Development of a short time model for predicting chloride ingress into normal and pozzolanic concrete

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Abstract. One of the major origins of corrosion in concrete reinforcing steel is chloride ion penetration. This process deteriorates concrete structures in many parts of the world. Chloride can penetrate into concrete structures mainly through diffusion and absorption. A good percentage of research work has been carried out on diffusion mechanism and the process even though slow is a well understood one. Conversely, few research work has been reported concerning absorption mechanism of chloride ion penetration in concrete. This is a fairly rapid transport process whose mechanism needs to be understudied. For this study, concrete cube specimens containing 100% OPC, 12% BLA and 12% GSA of mix 1:2:4 were prepared. The workability of the various mixes was obtained through slump and compaction factor tests. The hardened cubes, 150 mm³, were thereafter immersed in sodium chloride (NaCl) solution for the specified durations. Compressive strength, absorption and spray tests were carried out on the designed specimens at 28, 56 and 90 days. Numerical models were developed using data obtained from the various tests to predict the depth of chloride penetration into normal and pozzolanic concrete under the condition used in this study. The developed models indicate that absorption has substantial effect on chloride ingress. The models’ outputs further propose that the thickness of concrete cover in structures exposed to chloride attack should be greater than the present recommended values in use. However, thickness of concrete cover has practical limits based on the design and analysis, consequently, partial replacement of cement with pozzolans (Bamboo Leaf Ash and Groundnut Shell Ash) should be adopted.

Keywords Chloride ion ingress, Pozzolanic concrete, Concrete model, Compressive strength

1. Introduction

Reinforced concrete structures are often designed for safety and functionality for a long duration. Nevertheless, this long designed life can be truncated when reinforced concrete structures are exposed to chloride contaminated environments. In this case an important deterioration phenomenon that must be taken into account is corrosion of steel reinforcements due to chloride ions. When chlorides travel through concrete to meet the reinforcing steel bars in the presence of sufficient oxygen and moisture, corrosion is initiated [1]. Civil engineering structural elements like bridges and other marine structures that are subjected to repeated wetting and drying conditions are most susceptible to corrosion damage. Many concrete highway bridges designed for over 100 years life span experienced failure only after spending about one quarter or one third of their designed life span [2].
When steel undergoes corrosion, it occupies a larger volume than the initial steel thereby causing cracking, spalling or dilapidation of the surrounding concrete. This weakened the bond connecting the concrete to the reinforcement which in turns make it easier for destructive agents to have a direct contact with the steel thereby increasing the rate of corrosion. Corrosion also reduces the load carrying capacity of the steel while the compressive strength of the surrounding concrete will also be drastically reduced through the action the chloride ion on it [3,4]. If reinforced concrete structures like bridges and other seashore structures must achieved the desired service life, then there is need to impede the penetration of chloride ion into the concrete. One of the functions of concrete in reinforced structure is to serve as protection to the reinforcing steel; the more resistance the concrete to external aggression the better it is for the embedded reinforcement. A reliable solution to this problem is engaging the use of high quality and impervious concrete. Less pervious concrete can be achieved through the use of pozzolanic blended cement concrete and low water cement [5,6]. Alternative solution is by increasing the depth of the concrete cover to reinforcement but there is a limit to which this can go so as not to impair the tensile ability of such reinforcement thereby leading to surface cracks with increase width [7].

Standards and codes such as the British Standard [8] provides specification for different composition of concrete and the cover depth depending on the designed service life of the structure, degree of aggressiveness of the environment and the type of structure itself. Nevertheless, some chronic aggressive environments have defiled this specification; in that structures built in such environments have been reported to have experienced high level of corrosion and failure within a short period of time [9]. Therefore, considering this problem and limitation of concrete cover solution, the need to develop dependable models that can predict the depth of chloride penetration as a function of some important concrete properties and selected transport processes applicable to chloride penetration is critically essential.

Chloride ion ingresses into concrete either though diffusion or absorption. Diffusion takes place when there is chloride concentration gradient. Diffusion continues, provided there is chloride in the concrete in the presence of the pore liquid and it is as a slow process. Absorption takes place when there is tension at the surface of the concrete. The liquid through capillary forces will be drawn into unsaturated or partially saturated pores in the concrete [10]. The available prediction models for chloride ingress in concrete by past researchers concentrates mainly on the use of diffusion equation neglecting the absorption properties of chloride into the concrete. Predicting models showing interaction among absorption, depth of chloride penetration and some other properties of concrete (like compressive strength, curing age among others) subjected to chloride attack have not been established yet. Studying and providing predictive models in term of absorption characteristics will be a bonus and advanced development in mitigating corrosion in the concrete construction industries.

Pozzolanic cements when blended with ordinary Portland cement in producing pozzolanic concrete has the ability to improve the resistance of such concrete to chloride ion penetration and other aggressive media thereby increasing the durability of the produced concrete [11, 6, 12]. Commonly used pozzolans include: rice husk ash (RHA), silica fume (SF), locust beans pod ash (LBPA), pulverized fuel ash (PFA), bamboo leaf ash (BLA), sugarcane bagasse ash (SBA) amongst others [13,14]. Ordinary Portland cement (OPC) is naturally susceptible to attack by aggressive media, for example chlorides, sulphates, carbonation or combination of any of these depending on the residing environment [15].

Consequently, this research work evaluated the effects of absorption and pozzolans on chloride ingress. Numerical models were also developed for predicting chloride ingress into normal concrete as well as pozzolanic concrete.

2. Materials and methods

2.1 Materials
Lafarge elephant cement was used as the Ordinary Portland Cement (OPC). The pozzolans used for this research work were Bamboo Leaf Ash (BLA) and Groundnut Shell Ash (GSA). Bamboo leaves were obtained within the premises of the Federal University of Technology, Akure and groundnut shells were
obtained from a local groundnut producing industry in Oyo State. The bamboo leaves and groundnut shells were adequately dried and burnt to ashes at a controlled temperature of 600°C in a furnace [16]. The ashes were thereafter sieved to obtain the pozzolans. The coarse aggregates used were granite chippings of grading between 10 to 20 mm while the fine aggregates used were river sand. Sodium chloride (NaCl) solution was used as the curing medium for chloride ingress. Epoxy resin was used as the coating to prevent moisture loss through evaporation process and to ensure that sodium chloride solution penetration occurs in one-directional. Silver Nitrate (AgNO₃) with a concentration of 0.1M was used as the colorometric solution to evaluate the depth of chloride penetration.

2.2 Mix design and concrete specimens preparation
The mix design consists of three groups of samples which were OPC (containing 100% ordinary Portland cement), 12% BLA (replacing cement with 12% Bamboo Leaf Ash) and 12% GSA replacing cement with 12% Groundnut Shell Ash). Ikumapayi [12] in her research found that partial replacement of BLA and GSA with OPC at 12% gives optimum compressive strength and better durable concrete, therefore 12%BLA and GSA were adopted. Batching was done by weight. A mix ratio of 1:2:4 was used for the concrete mix, the details is as shown in Table 1. Iron cube moulds of 150 mm X 150 mm X 150 mm were used for casting.

| Specimen Group | Mass of cement (kg) | Mass of sand (kg) | Mass of granite (kg) | Mass of water (kg) | Mass of pozzolan (kg) |
|----------------|--------------------|------------------|---------------------|-------------------|---------------------|
| OPC            | 24.89              | 56.33            | 114.05              | 14.93             | -                   |
| 12% BLA        | 21.91              | 56.33            | 114.05              | 14.93             | 2.98                |
| 12% GSA        | 21.91              | 56.33            | 114.05              | 14.93             | 2.98                |

2.3 Concrete specimens preparation for tests
Concrete cubes were cast into 150 mm³ moulds using the mix ratio of 1:2:4 of cement, sand and coarse aggregates respectively. The concrete specimens were demoulded after 24 hours, weighed cured in water by immersion. The concrete specimens were brought out after 28 days of curing, weighed and then transferred to NaCl (sodium chloride) solution. Prior to this transfer, four parallel sides of the cubes were coated with epoxy resin; the coating was applied on the specified sides of the concrete specimens to prevent moisture loss through evaporation process and to ensure that sodium chloride solution penetration occurs in one-direction. After the coating, the concrete cubes were left for some minutes to allow the epoxy resin to cure. The concrete cubes were thereafter immersed in sodium chloride solution having a concentration of 3% NaCl (i.e. 3g of NaCl in 100ml of water). The cubes were left in the NaCl and later brought out for tests at stipulated time of 28 days, 56 days and 90 days.

2.4 Mechanical tests for materials and fresh concrete
2.4.1 Particle size distribution
This test for the sand or fine aggregates was carried out with the use of sieve analysis of various sizes. Percentage passing (or percentage finer) was computed with the use of Equations 1 and 2.

\[
\% \text{ Retained} = \frac{\text{Mass retained (g)}}{\text{Total mass (g)}} \times 100 \tag{1}
\]

\[
\% \text{ Passing} = 100 - \% \text{ Retained} \tag{2}
\]
2.4.2 Specific gravity test
The specific gravity of the fine aggregates ($G_s$) was carried out and calculated using Equation 3.

$$G_s = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)}$$

Where:
- $W_1 =$ mass of glass jar + lid
- $W_2 =$ mass of glass jar + lid + soil sample
- $W_3 =$ mass of glass jar + lid + water + soil sample
- $W_4 =$ Weight of empty jar + lid + water (full)

2.4.3 Aggregate crushing value test
The aggregate crushing value (ACV) was carried out for the coarse aggregates. The ACV is expressed as the ratio of the weight of sample passing the 2.36 mm sieve to the total weight of the sample as a percentage as shown in Equation 4.

$$ACV = \frac{W_3}{W_2 - W_1} \times 100\%$$

2.4.4 Slump and Compacting factor test
The slump test was carried out using Vicat’s apparatus and the height of the cone for various mixes were recorded. Likewise the compacting factors for the various mixes were determined using the standard hoppers and cylinders. The compacting factor value was calculated using Equation 5.

$$\text{Compacting factor} = \frac{m_p}{m_f}$$

Where; $m_p =$ mass of partially compacted concrete, $m_f =$ mass of fully compacted concrete

2.5 Compressive strength test
The compressive strengths of the various concrete cubes were determined using the compressive strength testing machine. The maximum loads at failure were obtained from the load dial gauge of the crushing machine. Individual load was expressed in Newton (N) and was divided by the area of the cube in square millimetres ($\text{mm}^2$) to give the compressive strength of the concrete in N/mm$^2$. The concrete cubes were crushed for each specimen group and the average compressive strength was determined for each group using Equation 6.

$$\text{Compressive strength (N/mm}^2) = \frac{\text{Maximum load (N)}}{\text{Area of cube (mm}^2)}$$

2.6 Absorption test
Absorption test was conducted for the various specimens at the specified mixes i.e. OPC, 12% GSA and 12% BLA. The result was obtained using Equation 7.

$$\% \text{ Absorption} = \frac{\text{increase in weight}}{\text{weight of dry specimen}} \times 100$$

2.7 Determination of chloride penetration
The Depth of chloride penetration was obtained using Silver Nitrate ($\text{AgNO}_3$) solution with a concentration of 0.1 M as the colorimeter indicator. The specimens, after each specified immersion time (i.e 28 days, 56 days and 90 days) were brought out of the NaCl solution, cut vertically and their cut surfaces were sprayed immediately with the colorimetric solution and left for about 5 minutes. The area affected or penetrated by the chloride ion changed colour to white or light grey because of the formation of silver chloride. The various depths of the chloride penetration were measured with the aid of a venier

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calliper as shown in Figures 1 and 2. The colour change was in line with the chemical reaction shown in Equation 8 and the past research work conducted by Otsuki et al. [17], who likewise found that duration of changing colour depends on reaction between chloride and AgNO$_3$ on the sprayed surface.

\[ \text{NaCl} + \text{AgNO}_3 = \text{NaNO}_3 + \text{AgCl} \] (8)

2.8 Scanning electron microscope
Scanning electron microscope was carried out to assess the microscopic structure of the specimens. It also helps to identify the composition and measure the abundance of elements in the sample.

2.9 Modelling of chloride ingress
As mentioned earlier, the process of chloride transportation in concrete is considered to be mainly governed by diffusion, and absorption (sorptivity). The proposed model was developed based on the experimental results and the effect of absorption. Data obtained from the tests were collated and analysed using Microsoft Excel and SPSS software packages. The models were thereafter developed for each sample group from the results of the data analyses. Parameters used in the model formulation include: depth of chloride penetration (d), compressive strength (f$_{cu}$), time (t) and absorption (i), in which the depth of chloride penetration was expressed as a function of compressive strength, time and absorption i.e. \( d = F (f_{cu}, t, i) \).

3. Results and discussion

3.1 Mechanical properties of the materials
The result of the particle size distribution carried out on the fine aggregates are shown in Figure 3. The results revealed that the sand sample was well graded and very appropriate for concrete work. Other mechanical properties obtained for the materials used and fresh concrete are shown in Table 2.
Figure 3. Particle size distribution (PSD) curve of the fine aggregate

| Property                        | Fine aggregates | Coarse aggregates | Fresh concrete | Comment |
|--------------------------------|-----------------|------------------|----------------|---------|
| Specific gravity (Gₚ)          | 2.65            |                  |                | This implies that the fine aggregates are within the acceptable range of 2.55 - 2.80 and therefore suitable for construction purpose. |
| Aggregate Crushing Value (ACV) | 29.8%           |                  |                | The ACV of 29.8% obtained for the coarse aggregates is within the acceptable limit prescribed by BS 882 (1992). This indicates that the aggregate is appropriate for concrete production. |
| Slump                          | 45 for OPC, 40 for 12%BLA and 40 for 12%GSA |                   |                | Workability of the fresh concrete reduces with partial replacement of cement with BLA and GSA. |
| Compacting factor              | 0.98 for OPC, 0.95 for 12%BLA and 0.96 for 12%GSA |                   |                | The compacting factor of the pozzolanic concrete mixes reduces slightly to that of OPC concrete |

3.2 Compressive Strength

The result of the compressive strength test of concrete cubes at 28 days in water is shown in Figure 4. Concrete cubes with 100% OPC were found to possess the highest compressive strength at 28 days in water compared to the pozzolanic concretes (%12 BLA and 12% GSA). This can be attributed to longer time needed by the pozzolans to attain full hydration. Likewise, the results of the compressive strength of the various concrete specimens cubes at 28, 56 and 90 days in NaCl solution are shown in Figure 3. Effect of sodium chloride solution on the compressive strengths of the various concrete cubes is clearly shown. All the concrete mixes, both normal and pozzolanic concrete specimens showed some degree of reduction in the compressive strength. These curves further showed that the longer the duration of various concrete specimens in NaCl the more the reduction of the compressive strength. However, concrete cubes containing 12% BLA shows a better resistance to compressive strength loss in the NaCl solution when compared to OPC and 12%GSA concrete. The compressive strength loss due to chloride ion attack in concrete is in line with the results from past researchers and can be attributed to the interaction of chlorides with calcium-silicate-hydrate (C-S-H) at three different forms. It can be as chemisorbed layer on C-S-H, in the C-S-H inter layer spaces or be intimately bound in the C-S-H lattice. Chlorides have been reported to aid leaching of Ca(OH)₂ and stimulate the formation of pervious
C-S-H involving complex reactions. The leaching of Ca(OH)$_2$, decalcification effects of NaCl as well as the formation of pervious C-S-H all have destructive effects on these concrete specimens [18]. BLA showed better resistance to the chloride attached because of its pozzolanicity effect as well as its degree of fineness as shown in the SEM images in Figures 7 to 9 at the later results. The fineness of the pozzolanic material controls its filler effect and it is a very essential factor for adjusting the aggregate-cement interface zone. Bamboo leaf ash (BLA) with a very minute average particle size has the highest compressive strength and GSA with a high average particle size has the lowest compressive strength. GSA rate of reaction is also lower when compared to BLA.

![Figure 4. Graph of Average Compressive Strength against Age/Curing Medium](image)

3.3 Absorption

The results of the absorption test on the various specimens groups at 28 days in water as well as at 28, 56 and 90 days in sodium chloride solution are shown in Figure 5. The results show that concrete cubes containing 100% cement (OPC) absorbed more water and salt compared to other cubes containing 12% GSA and 12% BLA. This can be traced to the well refined pore structure and consequently reduced permeability of the pozzolanic concrete. Concrete cubes with 12% BLA has a reduced permeability compared to others as revealed in Figure 6. As earlier stated, the fineness of the pozzolanic material controls its filler effect and it is a very essential factor for regulating the aggregate-cement interface zone. Bamboo leaf ash (BLA) with a very small average particle size impeded ingress of both water and salt compared to GSA with a higher average particle size as shown in SEM image in Figures 7 to 9.
3.4 Depth of Chloride penetration

The results of the spray test carried out to evaluate the depth of chloride penetration at 28, 56 and 90 days respectively in sodium chloride solution are shown in Figure 6. The depth of chloride penetration increases as the curing time increases. This is because depth of chloride penetration is a function of the salt solution quantity absorbed and the existing pore space in the concrete. The result shows that concrete cubes containing ordinary Portland cement (OPC) experienced a greater ingress of chloride compared to those containing 12% BLA and 12% GSA as revealed in Figure 6. This can be attributed to the fineness of the cement substitute and its filler effect in making the concrete cubes less permeable to chloride ingress. More so, cubes containing 12% BLA showed a better resistance to chloride ingress compared to those containing 12% GSA. This is owing to the fact that BLA has the smallest average particle size compared to others.
3.5 Results of Microstructure

Figures 7 to 9 show the Scanning Electron Microstructure (SEM) images of OPC, 12% GSA and 12% BLA concrete samples respectively at 28 days of water curing. The SEM photograph of OPC (Figure 7) showed high content of calcium silicate hydrate and calcium hydroxide as a result of hydration as well as large voids and some anhydrous cement particles. Figure 8 shows the SEM photograph of 12% GSA-Concrete. It showed a reduced quantity of calcium silicate hydrate formed and occurrence of extra-large voids in conjunction with conspicuous spots of anhydrous cement particles compared to normal OPC concrete. This can be attributed to low pozzolanic reaction of GSA with consequent reduced compressive strength of concrete and improvement in the morphology of the concrete when compared to control samples. Figure 9 shows the SEM photograph of BLA concrete which showed reduction in the voids in the concrete due to production of more compact fibrous C-S-H in the well-developed concrete as well as smooth platy crystals of Ca(OH)$_2$ giving a better packed concrete component. Nevertheless, little bright bits of anhydrous cement were still visible. The image however showed better dense microstructure than that of 12% GSA. This improvement can be traced to improved hydration as well as positive effect of the pozzolanic reaction of bamboo leaf ash (BLA). Figures 10 to 12 show the SEM photograph of OPC, BLA and GSA after 28 days in salt respectively. The figures indicate a disruption in the microstructure of the concrete cubes due to absorption of sodium chloride solution.

![Figure 7. SEM Image of OPC after 28 days in water](image1)

![Figure 8. SEM Image of GSA after 28 day in water](image2)

![Figure 9. SEM Image of BLA after 28 days in water](image3)

![Figure 10. SEM Image of OPC after 28 days in NaCl](image4)
3.6 Predictive models for depth of chloride penetration

3.6.1 Chloride ion predictive model for OPC concrete

The data obtained from the various tests carried out on OPC, BLA and GSA were analysed and regression analyses results obtained are shown in Figures 13 to 15. The regression analysis yields coefficient of correlation (R^2 value) of 0.967, 0.939 and 0.949 for OPC, BLA and GSA respectively. This indicates strong relationship between the depth of chloride penetration (d), and other parameters i.e. absorption (i), age (t) and compressive strength (f_{cu}).

The regression models is presented in Equations 9 to 10 for OPC, BLA and GSA respectively

\[
d = 1015t - 0.32f_{cu} + 5.30i - 1.89 \\
d = 0.19t - 1.72f_{cu} - 0.16i + 33.63 \\
d = 1.01i - 0.04t - 5.42f_{cu} + 97.74
\] (9)

(10)

(11)
Figure 13. Depth of chloride penetration against absorption for OPC

Figure 14. Depth of chloride penetration against absorption for BLA
4. Conclusions

From the experimental investigations conducted on OPC (normal), BLA and GSA (pozzolanic) concretes, the following conclusions can be drawn:

i. The absorption of BLA and GSA concretes were found to be less than that of the OPC (control) concrete. Hence, the presence of pozzolans (i.e. 12% BLA and 12% GSA) in the mix improves the durability of concrete by filling the voids in the cement. Pozzolanic concrete compared to control mixture may be acceptable from the durability point of view.

ii. Absorption plays a significant role in the penetration of chloride ions into concrete.

iii. Depth of chloride penetration increases as the exposure time to salt solution increases in all the mixes.

iv. Ingress of Sodium chloride (NaCl) solution causes compressive strength loss of concrete.

v. For a mix design of 1:2:4 used in this study-, the models showing the correlation between depth of chloride penetration and absorption, age of concrete and compressive strength should be adopted.

vi. The measured depths of penetration obtained for OPC and GSA concrete are higher than the nominal depth of concrete cover usually adopted. This suggests that concrete structures exposed to chlorides ion attack are vulnerable to corrosion damage unless special concrete mix like BLA pozzolanic concrete are adopted.

5. Recommendations

Based on the results of the research, the following recommendations are made:

i. The use of Bamboo Leaf Ash and Groundnut Shell Ash should be encouraged for partial replacement of cement in concrete production especially where durability is of utmost importance. This will reduce the cost of concrete production and reduce the permeability of concrete to chloride attack.

ii. The numerical models should be adopted to predict the ingress of chloride into normal concrete and pozzolanic concretes containing 12% Bamboo Leaf Ash and 12% Groundnut Shell Ash.
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