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Suitability of phenolphthalein indicator method for alkali activated concrete

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Abstract - Alkali activated cementitious materials (AACM) concrete is a sustainable alternative to Portland cement PC concrete by reducing CO2 emitted during the production of PC by 60%. Fundamental investigations on alkali activated concrete (AACM) has been on their engineering properties with less emphasis on the service life and durability properties. The carbonation effect on concrete is crucial for its service life prediction. This paper investigates the suitability of phenolphthalein indicator method to determine the carbonation front in AACM. The geopolymerisation products of AACM are different from the hydration products of PC concrete and consequently, the carbonation process of these two types of concrete is expected to be different. Three mixes of AACM 1, 2, 3 and control PC concrete were produced and cured in water ($20 \pm 2^{\circ}$ C) for 27 days and then in laboratory air ($20 \pm 2^{\circ}$ C, 65% R.H) for 42days. A total of twenty-four cylindrical specimens with 50mm diameter X 60mm depth were produced. All the specimens were exposed to 5% CO₂ inside a carbonation chamber at 20 \pm 2°C and 65% R.H for 327days. Carbonation depths were determined by phenolphthalein indicator method on twelve specimens. Powder samples were obtained from the carbonated and non-carbonated zones of the other twelve specimens to measure the pH of the carbonated concrete and powder. Results show that the pH of the carbonation in AACM concrete and mortar are above threshold 9 while it was below 9 in PC concrete. This suggests that phenolphthalein indicator method is inappropriate to determine the carbonation of alkali activated cementitious materials (AACM) concrete

Keywords: Alkali activated cementitious materials (AACM) concrete, carbonation depths, phenolphthalein indicator method, carbonated and non-carbonated zones.

1 Introduction

Research findings show attractive attributes of alkali activated cementitious materials (AACM) concrete as a construction material over Portland cement (PC) concrete. These attributes include low CO₂ emission and energy demand during its production [1, 2], superior strength and pore properties [3], lower bound chloride ingress [4]. However, limited knowledge on the durability properties and in-service performance of AACM exists in literature. Field applications of AACM are restricted due to the limited knowledge of its structural behaviour when subjected to exposure to corrosion initiators such as carbonation. Carbonation in PC concrete reduces the pH of its pore solution to below 9 [5, 6]. This occurs when atmospheric carbon dioxide, CO₂, dissolves in the concrete pore solution to form carbonic acid HCO₃ which then reacts with the main hydration products of concrete, Ca(OH)₂ and C-S-H, to form calcium carbonate CaCO₃ [7, 8]. The hydroxyl ion (OH)₂ within the pore solution is displaced by this reaction thereby depleting the protective passive film around the reinforcing steel in concrete. The progression of these reactions results in carbonation induced corrosion of reinforcement in the presence of oxygen and water. AACM concrete does not contain calcium hydroxide, Ca(OH)₂, which can react with carbonic acid, HCO₃, to produce calcium carbonate CaCO₃, unlike PC concrete which has calcium hydroxide Ca(OH)₂ as its hydration product. The reaction of atmospheric CO₂ with the geopolymerisation and hydration compounds of AACM and PC concrete respectively is dependent on the amount and type of these compounds.

The carbonated zone of PC concrete is determined by the phenolphthalein indicator test [9]. Spraying phenolphthalein solution on carbonated concrete surfaces indicates the depletion of $Ca(OH)_2$ within the concrete matrix, which is not present in AACM concrete. Therefore, the use of phenolphthalein indicator method to determine the carbonation front may not give a true extent of carbonation in AACM concrete. The suitability of the phenolphthalein indicator method for AACM concrete will be addressed in this study.

The chemistry of carbonation in PC concrete is fairly well documented while limited knowledge is available for AACM concrete [10]. Like other durability properties, the carbonation of AACM is significantly influenced by its pore chemistry which is significantly different from that of PC concrete. The mechanism of carbonation in PC concrete is represented in equation 1 -3:

$$H_2O + CO_2 = HCO_3^{-}(Bicarbonate ion) + H^+$$
(1)

$$HCO_3^- = CO_3^{2-}(Carbonate ion) + H^+$$
(2)

$$Ca(OH)_2 + 2H^+ + CO_3^{2-} = CaCO_3 + 2H_2O$$
 (3)

Bicarbonate ions are formed when CO₂ reacts with water at the carbonated zone (equation 1). These bicarbonate ions dissociate near the non-carbonated zone within the PC concrete to form carbonate ions due to the high pH of the pore solution (equation 2). The carbonate ions precipitate as calcium carbonate (CaCO₃) crystals when they react with the hydration product of PC concrete (Ca[OH]₂) (equation 3). These crystals are present in PC concrete in two forms: vaterite and calcite, the metastable vaterite turns to calcite over time [11]. This process will continue until all the hydration product, Ca(OH)₂, of PC concrete is consumed by the carbonate ions. The pH of PC concrete drops because of the low calcium ions present in the pore solution due to this process. The phenolphthalein test for PC concrete is suitable for detecting these chemical layers in PC concrete.

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The carbonation effect on AACM concrete by using the accelerated testing method may have overestimated its actual degradation in service life [12]. A change in AACM pore solution equilibria may induce accelerated carbonation compared to under natural carbonation giving a higher apparent carbonation rate [12]. Published results on the carbonation of AACM concrete are inconsistent in comparison with PC concrete. For example, high depths of carbonation were recorded for silicate-activated blast furnace slag concrete compared with PC concrete under accelerated carbonation conditions 10-20% CO₂ and 70% R.H. [13, 14]. Other studies show that the depth of carbonation of alkali activated blast furnace slag concrete and mortar is comparable to those of PC concrete or mortar [15]. The author [15] concluded that the refinement of the pore structure of alkali activated blast furnace slag concrete is responsible for its carbonation resistance, however, a high relative humidity of 90% was used for the accelerated carbonation test which would slow the diffusion of CO₂ within the concrete matrix and invalidate the author's results.

The rate of carbonation in concrete is a slow process which sometimes takes years to manifest. Concrete structures in large cities are susceptible to carbonation due to human activities involving high emissions of CO_2 , up to 1% by volume of air [16]. The carbonation chamber used in the laboratory for accelerated testing was maintained at 5% CO_2 concentration, 65% relative humidity and a temperature of $20 \pm 2^{\circ}C$ to achieve the optimum rate of carbonation in both AACM and the control PC concrete. The diffusion parameters of the concrete pores have considerable influence on the rate of carbonation. The diffusion rate of CO_2 within a concrete matrix is four orders of magnitude slower than O_2 when its pores are fully saturated while its diffusion remains inactive when the concrete pores are insufficiently saturated [16–18].

2 Experimental Programme

2.1 Materials and mixes

Details of AACM mixes 1, 2 and 3 and the control PC concrete are given in Table 1. AACM and PC concrete were produced from ground granulated blast furnace slag (GGBS) and ordinary Portland cement CEM 1 of grade 42.5R [19] respectively. 10 mm uncrushed gravel, 6 mm limestone and a medium grade sand of 80% particle size passing 1mm sieve were used as coarse and fine aggregates. The activator dilutions of 0%, 3.88% and 7.76% were used to prepare AACM 1, 2 and 3 mixes respectively. Liquid/binder ratio 0.47 was used for both AACM and PC concrete mixes. AACM mixes contained 2% by binder weight of shrinkage reducing admixtures (SRA) made from Alkyl-ether and 0.75% by binder weight of retarder R42 made from a blend of high grade polyhydroxycarboxylic acid derivatives. The shrinkage reducing admixture also enhances workability while the retarder reduces the setting time of AACM concrete.

Table 1: Composition of AACM 1, 2, 3 and the control PC concrete

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Mix	Binder Content (%)	Fine Agg. (%)	Coarse Agg. (%)		Liquid/	Activator	R42	SRA
			10mm Gravel	6mm Limestone	Binder Ratio	Dilution (%)	(% by weight of binder)	
AACM 1	25	18	29.3	15.7	0.47	0	0.2	0.5
AACM 2	25	18	29.3	15.7	0.47	3.88	0.2	0.5
AACM 3	25	18	29.3	15.7	0.47	7.76	0.2	0.5
Control PC	20	26	28.9	15.5	0.47(w/c)	-	-	-

*R42 is the retarder; SRA is the shrinkage reducing admixture

2.2 Specimen preparation

The concrete specimens were cast in a 0.0625^{3} (150kg) capacity Cretangle mixer. AACM and PC concrete specimens were cast in plastic cylinders of 50mm diameter X 60mm depth as shown in Fig. 1 in two layers and compacted on a vibrating table. Self-adhesive bitumen tape was fixed at the bottom of the plastic cylinder moulds before casting to prevent the concrete from pouring out (Fig. 2). The specimens were cured in the laboratory air ($20 \pm 2^{\circ}$ C, 65% R.H.) for 24hrs while covered with polythene sheets. They were left in the moulds to prevent the ingress of CO₂ through the bottom face and circumference of the specimens. The specimens were wet cured for 27days at a temperature of $20 \pm 2^{\circ}$ C followed by dry curing for 42days at 65% R.H. and a temperature of $20 \pm 2^{\circ}$ C until the change in their unit weight was less than 0.2% in a 24-hour period to reduce its moisture content. The specimens were then placed inside the carbonation chamber exposed to 5% CO₂ concentration at 65% relative humidity and temperature of 50°C for 327days. After exposure to 5% CO₂ concentration for 327days, the specimens were grouped into two batches. The first batch of AACM and PC concrete specimens were collected at the carbonated and non-carbonated zones in the second batch for pH analysis. A total of 24 specimens were produced for both AACM and the control PC concrete, 6 specimens per each mix in Table 1.





Fig. 1: Casting concrete specimens in cylindrical Fig. 2: Self-adhesive tape fixed at the bottom of plastic moulds

cylindrical moulds

2.3 **Test Procedure**

The cylindrical specimens were removed from the carbonation chamber after 327days exposure. These specimens were removed from their plastic moulds (sleeves) and split into halves vertically along their depths as shown in Fig. 3. The splitting tensile strength procedure was adopted to break the cylinders into two longitudinal halves. The depth of carbonation was determined by spraying phenolphthalein on the exposed (split) surface of one broken half of each cylinder according to standard procedure [9]. However, phenolphthalein was not applied to the surface of the second half of each cylinder as shown in Fig. 3.

The depth of carbonation profile obtained from the first half cylinder was marked on the second half of the cylinder that was not sprayed with phenolphthalein. This was carefully chiselled out to obtain concrete chunks of the carbonated and non-carbonated zones. This procedure was performed on six specimens for each AACM and PC concrete to obtain enough concrete chunks for the test. The chunks were ground to powder and passed through a 150 µm sieve to obtain concrete powder for the carbonated and noncarbonated zones (Fig. 4). A second set of powder test samples was similarly obtained by removing the coarse aggregate particles from the concrete chunks. The concrete pieces were crushed to separate the coarse aggregate particles from the matrix and they were removed by sieving. The remaining mortar material was ground to obtain mortar powder for the carbonated and non-carbonated zones. pH analysis was performed on these carbonated and non-carbonated powder samples.

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Fig. 3: Splitting specimen into halves and spraying one half Fig. 4: AACM and OPC concrete/mortar powder with phenolphthalein solution

stored in air-proof plastic vials

2.3.1 pH Analysis

The ex-situ leaching method of concrete [20] was adopted to obtain the solution used for the pH analysis of the concrete and mortar samples. This was carried out by dissolving 5grams of the powder sample in distilled water at a liquid/solid ratio of 1:1 in an air-tight plastic vial. The solution was shaken thoroughly for 2mins to ensure a homogenous mix of the powder. The powder solution in an air-tight plastic vial was left undisturbed for 5hrs to allow for leaching. The concrete powder solution was then filtered to obtain a solution that does not contain powder particles. A double junction electrode connected to a benchtop meter 3-in-1 was dipped inside the filtered solution to measure its pH. This device measures pH ranging from 0.00 to 14.00 and can measure sample volumes as small as 0.2mL with an accuracy of \pm 0.01.

3 **Results and discussion**

3.1 Depth of carbonation

The carbonation depths of AACM and PC concrete is shown in Fig. 5. The carbonated and noncarbonated zones are marked on the broken faces of both AACM and PC concrete (Fig. 5).

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Fig. 5: showing the carbonation and non-carbonated zones in AACM and PC concrete

Fig. 5 shows that the depths of carbonation front which occurred at the two opposite faces exposed to CO_2 while the other opposite two faces coated with bitumen paint have insignificant carbonation front. This shows that the bitumen paint is an effective barrier to CO_2 diffusion in concrete. The presence of optimum moisture content and CO_2 within the concrete matrix resulted in the decalcification of the Carich gel binder within the concrete matrix.

Increase in the depth of carbonation is observed in AACM concrete than the control PC concrete as shown in Fig. 5. The average carbonation depth in AACM 1, 2 and 3 are 25.19, 26.75 and 29.25mm respectively while it is 14.13mm for PC concrete after 327days exposure.

Increase in the depth of carbonation was observed for water-glass activated slag mortar [21]. The author [21] observed a much deeper and intense carbonation between 28 and 120 days while moderate increase in the carbonation depth was observed between 120 and 240 days. The specimens were cured in a closed chamber containing K_2CO_3 solution, kept at a relative humidity of 43.2%. The chamber was saturated with CO_2 twice daily with unknown concentration of the CO_2 .

3.2 pH of carbonated zone

The pH of the powder solutions extracted from AACM and PC concrete and mortar at the carbonated zones is shown in Fig. 6.

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Fig. 6: pH of AACM and PC powder solutions after 327days exposure to 5% CO₂.

The pH of the concrete powder solutions at the carbonated zones of AACM 1, 2, 3 and PC concrete are above the carbonated threshold of 9 as shown in Fig. 6. The pH values are 10.62, 10.46, 10.37, 10.25 and 9.56 for AACM 1, 2, 3 and PC concrete respectively. The pH of the mortar solution is also greater than the threshold value of 9 for all AACM mixes whereas the PC mortar has a pH value significantly lower than the threshold of 9. The high pH exceeding the carbonation threshold in AACM mortar solution is an indication that the phenolphthalein indicator method fails to detect carbonation in AACM concrete compared with PC concrete having pH of 8.69. The phenolphthalein indicator method produces no colour change when the pH of the pore solution is about 9 for PC concrete at the carbonated zone. However, in the case of AACM the phenolphthalein indicator method show no colour change when the pH of pore solution is above 10 at the carbonated zone (Fig. 6). The colourless change at the carbonated zone indicates the absence of $Ca(OH)_2$ [16]. Since $Ca(OH)_2$ is absent from the AACM pore solution, an alternative method besides using the phenolphthalein indicator method for assessing the carbonation in AACM is required.

The pH of mortar solutions at the carbonated zones for both AACM and PC are lower than that of the concrete. The pH of the mortar solutions of AACM 1, 2, 3 and PC at the carbonated zones are 10.33, 10.02, 10.1, 10.05 and 8.69 respectively. The difference in their solution pH is due to the release of alkaline content by the aggregate present in the concrete. PC concrete which had the highest aggregate content of 70.4% compared with 63% for AACM (Table 1) shows the highest pH difference between the solution extracted from concrete powder and mortar powder. The differences in pH between concrete and mortar for AACM 1, 2, 3 and PC concrete are 4.2%, 2.6%, 1.95% and 9.1% respectively.

3.3 Activator Dilution

The influence of activator dilution on the pH of powder solution from AACM concrete and mortar at the carbonated zones is shown in Fig 7.



Fig. 7: Influence of activator on the pH of AACM concrete and mortar solutions at carbonated zones

The difference in the pH of solutions extracted from AACM concrete and mortar powder at the carbonated zones is fairly constant at different activator dilution (Fig. 7). This is because the aggregate content of 63% is constant in AACM 1, 2 and 3 mixes are the same (Table 1). The release of alkaline content by the aggregate present in the AACM 1, 2 and 3 accounts for the increase in the pH of solutions extracted from the AACM mortar. On the other hand, carbonation reacts chemically with AACM mortar while the aggregate in the concrete will influence the diffusion rate of CO₂ and moisture due to the interfacial transition zones around the aggregate.

The pH of solutions extracted from AACM concrete and mortar powders is highest at the lowest activator dilution (Fig. 7). The differences in the pH of AACM concrete and mortar solutions for different activator dilution are attributed to their porosity [3] which affected the rate of carbonation. AACM with 0% activator dilution has the lowest porosity of 4.64%, followed by 3.88% activator dilution which has 6.67% porosity while 7.76% activator dilution has the highest porosity of 7.71% under 3days wet curing ($20 \pm 2^{\circ}$ C) following 24days dry curing ($20 \pm 2^{\circ}$ C, 65% R.H) [3]. The corresponding pH values of AACM concrete are 10.46, 10.3 and 10.25. The other factor influencing the difference in pH in both AACM concrete and mortar solutions is the aggregate content. Yinghong [22] studied the leaching of alkali content of an open graded recycled coarse aggregate and the adverse effect of carbonation on the pore solution. The author [22] observed an increased pH of the pore solution due to leaching of alkali content from the recycled coarse aggregate. The alkali content of the recycled coarse aggregate is consumed during carbonation. However the release of pH-dependent constituents that cause the drop in pH is delayed by the dense structure of concrete [22]. AACM represented in Fig 7 is denser at 0% activator dilution than at

7.76% activator dilution, which is the reason for the differences between the pH of concrete solution and mortar solution at higher and lower activator dilution.

3.4 Relationship between carbonation depth and porosity

The relationship between carbonation depth at 327days exposure to 5% CO_2 and effective porosity of AACM 1, 2 and 3 mixes at 28 days is shown in Fig 8. Details of the pore properties and the effective porosity of AACM mortar mixes are reported in the authors' previous publication [3]. The following linear relationship is established between the effective porosity and the depth of carbonation with a correlation of 0.98:



Fig. 8: The relationship between depth of carbonation dk and effective porosity of AACM concrete

 $d_k = 0.5929(P) + 1.2806$ with $R^2 = 0.98$.

where; d_k is the depth of carbonation (mm) and P is the effective porosity (%).

The depth of carbonation increases with greater porosity. Concrete diffusivity which is a function of porosity is a major controlling factor for carbonation [16]. The ingress of CO_2 and moisture from the environment into the concrete is inhibited by decreasing porosity. The carbonation process is complicated because it involves the transport of liquid and gas which cannot be represented simply with Fick's law [23]. Lagerblad [11] stated that it is difficult to apply the Fick's law equation because of the simultaneous inward and outward diffusions involving carbonate and calcium ions. The pore structure is altered during the inward and outward diffusions involving carbonate ions and calcium ions by reacting with the concrete matrix. For PC concrete, a dense pore structure evolves as the outward diffusion of CaCO₃ forms precipitates that block the concrete pores. In the case of AACM, the calcium ions from the environment. Whilst the chemical compound (CaCO₃) formed during carbonation in PC concrete blocks the pores, the disintegration of calcium ion due to decalcification in AACM concrete enlarges the pores.

4 Conclusions

This paper investigates the suitability of using phenolphthalein indicator method to determine carbonation front in alkali activated concrete AACM. Twenty-four AACM and PC concrete were produced and cured in water ($20 \pm 2^{\circ}$ C) for 27days and then in laboratory air ($20 \pm 2^{\circ}$ C, 65% R.H) for 42days. These specimens were exposed to 5% CO₂ inside a carbonation chamber at $20 \pm 2^{\circ}$ C and 65% R.H for 327days. Phenolphthalein indicator method was used to determine carbonation front of the AACM and PC concrete specimens. Powder samples were collected from the carbonated zones and their pH was determined. The following conclusions can be drawn from the study.

- The pH of mortar solutions at the carbonated zones for alkali activated cementitious materials (AACM) concrete is greater than the threshold of 9 while the corresponding PC mortar is below 9. For example, the pH of mortar solutions of AACM 1, 2, 3 and PC at the carbonated zones are 10.33, 10.02, 10.1, 10.05 and 8.69 respectively. This suggests that the application of phenolphthalein solution on faces of AACM produces pH higher than 9. The phenolphthalein method of determining the carbonation in AACM does not give a true representation and may not be an appropriate method for investigating the carbonation depth.
- The release of alkaline content by the coarse aggregate present in the AACM and PC concrete results in higher pH in concrete than the mortar excluding the coarse aggregate particles. The difference is greater with increasing coarse aggregate content.
- Activator dilution with water increases the pH of carbonated AACMs (mortar and concrete). The porosity of AACMs also increases with activator dilution as they both provides a linear relationship between the two parameters. For example, AACM with 0% dilution has the lowest porosity of 4.64%, followed by 3.88% activator dilution which has 6.67% porosity while 7.76% activator dilution has the highest porosity of 7.71% under 3days wet curing ($20 \pm 2^{\circ}$ C) and 24days dry curing ($20 \pm 2^{\circ}$ C, 65% R.H). The corresponding pHs of AACM are 10.46, 10.3 and 10.25.
- The relationships between porosity and depth of carbonation in AACM concrete is as follows: $d_k = 0.5929(P) + 1.2806$ with $R^2 = 0.98$.

where; d_k is the depth of carbonation (mm) and P is the effective porosity (%).

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