each solution of 3.5% NaCl and 3% Na_2SO_4 to assess the compressive strength at 7, 14 and 28 days. The slump test was used in accessing the workability of the fresh concrete mixes with EPP according to ASTM C143C. The cube compressive strength was obtained in accordance with ASTM C39. While, the sorptivity test conducted in compliance with ASTM C1585-04.

The concrete samples were immersed in water as shown in Figure 3 with water level not more than 2 mm above the samples support. The samples were sealed on all four sides and a loose plastic sheet was used to cover one end of the sample to prevent from water loss due to evaporation. Lastly, the samples were removed from water and wiped off with a paper towel and determined the water absorbed.

Figure 3: Specimens immersed in water

Sorptivity (S) is a material property which indicates the tendency of a porous material to absorb and transmit water by capillary. The water absorption values, i, can be expressed by

$$I = \Delta m / AP \qquad (1)$$

Where Δm = Cumulative change in mass because of water absorption
A = Cross-sectional area of test specimens, mm^2
P = Density of water

There is relation of the form exists shown by Hall C. (1989)

$$i = St^{1/2} \text{ (Darcy's Law)}$$

Where S = Sorptivity in mm/√min (mm/√min = 1.29×10^{-04} m/√s) and
t = time in minutes

Therefore, the cumulative values were used to plot a graph against the square root of the times and sorptivity.

Workability

Workability of a freshly mixed concrete is evaluated at early stage to determine its fresh properties (Vinay et al., 2015) and is measured by conducting a slump test. The control mix was designed in accordance to JKR Method (Ministry of Works, 2005) with fixed water to binder ratio of 0.45 adopted for all mixes. A total of four (4) concrete mixes were prepared and tested for the workability. Table 3 shows the slump test results for all concrete mixes.

From the findings in Table 3, it could be seen that all EPP concrete mixes exhibit lower value of slump in contrast to slump of control mix. It was also observed that among the EPP mix results, the higher the replacement of cement, the lower the slump. This possibly due to EPP has irregular, sharp, rough and porous surface, thus the specific surface area is suspected to be high and more water was required to wet the surfaces.

Meanwhile, Raihana et al. (2019) reported that EPP has larger of mean particle size. On the other hand, its porous structure caused more water was being absorbed by the EPP concrete compared to the control concrete. Hence, higher replacement of cement would cause the slump value to reduce. The same results were reported for other SCMs having irregular, rough and porous surfaces which have high specific surface area (Metha & P.K, 1992; Hwang & Wu, 1989) and lead to decrease in slump (Zhang & Malhotra, 1996; Ganesan, Rajagopal & Thangavel, 2008; Rafieizonooz et al., 2016).

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Table 3: Workability of Concrete

Concrete Mix	Slump Value (mm)	Remarks
Control Mix (E0)	52	-
Mix with 10% EPP (E10)	36	30% reduction
Mix with 20% EPP (E20)	23	55% reduction
Mix with 30% EPP (E30)	17	67% reduction

Compressive Strength

The compressive strength result for all concrete samples under normal curing is presented in Figure 4. Analyzing the results of compressive strength, the results demonstrated that concrete with 10% EPP was higher than the 20% (E20) and 30% (E30) of EPP concretes at early age. This is due to the high amount of Portland cement (90%) in EPP concrete which causes higher hydration reaction at the early age than E20 and E30 concretes. Meanwhile, compressive strength of E10 and E20 concrete were higher than E0 concrete. According to Osei & Jackson (2012), the concrete derives its strength from the pozzolanic reaction between silica in pozzolana and the calcium hydroxide acquitted during the hydration of OPC.

After 28 days of curing, the compressive strength of E20 was 34.2 MPa, which was higher than E0, E10 and E30 with 30.13 MPa, 32.10 MPa and 26.72 MPa, respectively. This can be explained by the fact that the strength and durability of concrete is affected by the presence of Ca (OH_2) which is it is visible that concrete with EPP has higher strength compare to control concrete at 28 days. This demonstrated that the strength and durability of concrete is affected in accordance with the presence of Ca (OH_2) which is water soluble (Osama, 2018). EPP is expected to bind the available Ca (OH_2) and produces more of the stable gel C-S-H that is liable for the strength development. The lowest strength was shown by E30 at all ages of testing. This could be attributed by few amount of total C-S-H gel produced by E30. Lower cement content causes lesser hydration process, thus resulting in generation of lower amount of calcium hydroxide to be used in pozzolanic reaction for formation of C-S-H gel (Khairunisa et al., 2015). The increase amount of cement replacement formed more C-S-H that leads to supply Ca (OH_2) become too small to be bound by the available SCM. Meanwhile, Wankhede & Fulari (2014) reported the effects of fly ash on the properties of concrete and concluded that the compressive strength was increased with 10% and 20% of cement replacement and 30% of cement replacement caused decrease in strength. According to Abdul & Basid (2017), they conclude that 30% replacement of cement is the optimum amount to achieve the higher strength. The current study also show that control concrete has high strength compare to E30.

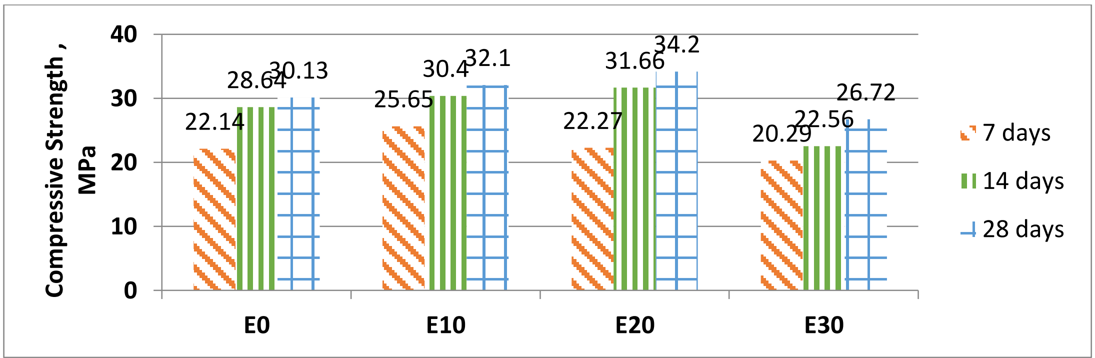

Figure 4: Compressive strength Result

Compressive Strength of EPP Concrete under Chloride Exposure

The compressive strength of concrete is provided in Table 4 and presented on Figure 5. In this study, the compressive strength was discovered after concrete specimens were immersed in 3.5% of sodium chloride solution at the age of 7, 14 and 28 days. It was noted that E0 has the highest strength loss at 28 days. Although, E0 possessed the highest compressive strength at early stage but it was only be able to retain 73.24% of its early strength. This indicates that E0 was affected the most under chloride exposure.

Incorporation of EPP has reduced the strength loss of concrete from 26.76% to 17.79%, 20.02% and 20.90% for E10, E20 and E30, respectively. This reduction is possibly due to the pozzolanic reaction occurred within the EPP concrete has led to the improvement of its microstructures, thus permeability of the EPP is reduced. J.Bai, S.Wild & B.B Sabir (2002) previously reported that SCM in concrete has led to the development of a finer pore structure, which reduces the rate of diffusion of aggressive solution into the concrete.

Furthermore, CH consumption reduced the chloride attack within the concrete (A.Ghazy, M.T & Bassuoni, 2017). It was also noticed that the higher the EPP replacement level, the higher the strength loss of the EPP concrete. E30 concrete has the highest strength loss of 20.90% compared to all EPP concretes.

Meanwhile, E10 concrete showed minimal changes in strength loss with less than 20%. There is slow improvement on the strength of E30 at 28-days possibly due to its slow pozzolanic reactivity. Hence, exposure of E30 in chloride solution has increased the damage. Perhaps, if the initial water curing of E20 and E30 was done in longer duration, the effect of EPP might improve. Nevertheless, all concrete samples have shown a declining in compressive strength trend. The reason which caused decrement in strength may be chlorides are known to develop the formation of porous C-S-H involving complex reaction (Lee et al., 2000). Hence, the leaching of calcium hydroxide and the formation of porous C-S-H attributed to concrete deterioration.

Table 4: Compressive Strength Results for 3.5% Sodium Chloride Exposure (MPa)

Mix	7-days	14-days	28-days	Loss (%)
E0	28.85	26.49	21.13	26.76
E10	28.81	27.79	23.74	17.79
E20	28.47	23.56	22.77	20.02
E30	27.89	26.21	22.06	20.90

Figure 5: Compressive strength of EPP Concretes under the sodium chloride immersion

Compressive Strength of EPP Concrete under Sulphate Exposure

From Table 5 and Figure 6, it can be seen that the compressive strength of all concrete specimens have shown a similar trend as concrete specimens exposed to chloride environment. E0 concrete has the highest loss in strength compared to the other concrete mix that is 22.42%. Formation of gypsum and ettringite within the concrete has led to the increase in the volume changes (Elffie Y. et al., 2020) which finally led to the compressive strength reduction. Similarly, consumption of EPP in concrete reduced the strength loss about 17.55% - 14.15%, possibly due to dilution of the tricalcium aluminate (C3A) which is susceptible to the sulphate attack.

Although the trend is similar, it seems that the percentage loss of concrete in sulphate solution is much lower than exposed in chloride solution. E30, E20 and E10 concretes show strength loss of 8.27%, 6.38% and 4.87% respectively. Again, the strength loss of E30 is higher than the E10 and E20 concrete. The strength loss may relate to the chemical reaction between sulphate solutions and cement hydration product. Ettringite and gypsum were to be found at later age. Ettringite was reported to caused expansion resulting to the failure of concrete specimens (Metha, 1983: Cohen, 1983; Odler & Gasser, 1988).

Table 5: Compressive Strength Results for 3% Sodium Sulphate Exposure (MPa)

Mix	Normal water curing	3.0% Sodium sulphate solution			Loss (%)
	28-days	7-days	14-days	28-days	
E0	30.13	28.63	26.09	22.21	22.42
E10	32.10	28.57	27.93	27.18	4.87
E20	34.20	28.35	27.88	26.54	6.38
E30	26.72	28.04	27.79	25.72	8.27

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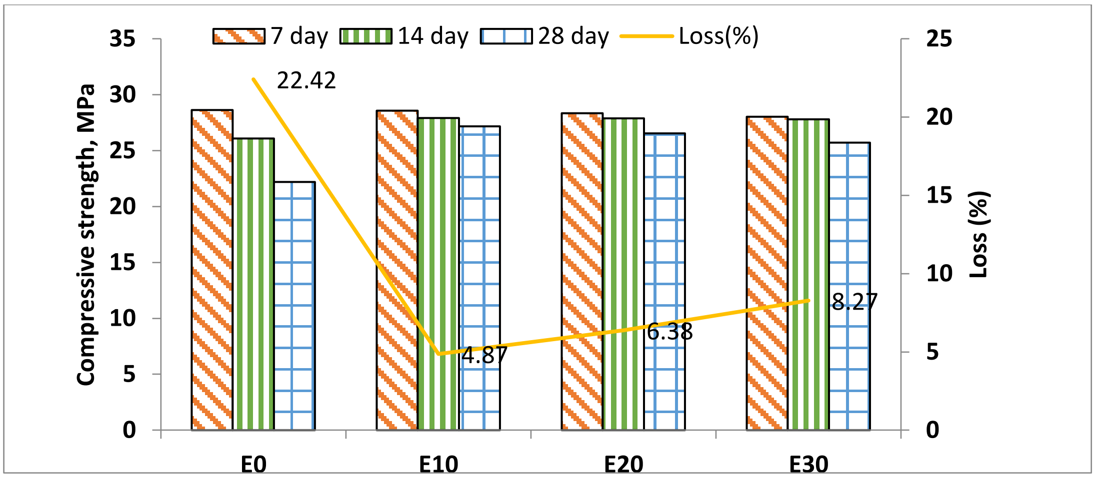

Figure 6: Compressive strength of EPP Concretes under sodium sulphate immersion

Sorptivity of EPP Concrete under Aggressive Exposure

The results of sorptivity test are given in Figure 7 and Figure 8 in accordance with curing condition and concrete mixes at 28-days. EPP concrete specimens give the lower sorptivity values compared to OPC concretes under aggressive exposure condition. The highest sorptivity values of 4.06 mm/s$^{1/2}$ and 3.01 mm/s$^{1/2}$ were obtained from OPC concrete cured in chloride and sulphate solution, respectively. Whilst, the lowest sorptivity values of 1.58 mm/s$^{1/2}$ and 1.935 mm/s$^{1/2}$ were obtained from E20 concrete. This indicated that the time taken for the water rise by capillary action in EPP concrete are longer, thus proved that these concrete are less porous compared to the OPC concrete. The possible reason for this is that the addition of EPP to concrete reduced the average pore radius of concrete by blocking the large voids. This pozzolanic reaction and finer particles of EPP fitting in between cement particles. The findings obtained from the study are in accordance with Speare at al. (1999). Meanwhile, since EPP is fine (Raihana et al., 2019), pores in the bulk paste or in the interfaces between aggregate and cement paste is filled by EPP, thus the capillary pores are reduced.

Kartini et al. (2010) reported that by replacing cement with pozzolan may attribute to densification of pore structure, thus reducing the permeability because of the formation of secondary C-S-H. The current study also shows that E20 concrete has the lowest sorptivity values when exposed in chloride and sulphate solution. E20 concrete probably has the highest amount C-S-H gel formed, which is also shown by its higher compressive strength (Figure 5 and Figure 6). The strength loss would decrease when sorptivity is low for both EPP concretes under chloride and sulphate exposure. This can be explained by pore structure has been refined which is mainly caused by the formation of the secondary C-S-H gel issued from the pozzolanic reaction (Aylin A., 2018). The EPP pozzolan also reduced the average pore radius of concrete by blocking the large void (pore discontinuity) in the hydrated cement paste through pozzolanic reaction and finer particles of pozzolan possibly fitting in between cement particle. The results are in line with Speare et al. (1999) and Nusret B. & Salih Y. (2010). Osei & Jackson (2012) reported that strength of concrete at both low and high percentage replacement is low which representing lesser amount of C-S-H gel formed. Meanwhile, E10 and E30 concretes show that the sorptivity is higher compare to the E20 concrete. On the other hand, E.Guneyisi, M.Gesogiu & K.Mermerdas (2008) stated that concrete produced using 20% Metakaolin replacement has the most impermeable structures.

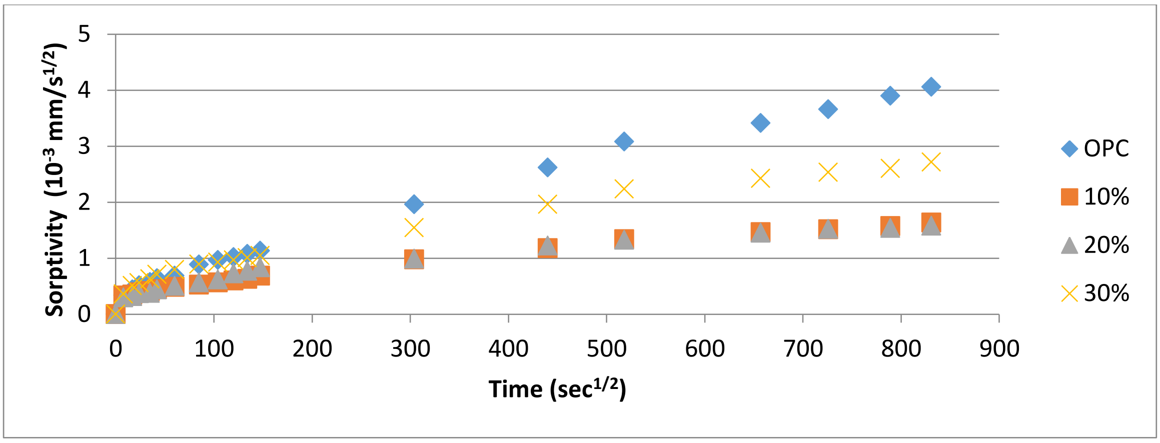

Figure 7: Sorptivity of sodium chloride immersion

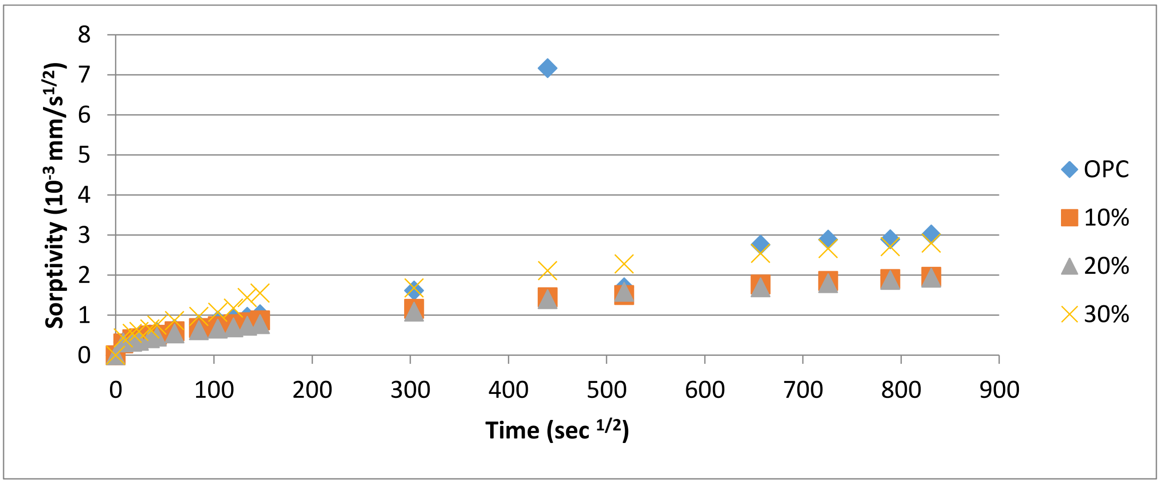

Figure 8: Sorptivity of sodium sulphate immersion

Relationship between Porosity and Compressive Strength

Figure 9 and Figure 10 give a comparison between sorptivity and compressive strength of concrete samples. In general, a good relationship is observed between the sorptivity and the strength values when exposed to aggressive environment. Therefore, as the strength of concretes increased due to hydration, the sorptivity reduced substantially denoting a denser microstructure.

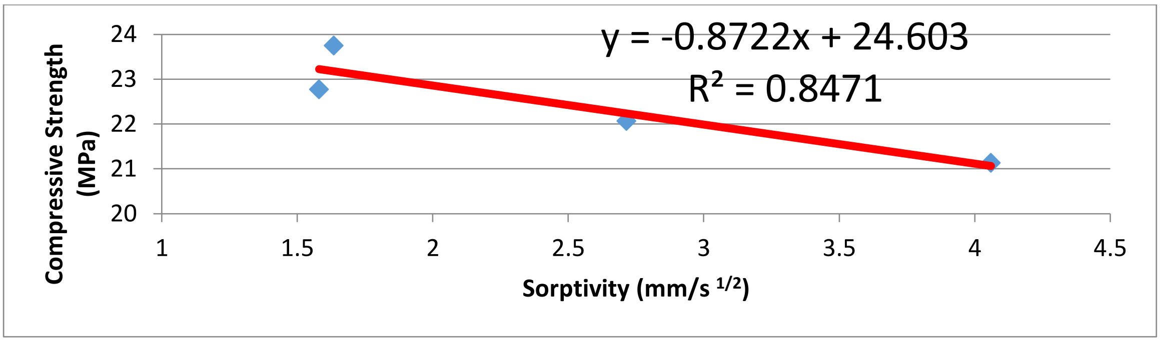

Figure 9: Sorptivity versus compressive strength under chloride immersion

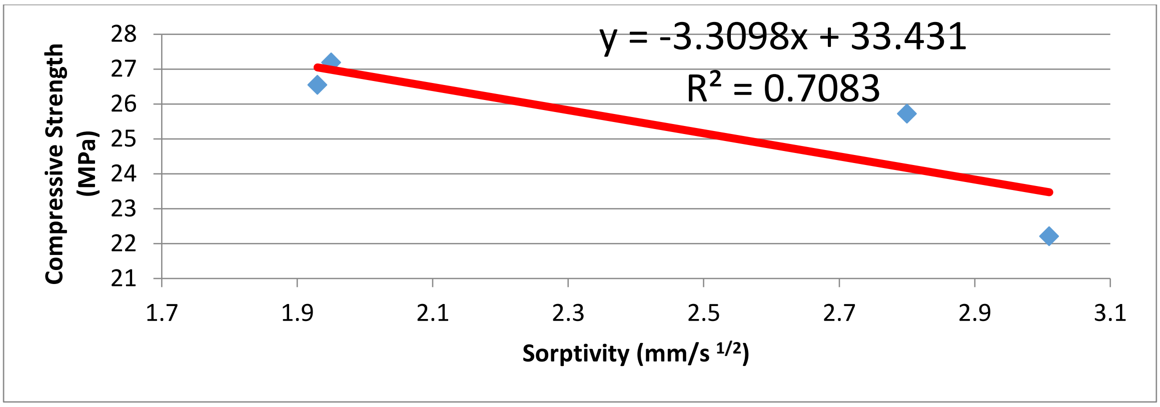

Figure 10: Sorptivity versus compressive strength under sulphate immersion

Conclusions

Based on the experimental outcome, the following conclusions were made:

i. The compressive strength performance of EPP concrete is higher than OPC concrete when cured in water at age of 28 days. The highest compressive was observed in 20% of cement replacement by EPP.
ii. EPP improves the durability of concrete as compared to OPC concrete when exposed to the sodium chloride and sodium sulphate solution. All EPP concretes showed lower strength loss compared to the OPC concrete. These results emphasized the beneficial effect of incorporating EPP as partial cement replacement in the concrete.
iii. The OPC concrete was more permeable than EPP concretes when immersed in aggressive environment. The EPP reduced the permeability of the concrete thus caused reduction in the porosity of the concrete.
iv. A good relationship was observed between the porosity and the strength of the concrete. As the strength of concrete increased the sorptivity reduced.

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