UTILIZATION OF COCKLE SHELLS AS PARTIAL BINDER REPLACEMENT IN CONCRETE

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ABSTRACT

The main aim of this study is to investigate the performance of calcined cockle shell as a partial binder replacement in modified concrete (MC). Cockle shell was selected based on its high calcium carbonate (CaCO₃) content; an active ingredient in cement manufacturing. The investigation was carried out by substituting the cement in the conventional concrete formulation with percentages of calcined cockle shell powder (10%, 20%, 30% and 40% respectively). Primary concrete parameters such as pH, carbonation depth, compressive strength and sorptivity were studied for the periods of 56 days. The results showed that modified concretes exhibit superior performance than the control specimens, which is strongly influenced by the amount of substitution. The addition of calcium carbonate in the mixture is proven to increase the physical performance especially carbonation depth. However, the modified concretes' strength developments are inversely proportional to the amount of substitution. It is believed that the aragonite polymorphs contribute to the low strength development due to its different symmetry and crystal shapes to calcite, thus leading to poor bonding properties of the matrix with aggregates. No significant strength developments are observed after 28 days for all modifications.

KEYWORDS: Binder; cement; cockle shells; modified concrete; carbonation

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1.0 INTRODUCTION

Concrete is widely used as the main construction material because of its excellent compressive strength, versatility, and economical values (Awang-Hazmi, 2007; Othman, 2007; Lee, 2017). It has made a prominent progress since the introduction of reinforced concrete (RC) and Integrated Building System (IBS) (Corradi, 2006). Cement, the main component in concrete that act as a binder is made from calcium carbonate (CaCO₃) material such as limestone, marl or chalk (Mohamed et al., 2012). This infrastructure development has led to increasing production demand for cement annually, particularly in new developing countries (Maitra, 2006). Due to this continuous demand, the production intensity has aggravated the environmental problems such as deforestation, land mining, depletion of non-renewable resources, contaminations and air pollution (Dennis & Pacciani, 2009). In addition, UNEP (2009) reported that the extraction, use and end-of-life disposal of construction materials are responsible of several adverse environmental effects. Therefore, the establishment of the Green movements (resources management and recycling of by-products) are important to protect natural resources, reducing carbon dioxide (CO₂) emissions and saving energy.

With concrete being pivotal material in construction activities, finding alternatives to improve its properties or maintain them while managing a lower production cost would be extremely beneficial and lucrative. For example, industrial by-products such as oil palm shell, glass, fly ash and bottom ash are widely used in the construction industry as an additive and/or admixture for concrete to decrease the cost while reducing the amount of waste. Most of the waste and by-products mentioned are commonly disposed in landfill without any pre-treatment because it was perceived as useless and has no commercial value even though some composite by-products have 3000 times greater (Mishnaevsky, 2007).

This study was set out to investigate the correlation between substitution rates (%) of calcined cockle shells and concrete performance. The laboratory test procedure is based on BS EN 12350-1:2009, BS EN 12350-2:2009 and BS EN 12350-6:2009.

2.0 COCKLE SHELLS AS ALTERNATIVE BINDER

The applications of using seashell by-products as construction materials are established since hundreds of years ago in coastal regions due to unavailability of limestone (Konecny, 1990; Kelly, 2009; Becker, 2012). The most prominent establishment is tabby construction; a cementitious greyish-white color, material made from calcined seashell byproducts (that acts as lime), water and sands (Monita, 2015). Apart from locally available resources, the tabby concrete production was simple and could be prepared by unskilled labor (Taylor, 2011). The lime was obtained by cooking whole shells in a kiln, then slaking the shells by adding water to them. Tabby was also used as a substitute for bricks, which is rare and expensive because of the absence of local clay (Hooton & Konecny, 1990). The tabby concrete technology seems to be diminished after the existence of Ordinary Portland Cement production, but the conservation and preservation of the tabby structures are still conducted to sustain the historical green design (Morris, 2005; Balzer, 2012).

Cockles (*Anadra granosa*) are an edible bivalve mollusc and one of the major seashell species in the Malaysian aquaculture industry. Locally known as *kerang* in Malay or blood cockle, 57,000 tons of cockle were harvested in Peninsular Malaysia in year 2011 (Dennis & Pacciani, 2009). The industry is currently valued at approximately RM160 millions (Spykerman, 2016). Cockles are highly demanded by the food industry due to its availability, delicious taste, nutrient and economical values (Smellie, 2015).

Even though the cockle breeding industry has impressively contributes to the development of fisheries economy, cockle shells have been discarded or abandoned after being cooked. This is apparent in areas with high numbers of seafood restaurant. Nor Hazurina (2012) reported that cockles shell wastes have become an environmental concern and an impending issue in coastal areas and landfills. The inappropriate disposal method can decrease the water quality thus obstructing the growth of shellfish (Folke & Kautsky, 1989). In addition, cockles shell waste that has been discarded and/or untreated for an extended amount of time can be a source of offensive odor as a result of the decay of the cockle meat or the microbial decomposition of salts into gases such as ammonia, hydrogen sulphide and amines (Mokhtar, 2009; Hamester et al., 2012). These by-products are perceived as no commercial values and discarded (Cox, 2009). In contrast, chemical and microstructure analyses revealed that cockle shells are predominantly composed of calcium carbonate (CaCO₃) and other mineral compounds as shown in Table 1.

	-				
Researchers	Mineral Composition (% wt.)				
	CaC	Mg	Si	Na	Others
Mustakimah et al. (2012)	98.99	0.51	0.078	n/a	< 0.1
Awang-Hazmi et al. (2007)	98.70	0.05	n/a	0.9	< 0.1
Bharatham et al. (2014)	95.7	0.13	n/a	2.1	0.54

Table 1. Mineral composition of cockle shells

The analysis of crystal structure using XRD clarifies that raw cockle shell is made up of aragonite; one of three CaCO₃ polymorphs (the others are calcite and vaterite) (Nurul et al., 2011; Zubaidi & Tabakhah, 2015). The vaterite and aragonite crystals formed as a result of pre-adsorption of organic substrates or additives on the surface. It is also known that the formation of the stabilized vaterite phase requires strong Ca–O interaction with the additive or the substrate surface (Choi & Kim, 2000). The acid or alkali treatment of the surfaces increases the number of polar functional groups such as carboxylate and amino groups, which are known to interact strongly with polar organic molecules through hydrogen bonds (Wang, 2000). This may result in the accumulation of organic molecules on the surface, which induce the nucleation of aragonite crystals (Lakshminarayanan et al., 2004).



Figure 1. The surface morphology of the cockle shells powder (Nurul et al., 2011)

Even though calcite is the most stable polymorphism of calcium carbonate, aragonite has higher density and hardness which make suitable material for manufacturing of

plastic, paper and glass fibre (Masami, 2002). Previous studies revealed that cockles shell is also utilized as construction materials, liming agent, agricultural fertilizers and pharmaceutical precursors (Barros, 2009; Mokhtar, 2009). Cockle shells revalorization also sparked numerous research interests in various fields of study, including food chemistry, biotechnology, material sciences, bio-mineralization and biomaterial engineering (Zakaria et al. 2004). Recently, the applications of cockle shells as aggregates and binder replacement are widely studied (Sugiyama, 2004; Falade, 1995; Yang et al., 2005, Yusuf et al., 2011; Muthusamy & Sabri, 2012). The studies were conducted due to cockle shell chemical properties that similar to limestone. The process of producing binder is based on lime cycle principles (Morandeau et al., 2014 & Mindess et al., 2003) as shown in Figure 2.



Figure 2. Lime Cycle (Schotsmans et al., 2011)

The calcium carbonate (CaCO₃) is converted to calcium oxide (CaO) through the calcination process at high temperature. This endothermic process uses high temperature of energy and release CO₂ as by-product. The CaO can be us as CO₂ adsorbent (BSI, 2009). Process of reacting CaO with water (H₂O) will cause the hydration process occurred which involve the production of slake or hydrated lime; calcium hydroxide (Ca(OH)₂). This process is an exothermic reaction. If the stoichiometric water is added, dry powdered precipitates will form and excess water lead to presence of final product in form of lime putty or aqueous dispersion of hydroxide crystal. The presence of CO₂ in atmospheric will provide a reaction with Ca(OH)₂ and release the final product of calcium carbonate (CaCO₃) and water precipitation. This process is called carbonation where the presence of CO₂ results in CaCO₃ crystal acts as a binder due to the interconnected microstructure joining with the rest of element in the mix.

3.0 MATERIALS AND METHODS

3.1 Raw Materials

The control and modified concrete (MC) was manufactured by using Ordinary Portland Cement (Type I) as shown in Table 2, river sand as fine aggregates (Specific gravity of 2.5 and size of 0.4mm), local harvested cockle shells and distilled water. Sand was used in the mixing ingredients to prevent the shrinkage and cracking of the modified mixture (Taylor, 2011). The river sand is preferred because the less amount of salt and silt contents compared to beach and dune sand. The beach sand can be used, but requires a pre-treatment where the sand needs to be washed until the salt content is decreasing (Balzer, 2012).

Table 2. Chemical composition of OPC Type 1

Type of compound	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	LOI
Amount (%)	64.67	18.59	4.62	4.17	2.35	3.32	0.92	1.03

3.1.1 Preparation of Cockle Shells Powder (CSP)

Cockle shells used in this study were collected from the coastal beach area in Pantai Remis, Kuala Selangor. The preparation of cockle shell powder (CSP) was conducted according to the process introduced by Islam et al. (2013). The shells were washed with water to remove dirt and boiled for 2 hours to remove cockle meat residues and odours. The shells, then were air dried for 12 hours. The calcination process was conducted using lab furnace (Carbolite Furnace) at a temperature of 500°C for 2 hours. The calcined cockle shell was then crushed by using a pestle and mortar before being pulverized into ¹/₂–1 mm range powder. The pH values for CSP throughout the process are shown in Table 3.

Matorial	II
Waterial	рп
Fresh cockle shell	7.11
Dried cockle shell	7.56
Calcine cockle shell	10.9
Fresh mix of modified concrete	11.6

3.2 Specimens Manufacturing

The concrete rheological properties were obtained through a slump test. Slump test is an empirical test to get an ideal amount of water during the mixing process to achieve standard consistency and good workability of concrete (BS EN 12350-2:2000). The standard slump test mould has been used; 30 cm (height), 20 cm (bottom diameter) and 10 cm (top diameter). The raw materials were mixed together by using 1:2:3 ratios (1 part of water; 2 parts of aggregates and 3 parts of cement). The results of slump test are shown in Figure 3.



Figure 3. Slump test results for pervious concrete control formulation

A study by Dennis (1999) pointed out that low humidity or insufficient water amount in the pores hinder CO_2 to form carbonic acid and react with the alkaline cement hydrates. Hence, while lower level of humidity, the CO_2 diffusion rate will also decrease.

3.2.1 Mixing Formulation

For modified concrete, the cockle shells were directly substituted for the cement at 10%, 20%, 30% and 40% (R10, R20, R30 and R40 respectively) by mass. The governing equations obtained from the slump test were used as the precursor for modified formulations. Due to changes in materials, the replacement percentages were calculated by using Relative Bulk Density (RBD) principles (Razali, 2014). Table 4 below shows the mix proportion of the specimens.

			0		
Sample	Sand (g)	Water (mL)	OPC (g)	Omitted OPC (g)	Cockles Shells Binder (g)
Control	1000	1185.2	1002	0	0
R10	1000	1259.3	1002	100.2	62.6
R20	1000	1296.3	1002	200.5	125.3
R30	1000	1333.3	1002	300.8	188.0
R40	1000	1370.4	1002	401.0	250.6

Table 4. Mixing formulation

The concrete was cast in 40mm x 40mm x 40mm of polystyrene mould. The concrete mixtures were poured into the mould and tempered properly to remove air. The specimens were demoulded 24 hours before set to curing in water. Testings were conducted over 7, 14, 28 and 56 days of casting.

3.2.2 Curing

Concrete's strength increases with age (time) through a hydration process that require moisture (water) and a favorable temperature. Curing assists the cement hydration reaction to progress steadily and develops calcium silicate hydrate (C-S-H) gel, which binds the aggregates leading to a rock solid mass, makes the concrete denser, decreases the porosity and enhances the physical and mechanical properties of concrete. According to Uddin et al., (2012), concrete properties are significantly influenced by curing since it greatly affects the hydration of cement. A proper curing maintains a suitably warm and moist environment for the development of hydration products and thus reduces the porosity in hydrated cement paste and increases the density of microstructure in concrete. As cement hydrates, the strength increases and permeability decreases. When hydration stops, strength gain ceases. Therefore, proper hydration of the cement is important in the development of strong durable concrete. Most concrete mixes contain an adequate amount of the mixing water for complete cement hydration.

After 28 days of casting, typically 90% of the final strength is reached and continuously developed (Neville & Brooks, 2010). The conversion of calcium hydroxide (Ca(OH)₂) in the concrete into calcium carbonate (CaCO₃) from absorption of CO_2 over several decades further strengthens the concrete and makes it more resistant to damage. However, the carbonation reaction lowers

the pH of the cement pore solution and can corrode the reinforcement of reinforced steel in concrete (Chang, 2004).

In this study, wet curing was selected as curing method. This method fulfil all the requirements of curing, which involving promotion of hydration, elimination of shrinkage and absorption of the heat of hydration. It is pointed out that even if the membrane method is adopted, it is desirable that a certain extent of water curing is done before the concrete is covered with membranes. Water curing was done in the way of total immersion of concrete cube in the container.

3.3 Chemical and Physical Testing

3.3.1 pH

The pH value was used to determine the changes during the hydration process. For fresh mixed concrete, the pH strips were dipped into the mixture. For hardened concrete (7 days and above), the method applied by Rasanen & Penttala (2004) was used. The concrete specimens were manually cut at 20 mm from the surface to avoid errors in measurements due to carbonation and moisture content gradient. The specimens then were crushed into a powder. 15 g of powder was mixed with 15 g of distilled water. The pH of the solution was then measured by using a pH meter (Mettler Toledo).

3.3.2 Carbonation Depth

The following reaction detailing the concrete carbonation mechanism. The first reaction is in the pores of the concrete where carbon dioxide and water react to form carbonic acid, as given in Equation (1) below: -

$$CO_2 + H_2O H_2CO_3 \tag{1}$$

The carbonic acid then reacts with the calcium phases and forms calcium carbonate, as expressed in Equation (2) as follow:

$$H_2CO_3 + Ca(OH)_2 CaCO_3 + 2H_2O$$
 (2)

Once the calcium hydroxide from the concrete is converted into calcium carbonate, the calcium silicate hydrates liberate calcium oxide, which further leads to calcium carbonate, as shown in Equation (3) below: -

 $H2CO_3 + CaO CaCO_3 + H_2O$ (3)

The pH value of pore solution of concrete is around 12.6 - 13 because of the dissolution of Ca(OH)₂, which leads to the presence of hydroxyl ions in the pore water. However, long time of carbonation reduces the pH of concrete, but an increase in the strength of the concrete. The carbonation depth was influenced by the diffusion rate of CO₂ into concrete, the pore size of concrete and also the water vapour precipitation of concrete as an agent to CO₂ to diffuse in the concrete.

The depth of carbonation after the specimens were casted at 7, 14, 28 and 56 days were investigated. The samples were sprayed with phenol phthalein solution (Fisher Chemical). The resulting pink stain indicated the carbonation activity. The average carbonation depth was measured by digital caliper (to the nearest 0.01mm).

3.3.3 Compressive Strength

The concrete strength is influenced by numerous factors such as void ratio, water-cement ratio, alite and belite contents, additives, aggregate size and aggregate-cement ratio. Due to variation of replacement percentage in formulations, the modified concrete is estimated to have variable strength as well. Therefore, the interaction of the CSP and OPC is critical to ensure the positive rate of strength growth and the final strength.

The specimens were tested for compressive strength in accordance with BS EN 12350-1:2000 - Testing Fresh Concrete & Sampling by using INSTRON Floor Mounted Material Testing System. The data produced illustrates the stress in the specimens up to failure.

3.3.4 Sorptivity Test (capillary absorption)

The durability of concrete, largely depends on the ease with which fluids enter and move through the matrix. Although, in general, permeability is taken as an indicator of a concrete's ability to transport water (or oxygen and carbon dioxide), more precisely there are two mechanisms controlling the uptake and transport of water. Permeability which is a measure of the flow of water under pressure in a saturated porous medium, and sorptivity, which characterises the material's ability to absorb and transmit water through it by capillary suction.

For this study, sorptivity test was conducted by adopting the direct gravimetric method (Razali, 2014). Prior to this, the specimens were oven dried for 1 hour before sorptivity test. Then, they were placed into 5mm of distilled water in a container as shown in Figure 4. The specimens were then weighed to determine the changes in weight over a specified time frame of 1, 3, 5, 10, 15, 30 minutes. A minimum of 5 measured weight gain points were then plotted onto a graph with the water absorption measured in g/m2 against the square root of time t1/2. The sorptivity was then determined from the gradient of the slope. From that, the sorptivity was calculated by using the formula given in Equation (4) below (Dias, 2000): -

$$\frac{W}{A} = k\sqrt{t} \tag{4}$$

where,

W = water absorbed (g) A = surface area (mm²) k = sorptivity coefficient t = time (s)



Figure 4. Sorptivity test (Ahmad Sani, 2017)

The capillary suction of the modified concrete interface relates to the sorptivity process; However, due to the presence of discontinuous pores which do not allow water movement by capillarity, therefore it does not contribute to sorptivity (Neville, 2000). This is because one of the main factor that influence the sorptivity of the water into a concrete structure is the pore size of the concrete. The larger pore size of the concrete gives higher diffusion of water through capillary action gravitational force.

4.0 RESULTS AND DICUSSION

4.1 pH

Figure 5 presents the pH result of MCs for 7, 14, 28 and 56 days of curing. Increasing day of curing, reduce the pH of concrete due to the carbonation process (Dangla, 2014). From the results, that there are no significant differences of pH between MCs and control specimens, but the evident trend can be established. All fresh mixed cements have a pH approaching pH 14. However, once the carbonation reaction takes place, pH reading becomes neutral (pH 7). Carbonation progressively lowers the pH in a mortar which maximizing the amount of Ca²⁺ ions. The pH variations could be attributed to the difference in material characteristic and their properties with a corresponding influence on the extent of carbonation of the concrete.





4.2 Carbonation Depth

As can be seen in Figure 6, carbonation depth increased with respect to time. This is due to the reaction of atmospheric CO_2 to the $Ca(OH)_2$ inside the cement structure producing $CaCO_3$. This reaction is continued inside the concrete

structure until they are completely carbonated. According to Fattuhi (1997), this reaction reduced the pH of concrete in increasing of carbonation time. Another study by Chang (2003) showed the same evidence where the concrete that exposed to atmosphere in a long period of time will reduce the pH of concrete.



Figure 6. Carbonation depth of concrete vs. time

It is apparent that all formulations of the specimens had been carbonated throughout the duration of the study. It was observed that carbonation depths are directly proportional to the amount of replacement. The higher the replacement percentages, the higher carbonation depth detected. Control specimens only carbonated from 8.1mm on day 7 to 8.9mm on day 56. Whilst the highest carbonation depth is detected at R40 specimens in which its carbonated 10.7mm on day 7 to 12.9mm on day 56. This observation is consistent with the carbonation principles that carbonation depth increases with time. During the carbonation process, the CO_2 in the surrounding environment is enough to make calcium hydroxide (Ca(OH)₂) reacted as stated in Equation (3).

A study by Somerville (2003) have reported that normal concrete will only carbonated after a long period of time, with carbonation depth in the range of 5 mm - 50 mm annually. The application of CSP in addition to cement as binding materials produced more $Ca(OH)_2$ in the concrete composition, hence reacted more with atmospheric CO_2 to be carbonated (Chang & Chen, 2006). This results in all Ca compounds to dissolve and finally forming calcium carbonate. The porosity of the concrete also influences the carbonation depth. An increase

in the proportion of CSP shows higher carbonation depth due to the pore size of the concrete that allow diffusion of CO_2 in concrete structure through the water in a form of carbonate ions that will react with the Ca ions of the pore water (Dennis & Paccian, 2009).

Table 5 illustrates the phenolphthalein stain of carbonated area of all specimens throughout the testing period. The pink stain at the middle of the concrete surface shows the absence of CO_2 and indicates that the concretes were not completely carbonized. While the colorless around the concrete specimen shows the presence of CO_2 that was diffused into the concrete from the atmosphere. The higher the carbonation depth, the higher the amount of CO_2 absorbed. The absorption of CO_2 in the surface of the concrete was influenced by the amount of the atmospheric CO_2 inside the concrete, the humidity and also the temperature during the curing process. These observations are supported by Papadakis & Ngala (2002).

Samples	Control	R10	R20	R30	R40
7 days			0	0	
14 days		0	9		
28 days					
56 days		1	•		

Table 5. Phenolphthalein stain of carbonated area

4.3 Compressive Strength

The compressive strength results of the MCs are presented in Figure 7. Overall, it can be suggested that the MCs are able to resist load better than the control specimens. All MCs achieved rapid strength development for the initial 14 days of the test. On day 14, the highest compressive strength was achieved

by R10 at day 14 (25 N/mm²). However, the decreased of strength were noticeably identified from day 14 to day 28. There were no significant strength developments of MCs after 28 days, although the control specimens kept on increasing its strength in small magnitude.



Figure 7. Compressive strength of concrete

These results indicate that the strength of the MCs depends on the amount of replacement, in which less amount of replacement material yield in higher strength. It is believed that MCs with low amount of sand leads to segregation and bleeding of concrete. Adding a larger quantity of sand makes the concrete denser and stronger. Apart from that, excessive amount of sand used causes high consumption of cement in concrete. According to Okpala (1999), the failure of the concrete at certain ages depending on the breakdown bond between the binder and the paste. Therefore, there may not be enough bond between CSP particles and cement paste to produce sufficient bond strength and ultimately sufficient compressive strength of the concrete. A study by Young & Darwin (2012) has highlighted that material with rough textured surfaces will improve the mechanical component of the bond. Such observation is possible since too much of CSP is an indication of high effective surface area, would lead to the insufficient proportion of cement, thus leading to poor bonding properties of the matrix with aggregates, thus results in poor strength. These results are consistent with other research, in which it was found that the stability of the binder and aggregates influence the concrete strength (Neville, 1995). The different characteristics of cement hydrates products which is calcium silicate

hydrates (C-S-H) in CSP and cement also influence the loner-term stability of the concrete as higher surface area of the hydrate cement product gives higher in strength development of concrete (Foster, 2004).

4.4 Sorptivity (capillary absorption)

According to Hall & Hoff (2009) the sorptivity of concrete is influenced by the materials behavior, with regards to variations in composition and curing conditions, in a rational way. Figure 8 presents the data on the average sorptivity value of MCs in different period of curing in which the control specimens have higher sorptivity values that indicate higher porosity value than MCs. MCs also exhibit decreasing sorptivity values that are like those observed by Parrot (1992). MCs with higher replacement percentages are more compact and have less capillary suction activity due to the less size of pores. This is due to the accelerated carbonation activity of the MCs throughout the testing period (as observed in Section 4.2).



R40 exhibit the lowest sorptivity value due to the less presence of pores on the surface of cross sectional area compared to control specimen. A study by Sabir (1998) proves that the matrix structure of concrete that relates to the transport of water is the pore system of cement paste, particularly on the aggregate-paste interface environment. These observations are supported by Wild (1998) in which it was reported that water movement in concrete is predominantly controlled by the bulk of the hardened cement paste, which is the continuous phase in concrete. The low volume of cement presence is contained in the hardened concrete gives a lower sorptivity activity of water throughout the concrete surface.

5.0 CONCLUSION

The most obvious finding to emerge from this study is that the modified concretes with percentages of CSP replacement performed better than the control specimens. These results are consistent for each test in which the modified concrete performances are strongly depending on the replacement rate. The higher replacement percentage is beneficial for the acceleration rate of pH, carbonation and compressive strength. However, in compressive strength, the modification is only efficient at a lower replacement rate. This is because the extra amount of CSP has caused the imbalance proportion of binder, thus leading to poor bonding properties of the concrete matrix (with aggregates). Therefore, higher CSP replacement specimens exhibit lower strength in increasing days of curing. The modification is not required if the specifier is looking for an increase in sorptivity performance. The findings of this study have a number of important implications for future applications, especially on the adaptability of alternative binder.

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