REVALORIZATION OF AQUACULTURE WASTE: THE PERFORMANCE OF CALCINED MUSSEL SHELLS AS PARTIAL CEMENT REPLACEMENT

Nadia Razali^{1,*}, Raja Nur Farhana Raja Aris², Nadlene Razali³ and Khairul Faizal Pa'ee⁴

^{1,2}Section of Environmental Engineering Technology, Universiti Kuala Lumpur - Malaysian Institute of Chemical and Bioengineering Technology, 78000 Alor Gajah, Melaka ³Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka, 76100 Durian Tunggal, Melaka ⁴Section of Food Engineering Technology, Universiti Kuala Lumpur - Malaysian Institute of Chemical and Bioengineering Technology, 78000 Alor Gajah, Melaka

*Email: nadiarazali@unikl.edu.my

ABSTRACT

The widespread adoptions of alternative binder in concrete formulations are playing an increasing role in carbon dioxide footprint reduction. With the development of green technology in construction, opportunities have been established in using waste materials as partial or full substitution of construction material that could save the production cost and exhibit a similar characteristics as its conventional counterpart. In this research, an investigation on the performance of calcined mussel shells (Perna viridis) as partial binder replacement in concrete has been carried out. Specimen cubes (40mm x 40mm x 40mm) with different replacement percentages (10%, 20%, 30%, and 40%) by volume were produced. Primary concrete parameters such as pH, carbonation depth, compressive strength and sorptivity (capillary absorption) were studied for 56 days. From the investigation, all modified concrete formulations performed better in carbonation depth, compression strength and sorptivity than control specimens on all testing days. It was been observed that the higher the replacement percentage, the better carbonation depth and sorptivity values. However, the opposing trend was detected in compressive strength; in which the higher the replacement percentage, the less strength the concrete possessed. 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 concrete matrix. These outcomes were meaningful for our understanding on the modified concrete formulation, specification and product design of cement materials and the revalorization of aquatic wastes.

Keywords: Cement, concrete, mussel shells, partial replacement.

INTRODUCTION

Concrete has been widely used as the main construction material because of its excellent compressive strength, versatility, availability, fire resistance and economical values [1, 3, 4]. It made a prominent progress since the introduction of reinforced concrete (RC) and Integrated Building System (IBS) [5]. Cement, the main component in concrete that act as a binder is made from calcium carbonate (CaCO₃) material such as limestone, marl or chalk [2]. Due to a continuous demand, the cement making industry intensify and aggravated the environmental problems such as deforestation, land mining, depletion of non-renewable resources, contaminations and air pollution [6, 7]. Therefore, the establishment of the green movement is not only intended to protect natural resources but also aims to reduce carbon dioxide (CO₂) emissions and energy saving [8]. With concrete being so pivotal material in construction activities, finding alternatives to improve its properties or maintain them while managing a lower production costs 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 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 were 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 has a fracture toughness 3000 times greater [9].

Mussel Shells as an Alternative Binder

Living in a coastal nation, Malaysian consumed approximately 1.6×10^6 metric tons of seafood annually with trading worth of RM4.2 million [10, 13]. Apart from being delectable shellfish, mussel is also an ideal bivalve mollusc species for aquaculture activities. This is due to its high growth rate and non-weather dependant reproductive capability [11]. In Malaysia, the most common species to be cultured is Asian green mussel (Perna viridis) [12].

Even though the mussel breeding industry has impressively contributed to the development of fisheries economy especially in Southern Malaysia region, mussel shells wastes have becoming an alarming environmental problems. This is apparent in areas with high numbers of canning factories and seafood restaurants. Martínez-García et al. [14] reported that amounting 1 million tonnes of mussel shells worldwide have becoming an environmental concern and impending issue in disposal management. The improper disposal method can effecting the water quality thus obstructing the growth of other aquatic lives [15]. In addition, mussel shells waste that has been discarded and/or untreated for extended amount of time can be a source of unpleasant odour as a results of the decay of the mussel meat or the microbial decomposition of salts into gases such as ammonia, hydrogen sulphide and amines [16, 17].

Usually, mussel seashells are perceived as waste and carrying no commercial values [18]. In contrast, chemical and microstructure analyses revealed that mussel shells composed approximately >90% of calcium carbonate (CaCO₃) as shown in Table 1 [19, 20].

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Table 1. Mineral compositions of mussel shell [20]							
Mineral	CaO	K ₂ O	SiO ₂	SrO	SO ₃	MgO	Al ₂ O ₃
Composition (%)	95.7	0.5	0.9	0.4	0.7	0.6	0.4

The applications of using seashell by-products as construction materials have been established since hundreds of years ago in coastal regions due to unavailability of limestone and aggregates [26, 27]. The most prominent establishment is tabby construction; a cementitious greyish-white colour material made from calcined seashell by-products (that acts as lime), water and sands [30-32]. With the development of green technology that is widely promoted in the past few years, many researchers had come across using seashell waste as sophisticated composite material in the construction practices [21, 22]. The chemical composition similarity to limestone powder makes seashell as practicable options for binder and aggregates replacements [23, 24]. In addition, Razali & Forster [25] stated that modified concrete formulations with seashells can improve its workability, hardening and strength characteristics.

METHODOLOGY

This study was set out to investigate the correlation of calcined mussel shells powder (MSP) replacement rate with concrete performances. The laboratory test procedure were based on BS EN 12350-1:2009, BS EN 12350-2:2009 and BS EN 12350-6:2009. The chemical reaction during reversible calcinations carbonation process of mussel shell is represented by the following equation [33];

$$CaCO_3 \leftrightarrow CaO + CO_2$$

(Equation 1)

According to Wang et al. [34] calcination is described as a five-step process which involves (i) heat transfers from the surrounding environment to the boundary layer of $CaCO_3$; (ii) heat transfers through the porous lime layer to the reaction interface; (iii) $CaCO_3$ decomposition; (iv) diffusion of CO_2 produced at the reaction interface to the particle surface through the product layer and (iv) CO_2 evaporation from the surface of the particle to the surrounding gas flow. Furthermore, the calcination process was fixed at their optimum conditions. Moreover, during the capture of CO_2 by lime considerable amount of heat is evolved and the decomposition of calcium carbonate is an endothermic reaction [35].

The control and modified concrete specimens were manufactured by using YTL Ordinary Portland Cement (Type I) as shown in Table 2, river sand as fine aggregates (specific gravity of 2.5 and size of 0.4 mm), local harvested mussel shell and distilled water. Distilled water was used to ensure salts and other impurities were removed.

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	

In this study, Asian green mussel (Perna viridis) and procured from local seafood vendor in Pantai Remis, Kuala Selangor. The preparation of calcined mussel shells powder (MSP) was adopted from Adewuyi et al. [35] and Islam et.al [36]. The shells were washed with tap water to remove dirt and boiled for 2 hours to remove the odour and remaining traces of organic substance. After the washing process was done, the mussel shells were air dried for 12 hours. The shells then calcined at the temperature of 550°C for 2 hours [37]. The calcined mussel shells then crushed and sieve in according to BS EN 933-1:2012.

The concrete rheological properties were obtained through slump test. Slump test is an empirical test to get an ideal amount of water during 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 ratio. Study by Dennis & Pacciani [7] pointed out that low humidity or insufficient water amount in the pores hindering CO_2 to form carbonic acid and react with the alkaline cement hydrates. As the humidity becomes low, the diffusion rate of CO_2 will also decreased.

In the experimental method for modified concrete, the MSP were directly substituted for the cement at 10%, 20%, 30% and 40% (R10, R20, R30 and R40 respectively) by volume. Equation obtained from the slump test was used as the reference for modified formulations. Challenges faced during the process especially to accurately substitute cement powder and MSP. Due to variations of material density, the replacement percentages were calculated by using Relative Bulk Density (RBD) principles established by Razali [39]. This was to ensure accurate volume of specimens were produced between control and modified concretes. Table 3 shows the mix proportions of the specimens.

Sample	Sand (g)	Water (mL)	OPC (g)	Omitted OPC (g)	MSP Replacement (g)
Control	1000	1203.7	833.3	0	0
R10	1000	1203.7	833.3	83.3	89.5
R20	1000	1203.7	833.3	166.6	179.0
R30	1000	1203.7	833.3	250.0	268.4
R40	1000	1203.7	833.3	333.2	357.8

The concrete mixtures were casted in 40mm x 40mm x 40mm of polystyrene mould. The mould then been tampered for a few times to eliminate bubbles. The specimens were demoulded 24 hours before set to curing in water. Testing days were conducted on 7th, 14th, 28th and 56th days. Concrete's strength increases with age (time) through hydration process that

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require moisture (water) and a favourable 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. [40], concrete properties are significantly influenced by curing since it greatly effects 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, 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 [41]. The conversion of calcium hydroxide (Ca(OH)₂) in the concrete into calcium carbonate (CaCO₃) from absorption of CO₂ over several decades further strengthens the concrete and makes it more resistance to damage. However, the carbonation reaction lowers the pH of cement pore solution and can corrode the reinforcement of reinforced steel in concrete [42].

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.

pН

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 [43] was used. The concrete specimens were manually cut and then were crushed into a powder. 15g of powder were mixed with 15g of distilled water. The pH of the solution then was measured by using pH meter (Mettler Toledo).

Carbonation Depth

The depth of carbonation was investigated upon each mortar sample at 7, 14, 28 and 56 days. The samples were sprayed with phenolphthalein solution (Fisher Chemical) [44]. The resulting pink stained on the indicated the carbonation activity. The average carbonation depth was measured by digital calliper (to the nearest 0.01 mm).

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 will be estimated to have variable strength as well. Therefore, the interaction of the MSP 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 state.

Sorptivity (Capillary Absorption)

For this study, sorptivity test was conducted by adopting the direct gravimetric method [45]. 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. The specimens were then weighed to determine the changes in weight over 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/m^2 against the square root of time $t^{1/2}$. The sorptivity was then determined from the gradient of the slope [46].

The capillary suction of the modified concrete interface relates to the sorptivity process, but having a discontinuous contain pores which are discontinuous and do not allow water movement by capillarity, and hence do not contribute to sorptivity [47]. Factors that influence the sorptivity of the water into concrete structure are the pore size of the concrete. Larger pore size of the concrete gives higher diffusion of water through capillary action gravitational force.

RESULTS AND DICUSSIONS pH

Increasing day of curing reduce the pH of concrete due to the carbonation process [28]. From the results, that there are no significant different of pH between MCs and Control specimens but evident trend can be established. All fresh mixed cements have a pH of 12. However, once the carbonation reaction takes places, pH reading becoming neutral (pH 7). Carbonation progressively lowers the pH in mortar with maximizing the amount of Ca^{2+} ions. The pH variations could be attributed to differences in materials characteristics and their properties with a corresponding influence on the extent of carbonation of the concrete.

Carbonation Depth

As can be seen in Figure 1, carbonation depth increased with respect to time. This is due to the reaction of atmospheric CO_2 with the $Ca(OH)_2$ inside the cement structure producing $CaCO_3$. This reaction keep continuously undergoes inside the concrete structure until they completely carbonated. This reaction reduced the pH of concrete in increasing of carbonation time. Other study by Chang [29] shows the same evidence where the concrete that exposed to atmosphere in long period of time will reduce the pH of concrete.

It were apparent that all formulations had 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 6.7 mm on day 7 to 9.7 mm on day 56. Whilst the highest carbonation depth detected at R40 specimens in which it carbonated 8.8 mm at day 7 to 11.9 mm at day 56. This

observation is consistent with the carbonation principles that carbonation depth increases with time. During carbonation process, the CO_2 in surrounding environment is enough to make calcium hydroxide (Ca(OH)₂) reacted as explained based on Equation 2;

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

(Equation 2)

Study by Somerville [31] found that the normal concrete will only carbonated about 5 - 50 mm annually. The application of MSP in addition to cement as binding materials produced more $Ca(OH)_2$ in the concrete composition hence reacting more with atmospheric CO₂ to be carbonated [33]. This resulting in all Ca compounds dissolving and finally forming calcium carbonate. The porosity of the concrete also influences the carbonation depth. Increase in proportion of MSP shows higher carbonation depth due to the pore size of the concrete that allow diffusion of CO₂ in concrete structure through the water in a form of carbonate ions that will react with the Ca ions of the pore water [7]. The higher the carbonation depth, the higher the amount of CO₂ absorbed. The absorption of CO₂ inside the surface of the concrete was influenced by the amount of the atmospheric CO₂ inside the concrete, the humidity and also the temperature during the curing process.



Figure 1: Carbonation depth of concrete vs. curing time

Compressive Strength

The compressive strength results of the modified concretes (MCs) are presented in Figure 2. Overall, the MCs were indicated to be able to resists load better than Control specimens. All MCs reached rapid strength development for the initial 14 days of test. On day 14, the highest compressive strength was achieved by R10 at day 14 (12.68 N/mm²). However, the anomalies trend of strength development were noticeably identified between day 7 and day 28. There were no significant strength developments of MCs occurred after 28 days although the Control specimens kept on increasing its strength in small magnitude.



Figure 2: Compressive strength of concrete

This results indicates that the strength of the MCs were depending on the amount of replacement in which less replacement percentage yielded higher strength. Poor bonding properties of the concrete matrix is identified as the major contributing factor. The aragonite polymorphs from MSP influenced low strength development due to its different symmetry and crystal shapes to calcite (from cement). On the other hand, MCs with low amount of sand leads to segregation and bleeding of concrete. Adding 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. The failure of the concrete at certain age depending on the breakdown bond between the binder and the paste. Therefore, there might not be enough bond between MSP particles and cement paste to produce sufficient bond strength and ultimately sufficient compressive strength of the concrete. Study from Mindess et al. [38] has highlighted that material with rough textured surfaces will improve the mechanical component of the bond. It is justified since too much of MSP which means higher effective surface area, would lead to insufficient proportion of cement thus leading to poor bonding properties of the matrix with aggregates thus exhibit lower strength. These results seemed to be consistent with other research which found that the stability of the binder and aggregates influence the concrete strength [47]. The different characteristics in cement hydrates products which is calcium silicate hydrates (C-S-H) in MSP

and cement also influenced the longer-term stability of the concrete as hhigher surface area of the hydrate cement product gives higher in strength development of concrete [48].

Sorptivity (Capillary Absorption)

According to Hall & Hoff [49], the sorptivity of concrete is influenced by the materials behaviour, with regards to variations in composition and curing conditions, in a rational way. Figure 3 presents the data of average sorptivity value of MCs in different period of curing in which the Control specimens has the higher sorptivity values that indicated higher porosity value than MCs. MCs also exhibited decreasing sorptivity values. MCs with higher replacement percentages were more compact and have less capillary suction activity due to the less size of pore. This is due to the accelerated carbonation activity of the MCs throughout the testing period.



Figure 3: Sorptivity

R40 yielded the lowest sorptivity value due to the less presence of pore on the surface of cross sectional area. This proves that feature of the matrix structure of concrete that relates to the transport of water is pore system of cement paste, particularly on the aggregate-paste interface environment. This is supported by water movement in concrete predominantly controlled by the bulk of the hardened cement paste, which is the continuous phase in concrete. The low volume of cement presence contain in the hardened concrete gives lower sorptivity activity of water throughout the concrete surface.

CONCLUSION

The most obvious finding to emerge from this study is that the modified concretes with MSP replacement performed better than Control. These results are consistent with each tests in which the modified concrete performances are strongly depending on the replacement rate. Higher replacement percentage is beneficial for acceleration rate of pH, carbonation and compressive strength. However, in compressive strength, the modification is only efficient at lower replacement rate. This is because the extra amount of MSP as led to imbalance proportion of binder thus leading to poor bonding properties of the concrete matrix. Modification is not required if the specifier is looking for the increase of sorptivity performance. These outcomes have important implications for future applications and the adaptability of alternative binder through the understanding on the modified concrete formulation, specification and product design of cement materials and the revalorization of aquatic wastes.

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