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COMPRESSIVE STRENGTH AND DURABILITY OF CONCRETE WITH COCONUT SHELL ASH AS CEMENT REPLACEMENT

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ABSTRACT: The durability of concrete is the ability to withstand induced damages over a long period of time. It can be measured by means of sorptivity or rate of water absorption and resistance to sulfate attack (RSA). Concrete with larger voids is more susceptible to deterioration through absorption of chemicals, such as sulfate found in soil and seawater, which forms gypsum and ettringite. Gypsum and ettringite cause reduction of strength, cracking, and expansion of concrete. To improve the durability of concrete exposed to sulfate, the National Structural Code of the Philippines (NSCP) suggests increasing the strength of concrete to 31 MPa, which would require more cement. This study aims to investigate the effects of partially replacing cement with coconut shell ash (CSA) in its compressive strength, sorptivity, and RSA. CSA in this study was classified as a Class N pozzolan, a cementitious material that makes a cheaper substitute for ordinary Portland cement (OPC). Results showed that the sorptivity of all concrete with CSA is within the acceptable limit. Based on the relationship of compressive strength with the CSA content, the optimum percentage replacement of OPC with CSA is 10%. From statistical analysis, it was determined that there is no significant difference in the compressive strength and expansion of concrete between the conventional and 10% CSA concrete. The strength of 10% CSA concrete is 92.10% of the strength of conventional concrete. The study reveals that CSA at 10% cement replacement is an effective pozzolan, which neither compromises the compressive strength and RSA of concrete.

Keywords: Coconut Shell Ash, Compressive Strength, Sorptivity, Resistance to Sulfate Attack

1. INTRODUCTION

The durability of concrete is the ability to withstand induced damages over a long period of time. There are multiple parameters in which the durability of concrete can be measured, two of which are sorptivity and resistance to sulfate attack (RSA). Sorptivity, or rate of water absorption, is when water undergoes capillary suction within the pore spaces of concrete. Durability is highly dependent on the capacity of concrete microstructure to absorb water [1]. This implies that concrete with greater voids between particles is highly susceptible to chemicals, such as sulfate ions. Sulfate ions that penetrate the concrete and incorporated with free lime and alumina would yield gypsum and ettringite, which causes expansion and deterioration of concrete. Most soils in the ground contain the element sulfate in different forms [2]. Since most of the concrete structures, like foundations, are exposed to the soil in its entire lifespan, protection against sulfate attack is important to ensure that reduction of strength, cracking, and expansion of the concrete does not occur especially on concrete exposed to soil with high salinity level. To improve the durability of concrete exposed to sulfate, the National Structural Code of the Philippines (NSCP) suggests increasing the strength of concrete to 31 MPa, which would require a smaller water-cement ratio. Hence, more ordinary Portland cement (OPC) is required, increasing the cost of concrete. A lower water-cement ratio leads to a decrease in total porosity and pore median of concrete [3]. Thus, decreasing water-cement ratio decreases voids and increases strength and durability due to less water penetration but increases also the cost of concrete. One way to lessen the cost of concrete without sacrificing the strength or durability is to partially replace OPC with a cheaper cementitious substitute. Several studies make use of waste materials that contain pozzolanic property as a substitute for cement in concrete. Fly ash [4], rice husk ash [5], palm oil fuel ash [6], and coconut shell ash [7] are some examples of viable substitute for cement.

The partial replacement of OPC with coconut shell ash (CSA) in concrete was found to be effective in reducing the cost of concrete and environmental pollution due to the accumulation of agricultural wastes as it incorporates recycling of wastes [8]. The study of Nagarajan, et al (2014) proved that the optimum percentage replacement of OPC with CSA is 10% to 15% based on compressive strength test [7]. This study aims to investigate if the effects of substituting OPC with CSA will produce acceptable results in terms of concrete compressive strength, sorptivity, and resistance to sulfate attack.

2. COCONUT SHELL ASH

The Philippines is one of the major suppliers of coconut in the world, producing more than 15 million tons of coconut annually or 26.4% of coconuts worldwide [9]. There are currently 500 million coconut trees in the Philippines that contribute to 10.4 million tons of biomass annually [10]. An estimated area of 178,180 hectares is allocated for approximately 23,329,000 coconut trees in Quezon province. The coconut shells used for the study were collected from a local farm in this province. The shells underwent calcination to produce CSA in uncontrolled combustion which lasted for 4 hours. The calcined coconut shells were then crushed and ground to produce CSA. To control the effect of fineness on the performance of CSA, only samples that passed through the No. 200 sieve was used for experimentation. The material was oven-dried for 24 hours prior to experimentation to maintain moisture content less than 3% as required by ASTM C618.

3. EXPERIMENTAL PROGRAM

3.1 Mixing and Curing of Concrete Test Specimens

In this study, CSA was used as partial replacement of cement in the concrete mix. The CSA content used for experimentation were 10%, 20%, 30% and 40% of cement by weight. The concrete material will be termed as the CSA concrete. Specimens without CSA were also prepared which serves as the control specimen and represents the conventional concrete. Concrete materials were proportioned in accordance with ACI 211.1. The water-cement ratio of 0.6 was maintained in all concrete mixes. Table 1 shows the concrete batch mix proportion for cylindrical specimens while Table 2 shows the mortar batch mix proportion for mortar prisms. The mixing, curing, and molding of the concrete specimens was in accordance with ASTM C192.

Table 1 Concrete mix proportion

CSA content	Sand (g)	Cement (g)	Water (g)	CSA (g)
0%	3850.0	1400.0	952.0	0.0
10%	3850.0	1337.1	952.0	62.9
20%	3850.0	1265.9	952.0	134.1
30%	3850.0	1184.9	952.0	215.1
40%	3850.0	1091.7	952.0	308.3

Table 2 Mortar mix proportion

CSA content	Water (kg)	Cement (kg)	CSA (kg)	Gravel (kg)	Sand (kg)
0%	3.76	6.67	0.00	19.45	14.61
10%	3.76	6.37	0.30	19.45	14.61
20%	3.76	6.03	0.64	19.45	14.61
30%	3.76	5.64	1.02	19.45	14.61
40%	3.76	5.20	1.47	19.45	14.61

3.2 Compressive Strength Test

To determine the compressive strength of CSA concrete, a total of 150 cylindrical specimens with dimensions of 100 mm diameter and height of 200 mm were prepared and subjected to a uniaxial compression test in accordance with ASTM C39. The uniaxial compression test was conducted after the 7th, 14th, 21st, 28th, 50th, and 90th curing age. The 50th and 90th curing periods were conducted to determine the long-term compressive strength of CSA concrete.

3.3 Sorptivity

Sorptivity of CSA concrete was determined using cylindrical concrete specimens with a diameter of 100 mm and a height of 50 mm in accordance with ASTM C1585. The sorptivity test was done after the 28th-day curing period. This was done by submerging 1mm to 3mm of the samples in water and computing the absorption using the mass difference at different intervals until 9 days. The absorption result was applied to linear regression analysis to measure the initial (1st day of testing) and secondary sorptivity (2nd to 9th day of testing) of the specimens.

3.4 Resistance to Sulfate Attack

Resistance to sulfate attack (RSA) was done by measuring the length change of mortar prisms with dimensions of 25 mm by 25 mm x 275mm in accordance with ASTM C1012. RSA was also measured through a mass change of cube specimens with a side dimension of 100mm following the procedure described in the study of Wang, Zhou, Meng, and Chen (2017) [11]. The specimens were submerged in a sulfate solution after the 28th-day curing period. The sulfate solution has a 5% concentration where 50g of anhydrous sodium sulfate was diluted for every 950g of water. Hence, the exposure class, based on the concentration of the sodium sulfate, is classified as S3. The measurement of expansion through length and mass were conducted once a week until 9 weeks.

3.5 Safety Limits for Sorptivity and RSA

The safety limits for RSA and sorptivity were in accordance with the standards set by the Association of Structural Engineers of the Philippines (ASEP) [12] and by Alexander (2004) [13] which are shown in Tables 3 and 4, respectively.

Table 3 Requirement for establishing the suitability of the combination of cementitious materials exposed to water-soluble sulfate [12]

Maximum expansion strain of mortar prism			
Exposure Class	At 6 mos.	At 12 mos.	At 18 mos.
S1	0.10% ΔL	N/A	N/A
S2	0.05% ΔL	0.10% ΔL	N/A
S3	N/A	N/A	0.10% ΔL

Table 4 Acceptance limits for durability indexes [13]

Acceptance Criterion		Sorptivity ($mm/hr^{1/2}$)
Laboratory concrete		< 6.0
As-built Structure	Full acceptance	< 9.0
	Conditional acceptance	9.0 – 12.0
	Remedial measures	12.0 – 15.0
	Rejection	> 15.0

4. TEST RESULTS

4.1 Pozzolan Classification

To determine the pozzolan classification of CSA, the chemical composition, obtained using Energy Dispersive X-Ray Analysis (EDX), was compared with the chemical requirements for calcined natural pozzolan in accordance with ASTM C618. The oxides formed in calcinating the raw materials of OPC such as calcium oxide, silicon oxide, aluminum trioxide, and iron oxide, are also found in CSA. As shown in Table 5, CSA contains 80.02% of a combined chemical compound of SiO_2, Al_2O_3, and Fe_2O_3. Hence, it signifies that the pozzolanic activity is greater. Silicon dioxide in OPC, commonly known as silica, is the main component that is responsible for the strength of concrete and mortar at an early age; thus, the abundance of silica in CSA denotes that it is a cementitious material and it can be used as a substitute of OPC. Based on chemical properties requirements as stated in ASTM C618, CSA can be classified as Class N pozzolan. Class N pozzolan is raw or calcined natural pozzolans that comply with the given requirements to possess some diatomaceous earth, such as opaline cherts and shales, volcanic ashes or pumicites, and some clays and shales that require calcinations. The loss on ignition is below the 10% criteria for Class N pozzolan. This denotes that the amount of unburnt carbon in CSA is very minimal. This is a desirable property of Class N pozzolan because if the percentage of loss on ignition is greater than 10%, it indicates that the pozzolan has an abundance of unburnt carbon that could decrease the pozzolanic activity [7]. A decrease in pozzolanic activity can lead to a decrease in the strength of the concrete. On the other hand, the moisture content of CSA is greater than the minimum requirement for Class N pozzolan; thus, oven drying before use of CSA is recommended.

Table 5 Chemical properties of CSA

Chemical Compound	CSA %	Class N Pozzolan* %
Silicon dioxide (SiO_2)	60.91	
Aluminum trioxide (Al_2O_3)	17.38	
Iron Oxide (Fe_2O_3)	1.72	
SiO_2 plus Al_2O_3 plus Fe_2O_3	80.02	70 min.
Sulfur trioxide (SO_3)	0.5	4 max.
Calcium oxide (CaO)	0.84	
Moisture Content	4.58	3 max.
Loss on Ignition	5.54	10 max.

*Standard requirements as per ASTM C618

4.2 Compressive Strength of CSA Concrete

The CSA concrete samples were subjected to a uniaxial compressive test to obtain their corresponding strengths on the 7th day until the 90th day of curing. This test was done before conducting sorptivity and RSA tests to verify whether the CSA concrete samples passed the required compressive strength.

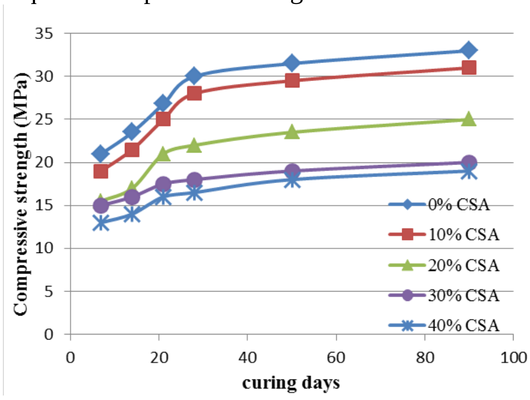


Fig. 1 Compressive strength of CSA concrete

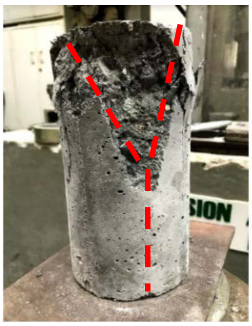
Figure 1 shows the collective compressive strength results from the 7th to 90th-day curing period. It can be observed from the graph that the compressive strength of the samples increases with the curing time. However, as the CSA content increases in the concrete, the compressive strength tends to decrease

4.2.1 Modes of Failure

The failure modes of the samples were observed after conducting the uniaxial compressive test. The control specimens (0% CSA) and the specimens with 10% CSA exhibited a diagonal shear failure (Figs. 2a & 2b). The failure is induced by diagonal shearing which means that samples under uniaxial loading failed in the state of compression because of the insufficient shear strength along the critical planes. Samples that have diagonal shear as a mode of failure tend to have greater strength [14]. Comparing the degree of damage between concrete samples with 0% and 10% CSA, samples with 10% CSA showed more cracks when subjected to a state of compression. Specimens with 20% and 30% CSA exhibited 'Y' cone-split mode of failure (Figs. 2c & 2d) while specimens with 40% CSA showed a single split mode of failure (Fig. 2e). The 'Y' cone-split and single split failures are modes of failure that precipitated from axial splitting. This implies that the sample failed under the stress state of tension due to insufficient tensile strength. Thus, this establishes a weaker strength value on the concrete sample as compared to the shearing modes of failure.

a.) 0% CSA

b.) 10% CSA



c.) 20% CSA

d.) 30% CSA

e.) 40% CSA

Fig. 2 Modes of failure of concrete specimens

4.2.2 Statistical Analysis of Compressive Strength

The Two-tailed T-test is applied to the compressive strength test results of the concrete cylinders. Comparing the compressive strength of CSA concrete specimens with that of the control specimens, the computed T-critical value is 1.8331; while the *p*-value is below 0.5, which implies that the probability of the observed results due simply to chance is very low. The T-values of specimens with 10% CSA from 7 to 90 days curing period is less than the T-critical; hence, the compressive strength of concrete with 0% CSA and 10% CSA concrete is statistically the same and the replacement of OPC with 10% CSA did not affect the compressive strength. Thus, increasing the CSA content until 10% would significantly decrease the cost of concrete while retaining the identical strength performance it has to the conventional concrete.

Response surface methodology (RSM) was used to determine the optimum CSA replacement based on the compressive strength obtained at all curing days. The statistical method revealed that 10% CSA generated the highest compressive strength among all the other concrete design mixtures while the 40% CSA mix obtained the lowest compressive strength. It can be inferred that the optimal CSA content based on compressive strength is 10%. Empirical model as shown in Eq. (1) was derived from RSM in order to predict the compressive strength of concrete with CSA at any curing period. Using the formulated model, the compressive strength of 10% CSA concrete is 92.10% of the strength of the conventional concrete at 28^{th}-day curing period.

$$fc = 15.81 - 0.111(x) + 0.70(t) - 7.020\times10^{-3}(x)(t) - 0.022(x^2) - 9.758\times10^{-3}(t^2) - 2.173\times10^{-5}(x^2)(t) + 5.956\times10^{-5}(x)(t^2) + 4.096\times10^{-4}(x^3) + 4.45\times10^{-5}(t^3) \quad (1)$$

where:
fc = compressive strength of concrete at *x*% CSA, in MPa
x = CSA replacement in decimal (i.e. 10% is 0.1)
t = curing days of concrete, in days

4.3 Sorptivity of CSA Concrete

The slope of the linear trendline of the initial and secondary absorption versus the square root of time is the sorptivity of the specimen. A typical test result is shown in Fig. 3. In all test trials, the initial rate of water absorption, which was taken on the 1st day of experimentation, is greater than the secondary sorptivity, which was taken at the 2nd to 9th day of experimentation, because the empty pores of the sample are being filled with water more rapidly. However, at the stage of the secondary sorptivity, the pores are already filled with water which causes the sample to have a lower sorptivity as time progresses.

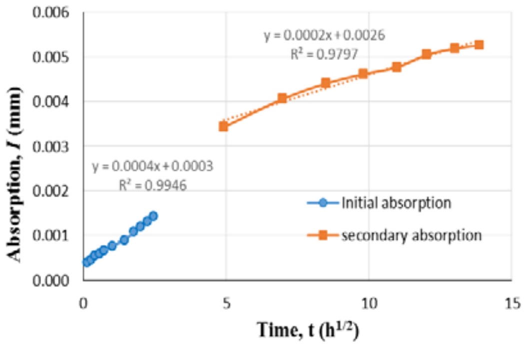


Fig. 3 Typical absorption result of CSA concrete

Figure 4 shows the collective sorptivity results for all CSA concrete samples. It can be observed from the graph that the sorptivity increases as the amount of CSA in concrete increases at the stage of initial sorptivity. This implies that conventional concrete absorbs less water for a certain duration that it is exposed to water. The sorptivity for all samples is acceptable since all concrete specimens passed the acceptable limits stated in Table 4.

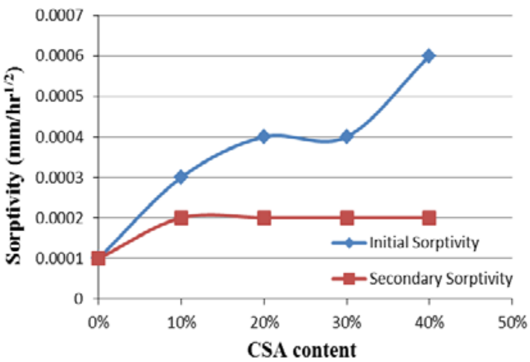
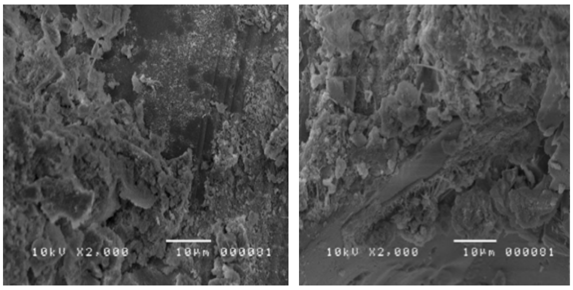


Fig. 4 Sorptivity of CSA concrete

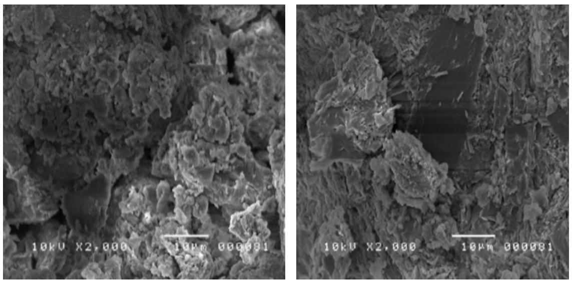
Statistical analysis using the T-test is applied to the results of sorptivity, comparing the sorptivity of conventional concrete (0% CSA) and concrete samples with CSA. The T-test results showed that there is a significant difference between the initial sorptivity of conventional concrete and the initial sorptivity of all concrete samples with CSA. Thus, increasing the CSA content until 40% would cause a significant effect on the absorption behavior of concrete, however, all of this CSA concrete is considered safe in terms of its sorptivity property.

4.3.1 Microstructure of CSA Concrete

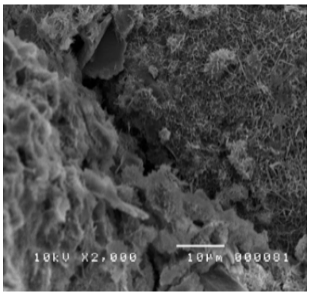
Figure 5 presents the microstructure of CSA concrete. It can be observed that increasing the amount of CSA in the concrete could also increase the void spaces in between the aggregates; hence, the sorptivity increases.



a.)conventional concrete (0% CSA) b.) 10% CSA



c.) 20% CSA d.) 30% CSA



e.) 40% CSA

Fig. 5 Microstructure of CSA concrete

Generally, the cement paste, consisting of OPC and water, is more bonded with the flaky CSA particles in the concrete; thus, aggregates are less bonded with the cement paste. This resulted in greater void spaces in between aggregates when the CSA content is increased because the cement paste seeps through the intergranular voids of CSA. Furthermore, CSA has a tendency to clump together with the cement paste; hence, the cement paste was hindered from filling voids and coating aggregates. The increased amount of void spaces also decreases the compressive strength of the sample; because aggregates are less bonded with each other. CSA is composed of 60.91% silica and 0.84% calcium oxide, which is responsible for the early and late strength of concrete, the effect of CSA on the bond of the aggregates and cement paste outweighs its promising chemical composition. Thus, the optimal CSA content is 10%, which has a statistically identical strength as the conventional concrete and acceptable sorptivity.

4.4 Resistance to Sulfate Attack of CSA Concrete

Gypsum, a form of sulfate, is usually added to the production of cement to prevent the flash setting and improve the performance of concrete; furthermore, it is composed of hydrated calcium and sulfate. On the other hand, sulfate combined with alumina and calcium produces ettringite, which is normally present in concrete at early ages of the concrete [15]. However, excessive amount of ettringite in the void spaces of the concrete cause deterioration, in the form of expansion, in concrete and mortar.

The length change or expansion of CSA mortar prisms was obtained to determine if the samples would not go over the NSCP limit for concrete susceptible to sulfate attack. Since the solution wherein samples were submerged contains 5% sodium sulfate (exposure class S3), the limit is 0.1% in 18 months. Figure 6 shows the relationship of the length change of CSA mortar prisms with the time when it is immersed in a sodium sulfate solution. It can be observed that as the time of immersion increases, the expansion increases as well. Moreover, the expansion of mortar also increases as the CSA content increases. It can be observed that at the 9th week, the mortar with 0% and 10% CSA surpassed the acceptable limit while the expansion of other CSA content exceeded the limit at the 7th and 8th week. Previous research showed that it took 49 days for the conventional concrete mortar to reach 0.1% expansion when submerged in a 5% sodium sulfate solution [16]. In this study, the limit of 0.1% expansion is attained sooner than 18 months even for mortar without CSA. Statistical analysis using the T-test is applied to the results of length change of mortar prisms submerged in sulfate solution, comparing the length change of mortar prism without CSA and mortar prisms with CSA. There is no significant difference between the length changes of 10% CSA mortar prism and mortar prism without CSA before the expansion exceeds the acceptable limit. Thus, increasing the CSA content until 10% does not affect the change in length and the length of expansion is within the acceptable value.

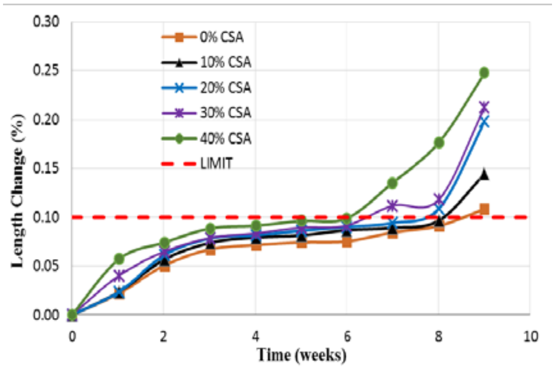


Fig. 6 Length change of CSA mortar prism

The expansion of CSA concrete cubes in terms of mass change was obtained to determine the effect of sulfate attack on the exposure of concrete to saline soil and groundwater or saltwater containing sodium sulfate in its aqueous state. The test results are presented in Fig. 7. It can be observed that the mass change is directly proportional to its time of exposure. As the CSA content increases, the expansion in terms of mass change also increases as time progresses. Statistical analysis showed that there is no significant difference between the mass change of conventional concrete and 10% CSA concrete. The performance of 10% CSA concrete is statistically identical to conventional concrete in terms of expansion or mass change.

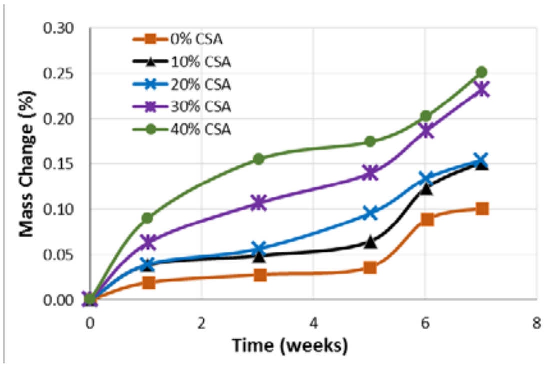
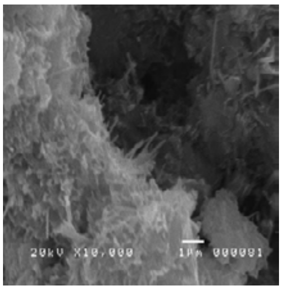


Fig. 7 Mass change of CSA concrete

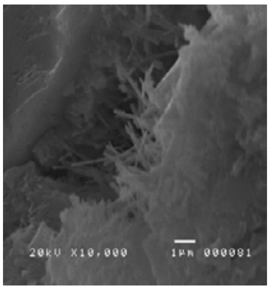
4.4.1 Microstructure of CSA concrete after exposure to Sulfate solution

The microstructure of CSA concrete after exposure to sulfate solution is shown in Fig. 8.

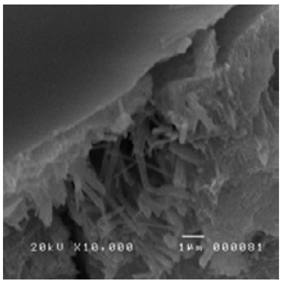
Since sulfate can only be detrimental to concrete in its aqueous form, sulfate will only cause deterioration if it seeps through the void spaces and bond with the excess calcium oxide, also known as lime, and alumina in the concrete. It can be observed that there are needle-like particles, ettringite, between the void spaces of CSA concrete. Ettringite is a compound composed of calcium, sulfate, and aluminate. The sulfate interacted with the free lime and alumina in the concrete, from the OPC and CSA, which resulted to ettringite that was displaced into the intergranular and trans-granular voids and caused expansion of the concrete. This expansion in the concrete can be detrimental and cause spalling, onion peeling, or scaling. Furthermore, ettringite is expansive; hence, if it expands, it can lead to the cracking of concrete, which ultimately decreases its strength [17].



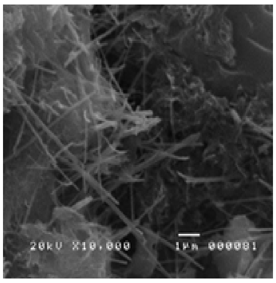
a.) conventional concrete (0% CSA)



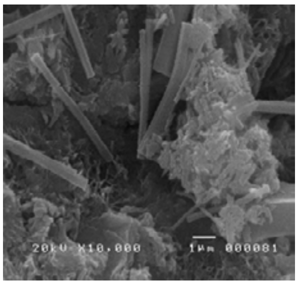
b.) 10% CSA



c.) 20% CSA



d.) 30% CSA



e.) 40% CSA

Fig. 8 Microstructure of CSA concrete after exposure to sulfate solution.

Gypsum is also present in the concrete together with ettringite in between the voids, which was produced when the free calcium ions in the concrete mix reacted with the sulfate ions. Gypsum is expansive and has a softening effect that can cause strength loss [17]. However, the cause of the expansion of the concrete immersed in the sulfate solution is not exclusive to the expansive property of gypsum and ettringite. The tensile stresses that may have accumulated in the concrete could have also caused the expansion [17]. The cracking of concrete could lead to bigger void space wherein the sulfate could penetrate the concrete and cause more expansion due to ettringite and gypsum.

5. CONCLUSION

Based from the experimental results, the following conclusions can be drawn:

Coconut shell ash (CSA) contains 80.02% of the combined silicon dioxide, aluminum trioxide, and iron oxide classifying it as Class N pozzolan. The high pozzolanic activity makes it suitable to be used as a replacement for ordinary Portland cement (OPC).

The microstructure of conventional concrete and CSA concrete shows that there is an increase in voids between aggregates when the CSA content increases; thus increasing CSA content increases sorptivity and make it more susceptible to sulfate attack. The sorptivity of the conventional concrete and all CSA concrete mixture up to 40% CSA is deemed to be acceptable and is less than the limit which is $6mm/hr^{1/2}$.

There is no significant difference between the conventional and 10% CSA concrete in terms of compressive strength and mass change due to expansion after immersion in sulfate solution at all curing ages. The compressive strength of concrete with 10% CSA is 92.10% of the compressive strength of the conventional concrete.

The microstructure of the CSA concrete after immersion in the sulfate solution shows the presence of ettringite and gypsum, which causes softening and expansion effect in the concrete.

The results obtained proved that CSA is suitable to replace OPC because it passed the criteria to classify it as a cementitious material, however, the optimal percentage of replacement of OPC that will not affect the durability and strength of concrete should be considered. The optimum level of CSA content as replacement of OPC is 10% wherein the durability and strength are acceptable and can lead to a lower cost in concrete production. CSA concrete can be used as an economical approach in having an acceptable level of durability of concrete structures without increasing the cement usage in any construction project; however, it is recommended that further study on the evaluation of the long term behavior of hardened concrete, specifically on its resistance to sulfate attack, be conducted.

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