

Chloride Ingress Resistance in Selected Calcined - Clay - Portland Cement Blends

Marangu Joseph Mwiti

Chemistry Department, Meru University of Science and Technology, Meru, Kenya

Abstract: This paper presents the results of an experimental study which was carried out to investigate the chloride ingress resistance of the blend of the calcined clays with Ordinary Portland Cement (OPC), vis a vis commercial OPC and Portland Pozzolana cement (PPC). Clays were sampled from selected regions of Runyenjes – Kenya. The sampled clays were activated at 800 °C for one hour and blended with OPC at different replacement levels. The blends were subjected to accelerated chloride ingress in accordance with ASTM C1202 with some modifications. The objective of the study was to investigate the durability of blended cement in chloride rich media. Knowledge of the chloride ingress resistivity will lead to research on possible ways of improving the test cement. The study was thus engineered at establishing the robustness of the test cement vis a vis the commercial OPC and PPC. The blended cement and commercial PPC exhibited the highest resistance to chloride ingress in terms of the quantity of electric current allowed in compared to neat OPC in a simulated solution of 3.5 percent sodium chloride solution.

Keywords: Blended Cement, Calcined Clays, Pozzolanic Cement, Resistivity, Water / Cement ratio

1. Introduction

The durability of cement concrete is refers to its ability to resist weathering action, chemical attack, or any other process of deterioration (Poon *et al.*, 2006). The use chloride ingress resistance is increasingly being used as parameter of durability of in concrete structures (Park *et al.*, 2006). Many researchers are interested in low cost quality materials for use in housing and general construction. This is because high strength binders such as Portland cement are not a necessity in most housing projects (Muthengia, 2008). The construction materials being sought should meet the basic standards for general construction purposes. Frequently, reinforced mortar/ concrete structures are exposed to salts-laden environments yet they are expected to last with little or no repair or maintenance for long periods of time (often 100 years or more) (Ahmed *et al.*, 1997). One of the major forms of environmental attack in such structures is chloride ingress, which leads to deterioration of the reinforcing steel and a subsequent reduction in the strength, serviceability and aesthetics of the structure (Byfors *et al.*, 1986). This may lead to early repair or premature failure of the structure. This results to high cost implications in terms of the maintenance of these structures in their desired form.

In order to evaluate the performance of mortar/ concrete with respect to durability in aggressive environments, a greater understanding of chloride ingress in particular cement is necessary. The main factor of interest when it comes to such a study is the chloride resistivity of concrete. Estimating the chloride resistivity assists to predict the depth of chloride penetration in concrete upon exposure to chloride rich media. This is important as it forms the basis upon which predicting the service life of cement mortar/ concrete structures can be done.

A major concern and the point of reference in this study revolved around assessing the durability of the PCC in a

simulated salty environment of chlorides. This entailed estimation of the quantity of electricity allowed in mortar verses depth of cover. This was to give an accelerated test for the performance of the cement in the chloride rich environment. Investigation on the durability of the PCC cement is necessary for making decisions on conditions under which the cement is applicable. In assessing its durability vis-a-vis OPC and PPC, the study was aimed at establishing the suitability or otherwise of the test cement with respect to chloride environment.

2. Materials and Methods

a. Preparation of NaOH solution

The requisite amount of dried NaOH at 110 °C was dissolved in water to make a solution of 0.3 M solution.

b. Preparation of 3.5 percent NaCl solution

35 g of analytical grade sodium chloride was dissolved in requisite amount of water to make 3.5 per cent sodium chloride solution in 1-litre volumetric flask. The volume of the resulting solution was made up to the mark.

c. Preparation of Clay-Based Cement Blends

In this study, raw clays were sampled from three different places within Runyenjes region in Kenya. The samples from a given place were mixed mechanically. The sampled clays were dried to a constant weight at 110 °C using an Electrical Muffle furnace model number OSK 9540-MK-SP and allowed to heat for one hour at 800 °C. The resultant clay was cooled, finely ground using laboratory ball mill to 90 µm mesh size. To make PCC (clay-based cement blends), requisite amounts of calcined clay and OPC were mechanically mixed using trowels on a tray to make PCC's at 25, 30, 35, 40, 45 and 50 per cent substitution of OPC. They were labeled PCC25, PCC30, PCC35, PCC40, PCC45 and PCC50 respectively.

d. Casting of Mortar prisms

This was done in accordance with EAS 148-1(2000) specifications with slight modifications. Three mortar

prisms each having dimensions of 160 mm × 40 mm × 40 mm were prepared at a go. This was done by mixing 450 g of OPC and 1350 g of sand with two trowels on a non – porous plate for one minute. Requisite amount of water was added and mixing continued for further four minutes. During the mixing process, care was taken to ensure that none of the materials spilled over.

A thin film of petroleum jelly was applied to the joints of the two halves of cleaned mould, the contact surface of the bottom of the mould and its base plate. The mould was then assembled by screwing. The assembled mould was placed on a vibrating machine and firmly held in position by means of a clamp. The prepared cement mortar was filled in the mould using a hopper. Compaction of the mortar was done by vibration for two minutes at 12000 ± 450 vibrations per minute on the vibrating table. The above procedure was repeated with PPC and the various PCC's. In each cement category, nine prisms were prepared. In each preparation, 180 ml, 247.5 ml and 283.5 ml of water was used. This corresponded to w/b ratio of 0.4 to 0.55 and 0.63 respectively.

After compaction, the mould with mortar was covered with a polythene sheet. They were then placed in a curing room with relative humidity above 90 percent and at a temperature of 27 ± 2 °C for 24 hours ± 30 minutes. After 24 hours ± 30 minutes, they were de – moulded and marked accordingly for identity purpose. The marked prisms were submerged immediately in curing tank maintained at 23 °C for twenty seven days. On the twenty eighth day, three prisms for each cement category were removed from curing tank and allowed to drain for thirty minutes. The prisms were stored in dry plastic bags until they were used in the electrochemical set up cell.

e. Chloride ingress resistance

This was done in accordance with ASTM C –1202 with some modifications. The cement mortar prisms cured at 28 days were reduced to 100 mm length using cutting machine model number MC 100 type EFNOUT KT with a 1.5 mm blade. Cathode compartment of an electrochemical set up was filled with 500 cm³ of the 3.5 percent sodium chloride solution. The anode compartment cell was filled with 500 cm³ of the NaOH solution.

A mortar prism was firmly placed in the assembled test cell using epoxy coating. A stainless steel rod was dipped in each cell such that they were in contact with the periphery faces of the specimen. The rods were fastened with a clamp at both ends to hold the whole assembly together. An external voltage cell was used to apply a voltage difference of 12 ± 0.1V between the electrodes.

After the whole assembly was completed and the solutions in both the anode and cathode compartments were in place, the power was switched on. The electric current in the test cell was monitored by use of a digital ammeter at intervals of thirty minutes for a period of thirty six hours. The chloride

ingress resistance is computed as the total charge allowed in mortar prisms. The formula in equation 2.0 was used

Chloride ingress resistance, Coulombs = (I₀ + I₁ + I₂ + I₃ + I₄ + I₅+..... I₇₂) mA × 0.001× 60 × 602.0

Where

I₀, I₇₂ are the initial and final currents

I₁, I₂, I₃, I₄, I₅,.....I₇₁ are the intermediate currents



Plate 2.1: A Modified Electrochemical Cell for electrical resistivity (courtesy of Mutitu D. Karanja)

3. Results and Discussion

3.1 Cement type verses water/ cement ratio

The figure 3.1 shows the variation of the total quantity of electricity passed with cement categories.

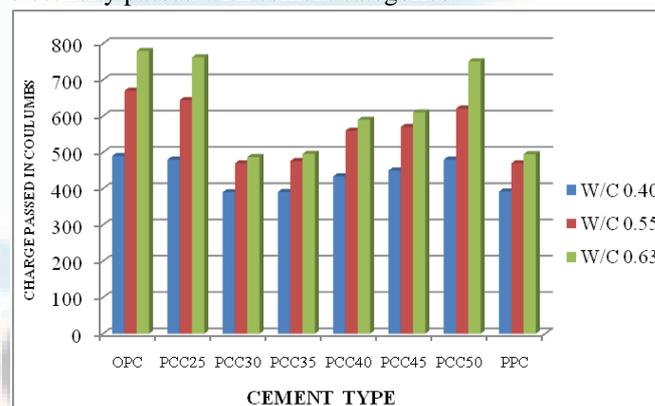


Figure 3.1: Variation of The total Quantity of Electricity Passed with Cement Categories at different w/c

It was observed that OPC showed the least chloride resistivity compare to the blended cements used in this study. The use of pozzolana has been reported to lower the chloride ingress in mortar /concrete (Al-Rawas *et al.*, 2001; Gonçalves, 2009; Muntasser, 2002). The resistance to aggressive attack of PPC's has been attributed to factors such as the pore refinement of its microstructure, the low alkalinity of its pore solution, and the low portlandite, Ca(OH)₂, content. These factors, which arise from the pozzolanic reaction, impede diffusion of aggressive ions such as chlorides (Lorenzo *et al.*, 2002). The presence of pozzolana leads to a greater precipitation of cement gel

products than occurs in OPC, which more effectively block the pores more effectively and therefore helping to reduce permeability (García *et al.*, 2000). The water-soluble calcium hydroxide liberated by hydrating cement may leach out of hardened concrete and leave voids for the ingress of water. In the pozzolanic reaction, by combining with the Calcium hydroxide (CH) directly reduces the amount of CH, which can reduce the leaching of CH (García *et al.*, 2000). The additional products from pozzolanic reaction, C-S-H will close the voids, which result in more dense concrete, consequently increase the chloride ingress resistance to due to pore refining process (Agarwal, 2006; Bai *et al.*, 2003; Elinwa, 2004; Taylor, 1990).

Partial substitution of OPC with calcined clays in the range of 30 percent to 35 percent exhibited the highest resistance to chloride ingress. Beyond 35 percent replacement of OPC, the amount of chloride ingress in cement mortar gradually increased at all w/b. This is perhaps due to the fact that replacement of OPC beyond 35 percent, the added pozzolana only serves as a filler material with low resultant CSH. Similar observations were made by Khater *et al.* (2010) although studying the Influence of metakaolin on resistivity of cement mortar to magnesium chloride solution. The researchers attributed this to low chloride binding with increase in amount of pozzolana added due to formation of highly porous and less dense cement mortar with low amount of CSH. This implies that replacement of OPC with calcined clays beyond 35 percent does not improve the blended cement resistivity to chloride ingress.

As the w/b ratio increased, there was a marked rise in the chloride ingress in all the profile depths of the test cements in the order $0.4 < 0.55 < 0.63$. A high w/c ratio is known to contribute to a higher permeability consequently increasing the ingress of chlorides in cement pastes (Bentz, 2006; Page *et al.*, 1986). When the w/b ratio increases, the porosity of the resultant mortar increases (Wild *et al.*, 1996; Sabir *et al.*, 2001). This results in higher diffusivity of the chloride into the mortar. Vaishali *et al.* (2011) although, studying the chloride ion permeability studies of metakaolin based high performance concrete observed that cement mortar with w/b ratios of 0.3 had a lower chloride content than those cast at a w/b ratio of 0.5. The workers attributed this to the fact that decreased w/b ratio increases the resistance of concrete/mortar to chloride ingress by lowering permeability and porosity.

There was no significance difference in terms of chloride ingress between PCC35 and commercial PPC at w/b = 0.40, w/b = 0.55, w/b = 0.63 as the T- calculated values were 0.1621, 0.5321 and 0.2871 respectively. The T- calculated values were far below the T- critical value of 6.314.

The values in the table 3.1 were used a guide on the assessment of chloride ion permeability.

Table 3.1: shows the Chloride ion penetrability based on charge passed (Whiting, 1981)

Charge passed	Chloride ion penetrability
>4,000	High
2,000–4,000	Moderate
1,000–2,000	Low
100–1,000	Very Low
<100	Negligible

It was observed that chloride resistance for blended cements were in the range 100–1,000 (Columbs) of the quantity of electricity passed. This implies that the chloride resistance of concrete is thus highly dependent on the porosity of concrete in terms of pore size, pore distribution and interconnectivity of the pore system. The porosity of concrete is determined by; the type of cement and other mix constituents, concrete mix proportions, compaction and curing. The type of cement influences both the porosity of the concrete and its reaction with chlorides. The porosity of concrete is highly dependent on the water-cement and aggregate-cement ratios whereas the type and amount of cement affect the pore size distribution and chemical binding capacity of the concrete

PPC generally had higher Al_2O_3 content than OPC. This results from included pozzolana (Otsuki, *et al.*, 2003). Al_2O_3 is known to bind Cl⁻ thus decreasing their ingress. This results in mainly the Friedel's salt (Suryavanshi, *et al.*, 1996). When more chlorides are bound, the ingress of chlorides is lowered (Hossain and Lachemi, 2004; Luo, *et al.*, 2003). The binding effect of the PPCs may not have been sufficiently predominant to override the other beneficial effects of pozzolana in improving the resistance of the blended cements. It would then seem that the reduction in porosity due to additional cementitious material from pozzolanic reaction and the packaging of the pozzolana grain between aggregates and cement grains played a significant role in improving the resistance of the blended cements (Cheng, *et al.*, 2005; Hossain and Lachemi, 2004). Bai *et al.* (2003) observed that due to pozzolanic reactions, the pores become finer and hence the chloride resistivity drops.

3.2 Chloride Ingress Verses Depth of Cover

Figure 3.2 - 3.4 shows the graphs of electric current allowed verses the depths of cover for each cement category at different water to cement ratios.

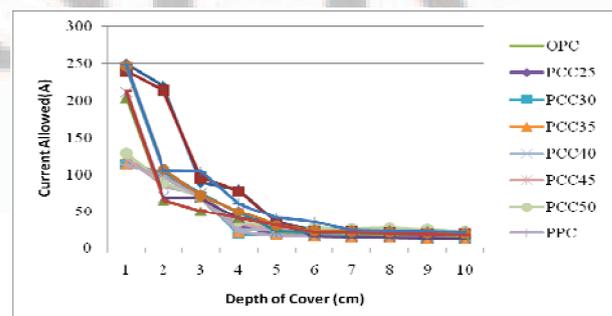


Figure 3.2: Graphs of Electric Current Allowed Verses the Depths of Cover for Each Cement Category at w/c 0.40

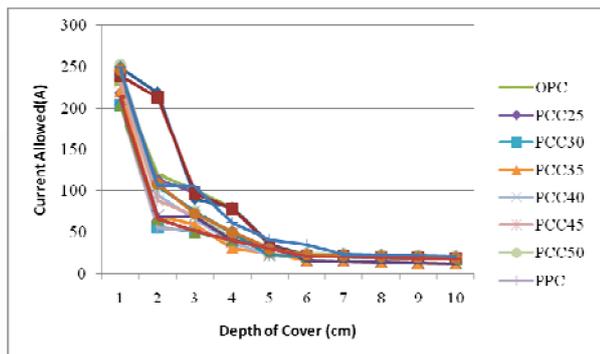


Figure 3.3: Graphs of Electric Current Allowed Verses the Depths of Cover for Each Cement Category at w/c 0.55

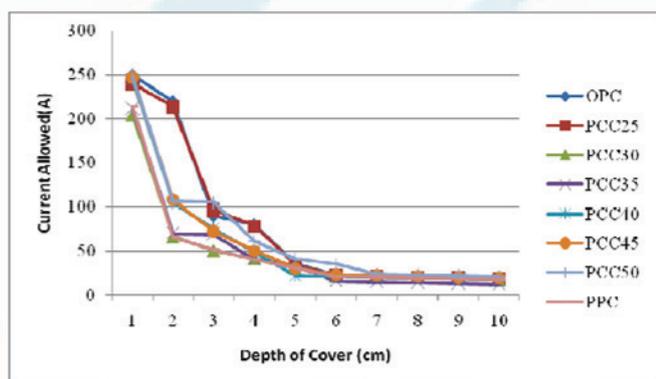


Figure 3.4: Graphs of Electric Current Allowed Verses the Depths of Cover for Each Cement Category at w/c 0.63

It was observed that in all categories of cements, the total current allowed in mortar decreased with increase in depth of cover. This was even more pronounced as the w/c ratio reduced. There is sharp decrease in the amount of chlorides at the shallow depths of cover (<20 mm). This could be attributed to chloride binding capacity and diffusivity of chlorides in the cement mortar (Arya *et al.*, 1990; Qiang, 2009). All cements bind a proportion of the chloride present (Hirao *et al.*, 2005). Chloride binding reduces migration rates of chlorides into the cement bulk of the cement involved (Hirao *et al.*, 2005). Similar observations were made by Gerard and Marchand (2000) although studying the influence of chloride binding on the chloride induced corrosion risk in reinforced concrete. The workers attributed this to the increased chloride binding by the mortar at the surface due to increased CSH content. The increased CSH bind Na^+ in NaCl solution and hence retarded the ingress of the chloride ions.

At greater depths of cover (>20mm), it was observed that the OPC exhibited the least chloride ingress resistivity especially at higher w/c ratios. This can be attributed to the fact that blended cements have lower CH than OPC in their pore solution (Bao-min and Li-Jiu, 2004; Hirao *et al.*, 2005). A low amount of CH in blended cements is as a result of pozzolanic reaction (GjØrv and Vennesland, 1979). CH, acts as the main source of OH^- hence the decreased amount of OH^- leads to a lower exchange capacity between the OH^- and the chlorides in the pore water (GjØrv and Vennesland, 1979). Similar observations were made by Daniel *et al.* (2011) who investigated chloride diffusivity in Red Mud-

Ordinary Portland Cement Concrete (RMOPC). These workers attributed the reduction in ingress in blended cement to lower exchange capacity between the OH^- and the chlorides in the pore water, reduced porosity, denser mortar, and higher binding capacity. These factors clearly explain the high chloride ingress resistivity in blended cements used in this study.

GjØrv and Vennesland (1979) observed that although w/c ratio may affect the ingress of chlorides into concrete, the effect was only significant for a short distance. They observed that, for a longer depth in the bulk of the concrete, the effect was minimal. Similar observations were made by Yiğiter, *et al.* (2007) though he was working with blast furnace slag cements. The workers (Yiğiter, *et al.*, 2007) observed significant effects in OPC where chloride ingress increased with increase in w/c ratio. GjØrv and Vennesland (1979) reported that the ingress had to do with the property of the concrete (GjØrv and Vennesland, 1979; Hossain and Lachemi, 2004; Luo *et al.*, 2003).

As the profile depth increased (>20 mm), it was observed that the OPCs exhibited higher chloride ingress especially at higher w/c ratios than PPCs. OPC has a high $\text{Ca}(\text{OH})_2$ content as well as higher pH in pore water than PPC (Byfors, 1987; Diamond, 1996; Mehta and Monteiro, 2005 and Rasanen and Pentalla, 2004). This offers a higher exchange capacity for Cl^- to OH^- (Rowell, 2010; Neville, 2008; Maria, *et al.*, 2008; Weyers, *et al.*, 1993). This in turn increases the permeability of Cl^- in OPC than PPC (Poon, *et al.*, 2009; Vaishali and Sudarsana, 2011). Rasanen and Pentalla (2004) and Yiğiter, *et al.* (2007) observed similar trends.

4. Conclusion

PPC, PCC30 and PCC35 exhibited similar performance in terms of Cl^- ingress resistivity when exposed in Chloride ion media. The test cements, PCC30 and PCC35 can thus be used in similar chloride environments as commercial PPC.

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Author Profile



Marangu Joseph Mwiti: Received the B.Sc. in Chemistry degree from Jomo Kenyatta University of Agriculture and Technology in 2009 and M.Sc. in Chemistry degree from Kenyatta University in 2013.

He is currently pursuing a Ph.D degree at Kenyatta University in Kenya. He has actively participated in research on cement technology especially on low cost cement. His research interests are mainly on cement chemistry and corrosion science. Furthermore, he is teaching chemistry at Meru University of Science and Technology.

IJSER