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THE EFFECT OF LIMESTONE MINERAL ADDITION AND CEMENT KILN DUST ON THE CHLORIDE INGRESS INTO MORTAR SPECIMENS MADE WITH CEMENT, FLY ASH AND SLAG

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ABSTRACT

Research into the effect of increased levels of limestone mineral addition plus cement kiln dust on the rate of chloride ingress through mortar and concrete was commenced by the lead author in 2010 following changes to the Australian cement standard, AS 3972 Portland and blended cement (1997). The research also incorporates the effect of replacing some of the cement with fly ash and ground granulated blastfurnace slag. This paper concentrates on the chloride ion ingress data that has been produced as part of the research. The mixes are made with cement containing limestone mineral levels of 4%, 10% and 15% with and without 5% cement kiln dust additions and mixes where the cement was replaced with 20% fly ash or 30% slag, again with and without 5% cement kiln dust. The chloride ingress of the mortar has been determined on specimens that have been subjected to standard water curing for up to one year and tested using a method based on Nord Test Build NT Build 492 (1999).

KEYWORDS

Limestone mineral addition, cement kiln dust, fly ash, ground granulated blastfurnace slag, Type GP cement, Chloride ion ingress.

INTRODUCTION

This paper presents chloride ion ingress results of mortar made with cement containing elevated levels of limestone mineral addition when used in combination with cement kiln dust (CKD). The mixes were based on both cement only mixes incorporating LMA and CKD at various percentages and mixes containing supplementary cementitious materials (SCM), where the cement was replaced by either fly ash (FA) or ground granulated blastfurnace slag (GGBS). The test specimens were cut from 100 mm × 200 mm standard cylinders, made and cured as specified by AS 1012.8.1 (2014). The rate of chloride ion ingress was determined using a method based on the NordTest NT BUILD 492 (1999) with the specimens tested at various ages up to a year. This paper reports on only some of the results of the full research program currently being undertaken at the University of South Australia (UniSA), investigating what effect increased levels of LMA, when used in conjunction with CKD, will have on the chloride ion ingress of mortar/concrete. The other aspects investigated include compressive strength and chloride diffusion, using the NT Build 443 (NordTest 1995), on both mortar and concrete specimens.

The research was prompted by the 2010 revision of the Australian cement standard AS 3972 Portland and blended cement (Standards Australia 1997). An extensive industry testing program, based around limestone as a mineral addition, commenced in 2008 and was designed to demonstrate that an increase the levels of mineral additions was feasible without negatively affecting the performance based characteristics of the 1997 version of the cement standard AS 3972 (Standards Australia 1997). These changes reduce the "carbon footprint" of cement manufacture and contribute to the government program of reducing greenhouse gas emissions as stated in the preface of the 2010 revision of the standard (AS 3972, 2010). The revision of the standard resulted in the following changes:

- The name of the standard was changed to AS 3972 – General purpose and blended cement (2010).
- The maximum mineral addition level was increased from 5% to 7.5%.
- Up to a maximum of 5% of the 7.5% mineral addition could be minor additional constituents such as cement kiln dust.
- A maximum chloride level of 0.10% in all cement was introduced.
- A new cement type; "General Limestone Cement", Type GL, with a limestone content greater than 7.5% and not greater than 20% was incorporated.

BACKGROUND

The cement standard AS 3972 (2010) was changed in 2010 and in various parts of Australia the Type GP cement containing LMA up to 7.5% has now been used since 2011 without any reported issues. The Australian Type GP cement is equivalent to the European CEM I (EN 197-1, 2000), the Canadian Type GU (CAN/CSA A3001, 2008) and the American Type 1 as specified in ASTM C150 (2005) and ASTM CC595/C595M (2012). The Type GL cement, containing LMA greater than 7.5% is not yet commercially available, due to manufacturing and storage constraints.

The research program is primarily aimed at determining the influence of cements containing different levels of LMA and CKD on chloride ingress. In addition to different levels of LMA and CKD, both fly ash and GGBS were used as cement replacements. The cementitious material, referred to as binder, was made up of either:

- Cement plus mineral addition (i.e. LMA and/or CKD) or
- Cement, mineral addition and SCM (i.e. fly ash or slag).

In the research the control mortar mix was made with cement only containing nominally 4% limestone mineral addition and compared to:

- Mortar mixes made with cement containing nominally 10% and 15% LMA.
- Mortar mixes containing LMA and either 2% or 5% CKD and
- Mortar mixes in which the cement was replaced by either 20% or 40% fine fly ash or by 30% or 60% blastfurnace slag.

This paper will consider the chloride ingress into mortar mixes made with cement only, see Table 1, containing nominally 4%, 10% and 15% LMA and compare these to:

- Cement only mixes containing the addition of 5% CKD by mass of the LMA and
- Mixes where the cement was replaced by either 20% fly ash, Table 2, or 30% slag, Table 3, as these SCM levels are typical of local commercial mixes.

MATERIALS AND LABORATORY METHODS

Mix Details

The mix proportions of the mixes discussed in this paper are detailed in Table 1, Table 2 and Table 3. The high binder content was dictated by the water demand of the sand required to produce a slump of around 90 mm in both concrete and mortar with a water/binder ratio of 0.45 and 0.40 respectively. The mix code used, for example M 04.0.00, describes the mix as follows:

- M indicates cement only mortar, MF indicates mortar with fly ash and MS indicates mortar with slag.
- The first two digits 04, 10 or 15 indicate the limestone level as percentage of cement,
- The third digit zero, 2 or 5 indicates the CKD level as a percentage of the LMA and
- The last two digits 00, 20, 40, 30 or 60 indicate the FA or slag level as a percentage of the total binder.

Table 1. Mix proportions cement only mortar mixes

Mix Code	**M 04.0.00**	**M 10.0.00**	**M 15.0.00**	**M 04.5.00**	**M 10.5.00**	**M 15.5.00**
	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3
Cement binder	670.0	670.0	630.6	668.7	666.7	625.5
Limestone added	-	-	39.4	-	-	39.4
CKD added	-	-	-	1.3	3.3	5.1
Fly ash	-	-	-	-	-	-
Slag	-	-	-	-	-	-
Sand	1340	1340	1333	1339	1339	1332
Water	270	270	270	270	270	270
water/binder ratio	0.40	0.40	0.40	0.40	0.40	0.40
sand/binder ratio	2.00	2.00	1.99	2.00	2.00	1.99

Table 2. Mix proportions cement/fly ash mortar mixes

Mix Code	**MF 04.0.20**	**M 10.0.20**	**MF 15.0.20**	**MF 04.5.20**	**MF 10.5.20**	**MF 15.5.20**
	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3
Cement binder	536.0	536.0	504.5	534.9	533.3	500.4
Limestone added	-	-	31.5	-	-	31.5
CKD added	-	-	-	1.1	2.7	4.1
Fly ash	134.0	134.0	134.0	134.0	134.0	134.0
Slag	-	-	-	-	-	-
Sand	1295	1295	1290	1295	1295	1289
Water	270	270	270	270	270	270
water/binder ratio	0.40	0.40	0.40	0.40	0.40	0.40
sand/binder ratio	1.93	1.93	1.93	1.93	1.93	1.92

Table 3. Mix proportions cement/slag mortar mixes

Mix Code	**MS 04.0.30**	**MS 10.0.30**	**MS 15.0.30**	**MS 04.5.30**	**MS 10.5.30**	**MS 15.5.30**
	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3
Cement binder	469.0	469.0	441.4	468.1	466.7	437.8
Limestone added	-	-	27.6	-	-	27.6
CKD added	-	-	-	0.9	2.3	3.6
Fly ash	-	-	-	-	-	-
Slag	201.0	201.0	201.0	201.0	201.0	201.0
Sand	1328	1328	1324	1328	1328	1323
Water	270	270	270	270	270	270
water/binder ratio	0.40	0.40	0.40	0.40	0.40	0.40
sand/binder ratio	1.98	1.98	1.98	1.98	1.98	1.97

Materials

A summary of some of the properties of the binder materials is provided in Table 4. Two cements, containing limestone nominally at 4% and 10% respectively, were produced by Adelaide Brighton Cement by intergrinding the limestone with the clinker and gypsum. The laboratory mixes made with cement containing 15% LMA were a blend of cement with 10% interground limestone plus an additional 5% finely ground high grade limestone that had calcium carbonate content >80% as required by AS 3972 (2010).

Table 4. Chemical and physical properties of binder materials

Sample	Units	Type GP (4% LMA)	Type GP (10% LMA)	Fine Fly Ash	Slag	CKD	High Grade L/S
Date		Jan-2014	Jan-2012	Apr-2014	Apr-2014	Sep-2012	Jun-2015
SiO_2	%	20.15	19.74	56.46	32.97	13.42	4.44
Al_2O_3	%	4.69	4.37	28.82	13.58	3.88	0.75
Fe_2O_3	%	2.94	3.04	2.52	0.40	2.31	0.80
CaO	%	63.88	62.76	3.42	42.21	59.05	51.85
MgO	%	1.90	1.21	1.86	5.41	1.31	0.66
Na_2O	%	0.26	0.15	1.93	0.22	0.51	0.05
K_2O	%	0.39	0.38	1.23	0.33	4.07	0.16
SO_3	%	2.77	2.57	0.20	1.85	2.48	0.18
TiO_2	%	0.26	0.28	2.08	0.52	0.25	0.06
Chloride	%	0.013	0.008	*	*	2.060	*
Specific Gravity		3.11	3.06	2.22	2.85	*	*
Surface Area	m^2/kg	405	420	*	*	*	*
45µm	%	>96	>97	82.9	>93	*	*

The CKD, typical of material extracted from the bypass of the kiln at the Adelaide Brighton Cement works, was used at additions of 2% or 5% as a percentage of LMA (i.e. CKD is 0.05x(0.04x401.2)= 0.8 kg in MF 04.05.40). The fine fly ash was sourced from the Port Augusta power station in South Australia and the GGBS, obtained from Independent Cement and Lime Pty Ltd of Melbourne, is milled from granulated slag imported from the Nippon Steel & Sumitomo Metal Corporation of Japan. The SCM is given as a percentage of total cementitious binder (i.e. 20% of 670 kg). The sand was a double washed natural concrete sand that is used by many of the pre-mix (ready-mix) concrete suppliers in South Australia. The mass of sand for both the mortar and concrete mixes containing FA and GGBS was adjusted to compensate for the change in relative density of these materials to ensure that the mixes yielded one cubic metre.

Mixing Procedure

The mixing was carried out in either a 70 litre forced pan mixer or a 10 litre Hobart mixer due to the different size mixes used in the different phases.

Cylinder Compaction and Curing

The cylinders were compacted on a vibrating table for 2½ minutes. They were demoulded at between 24 and 36 hours, then placed in a curing tank, with water at 23ºC ± 2ºC, and with pH approximately 9.5, as required by the Australian Standard (AS 1012.8.1, 2014). The specimens were taken from the curing bath as and when required for compression testing or chloride ingress testing.

Workability

The workability/consistency, defined as the ease with which the mortar/concrete can be placed, compacted and finished, was not measured for the mortar mixes but was visually assessed and the observations are detailed as part of the discussions.

RESULTS AND DISCUSSIONS

The results of each of the parameters that may have a major impact on the chloride ion migration are detailed and discussed in this section followed by the conclusions.

Workability

The consistency of the mortar was visually assessed, based on the lead author’s experience, and is summarised below:

- Cement only mixes – suitable for use as a brick bedding mortar.
- Mixes containing fly ash – slightly too soft for bedding bricks.
- Mixes containing GGBS – too stiff for use as a brick bedding mortar.

The improvement in workability when FA was used as a cement replacement in the binder was as expected for a constant w/b ratio (Addis 1986, p.66) because of the rounded particle shape (Day 1999, p.241). The lower workability observed when using GGBS was due to the high percentage replacement at 60% and because the slag was finer than the cement.

Density

The density of each cylinder was determined and this density was compared to the average density to assess both the consistency of weighing and compaction. Both the average densities and the coefficient of variation (CoV) were considered in the assessment and the analysis indicated that the mortar mixes were consistently weighed before mixing and were consistently and well compacted. This implies that any differences recorded in either the depth of chloride ion ingress or the non-steady state migration coefficient can be attributed to the binder, not the quality of the specimen.

This conclusion is based on:

- The requirement in AS 1012.12.1, (1998, p.3) that the density be rounded to 20 kg/m^3 and because the range in the density across the binder type was less than this amount.
- The recommend values for CoV from Table 3.5 in the “Recommended Practice for Evaluation of Strength Test Results of Concrete” (ACI 1965, p.271) that considers a CoV of less than 3.0 to be very good control, and the same values have been applied to the density of the specimens.

The differences in density of the cement only mixes compared to the FA and GGBS mixes were in line with the lower relative densities of the slag (2.92) and fly ash (2.23) versus the cement at 3.12.

Chloride ion ingress results

The data obtained for the depth of penetration of the chloride ions and the non-steady-state migration coefficient tests are set out in Table 5. Following curing under water, specimens of 45-50 mm were cut from the centre of the cylinders, placed back in the curing tank (pH ≈ 9.5) for at least 24 hours before being tested at the age indicted in the Table. The test duration was 23 hours ±15 minutes followed by splitting the cylinders, coating the surface with silver nitrite and measuring the depth of chloride penetration.

Table 5: Chloride ion penetration and migration coefficient data

	Depth of penetration [mm]			**Chloride ion migration coefficient [$\times 10^{-12}$ m^2/s]**		
Mix Code	**35 days**	**100 days**	**364 days**	**35 days**	**100 days**	**364 days**
M 04.0.00	14.8	14.4	8.7	11.2	13.9	4.3
M 10.0.00	14.2	18.6	9.8	15.8	20.9	4.6
M 15.0.00	15.9	18.3	13.4	17.8	22.9	7.1
M 04.5.00	11.1	14.8	7.5	10.7	14.2	3.3
M 10.5.00	14.7	18.6	9.8	16.4	23.2	4.7
M 15.5.00	16.5	21.1	10.1	18.8	26.5	6.7
MS 04.0.30	6.5	7.2	6.0	4.5	5.5	2.9
MS 10.0.30	6.4	7.4	5.1	4.0	5.0	3.1
MS 15.0.30	6.9	7.1	5.4	4.4	4.7	3.1
MS 04.5.30	6.8	7.3	5.0	4.3	4.9	2.8
MS 10.5.30	7.6	7.9	5.5	5.5	5.4	3.2
MS 15.5.30	9.4	9.5	6.3	4.9	5.4	3.4
MF 04.0.20	6.6	4.6	2.8	4.6	2.2	1.0
MF 10.0.20	7.9	5.0	3.6	4.9	2.4	1.4
MF 15.0.20	8.4	3.9	3.9	4.8	1.7	1.5
MF 04.5.20	7.8	4.2	3.2	4.9	2.0	1.2
MF 10.5.20	6.6	4.7	4.1	4.3	2.2	1.6
MF 15.5.20	8.1	5.3	4.0	4.7	2.2	1.5

Cement only mixes

Considering the cement only mixes it is evident from Table 5, Figure 1 and Figure 2 that with increased LMA levels (10% and 15%) as well as with the combination of LMA and 5% CKD the depth of chloride ion penetration and the chloride ion migration coefficient was greater than for the control mix with 4% LMA.

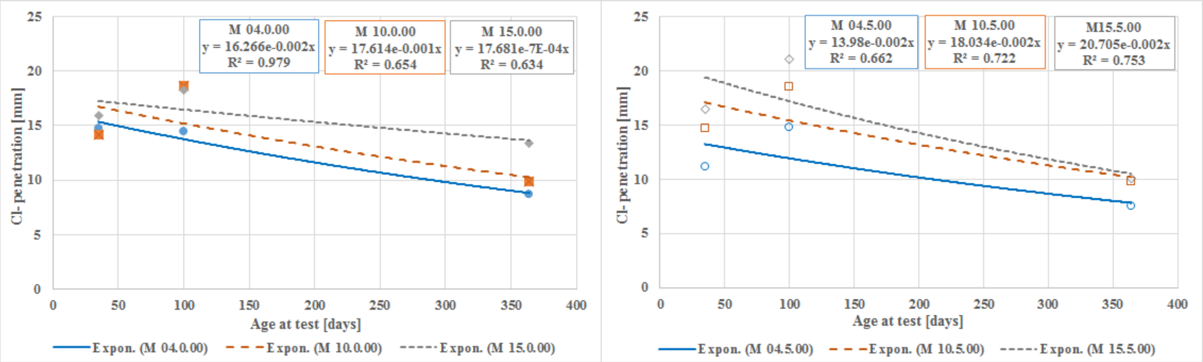

Figure 1a: Increasing LMA-cement only   Figure 1b: Increasing LMA plus 5% CKD-cement only

Figure 1: Trendlines (exponential) of chloride penetration with LMA and LMA plus CKD over 1-year

The trendlines shown in both Figure 1 and Figure 2, are mathematical exponential trendlines that show general trends with age of curing. It is also evident from the Figures that for ages up to 100 days the depth of chloride penetration and chloride ion migration coefficient increases in mixes with levels of LMA of 10% and 15% and LMA plus 5% CKD with levels greater than 4%. This increase could be explained by the findings of Waldemar and Lawrence (1988, p.66) that at up to 129 days more than 80% of the limestone had not reacted, probably indicating a more porous matrix. The significant reduction in both depth of penetration and in the migration coefficient from 100 days to 1-year may be due to a reduction in the pore size due to the formation of tetracalcium moncarboaluminate hydrate compound ($3CaO.Al_2O_3.CaCO_3.11H_2O$), as the LMA (calcium carbonate) and CKD (predominantly CaO) reacts with the C_3A and C_4AF in the cement as reported by Zielinska in 1972 (cited in Neville 2011, p.88).

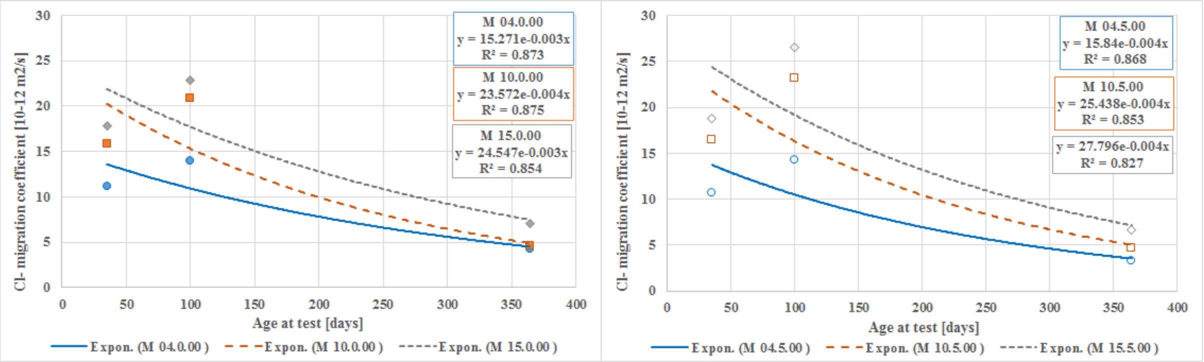

Figure 2a: Increasing LMA-cement only

Figure 2b: Increasing LMA plus 5% CKD-cement only

Figure 2: Trendlines (exponential) of chloride migration coefficient with LMA and LMA plus CKD after 1-year

It could also be argued that the increase in depth of penetration (<5 mm) and the increase in the chloride ion migration coefficient ($<7\times10^{-12}$ m^2/s) is not significant due to the accuracy of the testing because, as stated by Neville (2011, p.495), ”the scatter of permeability test results made on similar concrete at the same age, and using the same equipment is large.”

Cement/slag mixes

The GGBS data in Table 5 is shown in Figure 3 and Figure 4 with general trends indicated by the exponential trendlines. When 30% GGBS is used as a replacement for the cement there is a significant change in the behaviour with regard to the depth of chloride penetration as well as the rate of chloride ingress when compared to cement only mixes. The depth of penetration in specimens made with 30% GGBS was reduced by between 44 to 56 percentage points compared to cement only mixes. With cement/slag mixes the level of LMA did not make a significant difference to the depth of penetration, as depicted in Figure 3a. However, when 5% CKD was added the depth did increase as the LMA increased as shown in Figure 3b. This was particularly evident at ages up to 100 days, for the 10% and 15% LMA levels, and may well be related to the addition of CKD. The CKD, which is similar in composition to the “raw meal” fed to the kiln but which, because it has not been burnt, does not contain any of the cement compounds, and thus does not contribute to the production of hydroxyl ions. It is these hydroxyl ions which when reacting with the amorphous slag will govern the rate of hydration of the slag, which as stated by Neville is slow (2011, p.664).

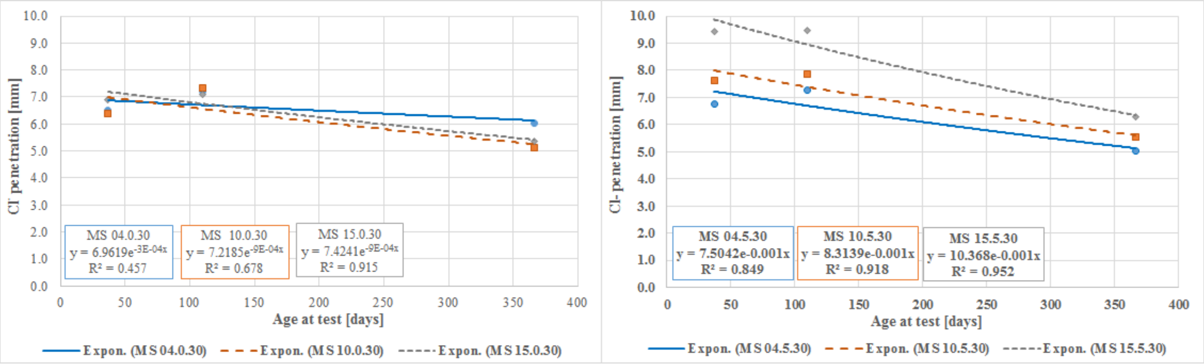

Figure 3a: Increasing LMA-30% GGBS

Figure 3b: Increasing LMA plus 5% CKD-30% GGBS

Figure 3: Trendlines (exponential) of chloride penetration with LMA and LMA plus CKD over 1-year

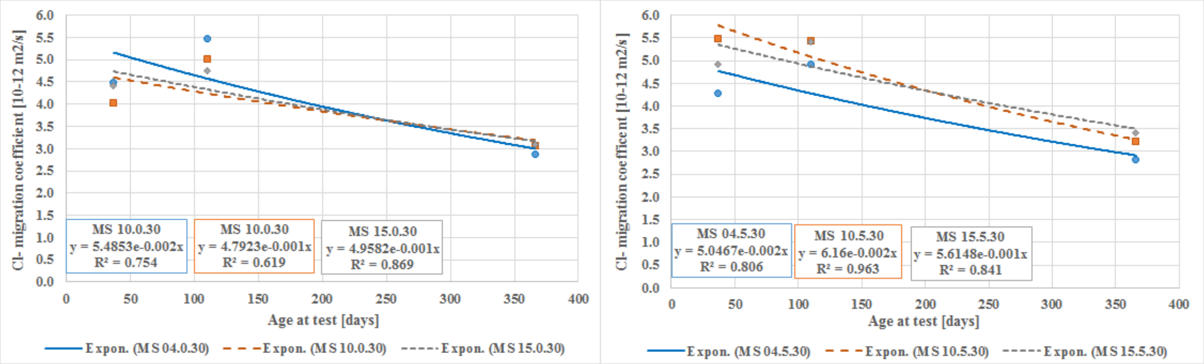

Figure 4a: Increasing LMA-30% GGBS

Figure 4b: Increasing LMA plus 5% CKD-30% GGBS

Figure 4: Trendlines (exponential) of chloride migration coefficient with LMA and LMA plus CKD over 1-year

A similar trend to the depth of penetration is evident when comparing the chloride ion migration coefficient as can be seen in Figure 4. The mixes that did not contain CKD did not show any change in the migration rate as the LMA increased, as shown in Figure 4a but with the 5% CKD addition the rate of chloride ion migration increases when the LMA increased to 10% but did not increase any further when the LMA was increased to 15% as shown in Figure 4b. The increase at 35-days was less than 1.5×10^{-12} m^2/s and was even smaller at 1-year at around 0.6×10^{-12} m^2/s. Again, as with the cement only mixes it could be argued that this change is not significant due to the large scatter of the permeability results as indicated by Neville (2011, p.495).

Although the additional limestone and CKD may provide nucleation points, depending on the fineness, the slow chemical reaction at early ages is likely to result in a more open matrix in the paste, which will become denser as the rate of hydration accelerates at later ages. In addition the relatively high alkali content, particularly the sodium and potassium, see Table 4, in the CKD could influence the activation of the slag hydration at later ages in conjunction with the cement, as stated by Neville (2011, p.665) ‘an important factor is the concentration of the alkalis in the total cementitious material.’ Another factor that would produce a denser microstructure in the mortar as the hydration of the cement and slag combination continues at later ages is the production of more calcium-silica-hydrate (C-S-H) and less lime (CaO) than in a cement only mix (Neville 2011, p.664 & 667) .

Cement/fly mixes

For the specimens made with a 20% replacement of the cement binder by fly ash the trend analysis indicated that a power curve was the best representation of both the depth and rate of chloride ion ingress, as opposed to the exponential relationship evident with cement only and cement/slag binders. Figure 5 indicates that as the LMA increases the depth of penetration generally tends to increase up to 10% LMA, although the actual differences in depth are less than 1.8 mm at 35 days and 1.3 mm at 1-year.

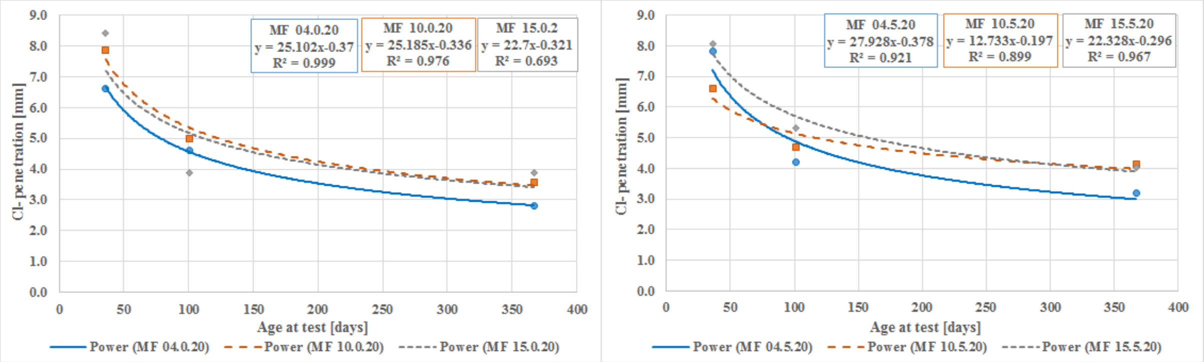

Figure 5a: Increasing LMA-20% FA

Figure 5b: Increasing LMA plus 5% CKD-20% FA

Figure 5: Trend (power curve) of chloride penetration with LMA and LMA plus CKD over 1-year

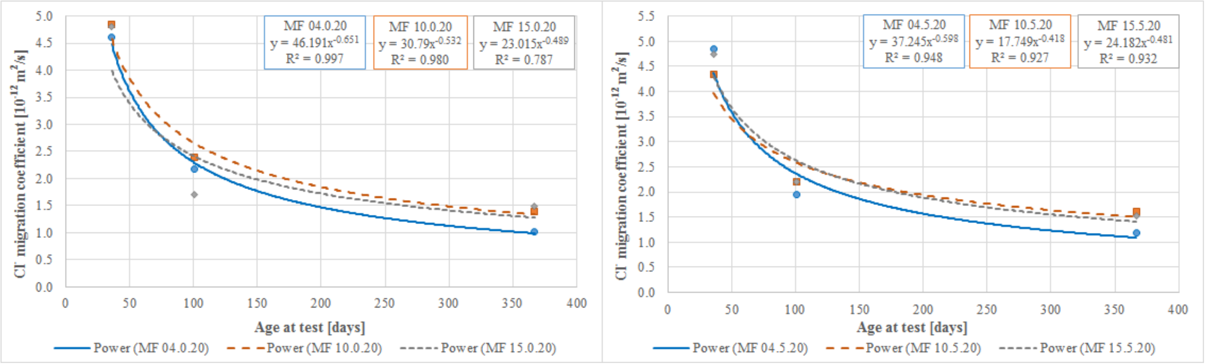

Figure 6a: Increasing LMA-20% FA

Figure 6b: Increasing LMA plus 5% CKD-20% FA

Figure 6: Trend (power curve) of chloride migration coefficient with LMA and LMA plus CKD over 1-year

The rate of chloride ion migration, as indicated by the migration coefficient, showed even smaller differences as the LMA was increased, as shown in Figure 6a. Figure 6b shows that the addition of 5% CKD did not change the rate of migration over the period of 1-year. When comparing the specimens made with cement/fly ash and the cement only, the cement/fly ash blend produced results of greater than 65% lower than the cement only.

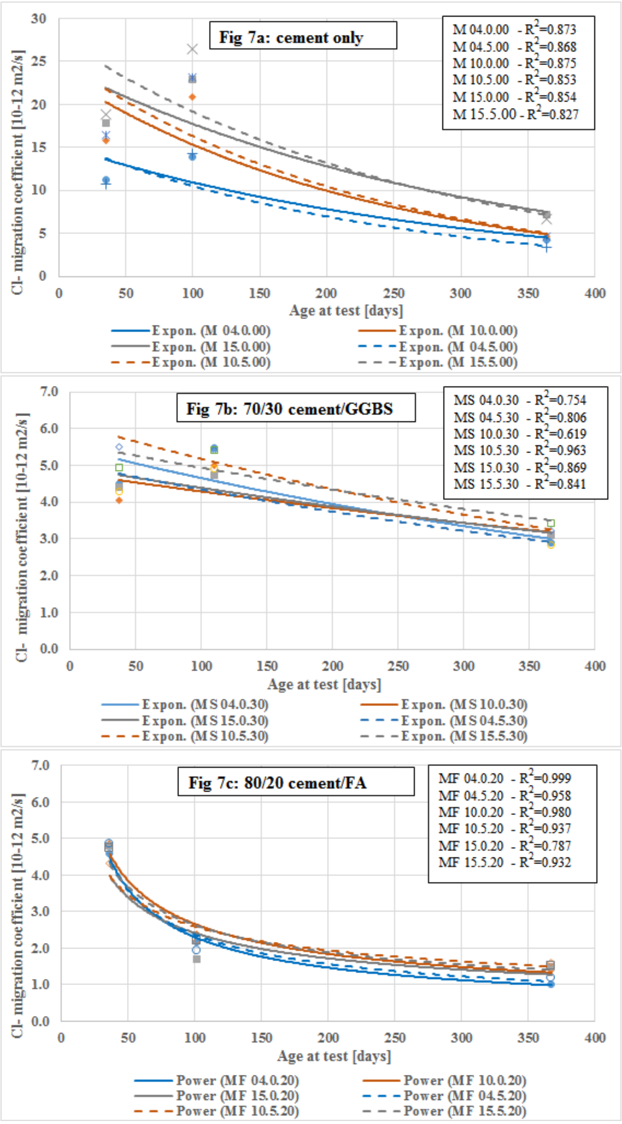

Figure 7: Comparison of chloride ion migration coefficient for different binders

An important factor that will influence the durability of concrete is the rate at which the chloride ions will penetrate the concrete matrix to the depth of the reinforcing steel and then potentially initiate corrosion. The chloride ion migration coefficient, which indicates the rate of penetration, for different 1) binder types; 2) amounts of LMA and 3) CKD is shown in Figure 7. The results discussed in this paper have been based on laboratory work and it should be remembered that the chloride ion penetration rate will also be influenced by:

- Wetting and drying cycles.
- Source of the chlorides (air-borne or groundwater).
- Initial chloride content of the concrete materials.
- Binding capacity of the binders.
- Depth of cover to reinforcement.

CONCLUSIONS

The results of the non-steady-state migration testing can be considered from the aspect of depth of penetration as well as the migration rate of the chloride ion. With respect to depth of penetration the conclusions are:

- The overall depth deceases as cement is replaced by either slag or fly ash
- The depth, at a given age, increases as the level of LMA increases up to 15% in cement only mixes.
- The addition of 5% CKD does not appear to influence the depth as LMA increases with cement only.
- There is no change in the depth as the LMA increases if 30% slag is used as a cement replacement.
- With the addition of 5% CKD the depth increases as the LMA addition increases even with the use of 30% slag.
- When 20% fly ash is used the depth increases with 10% LMA but there is no further increase in depth when the LMA is increased to 15%.
- The addition of CKD does not appear to influence the depth of penetration with a given LMA level.

The conclusions based on the rate of chloride ion ingress can be seen in Figure 7 and are:

- The rate of chloride ion migration deceases considerably as the cement is replaced by either slag or fly ash.
- Replacing cement with 20% fly ash deceases the chloride ion ingress even more than 30% slag replacement.
- In cement only mixes the rate of chloride ion migration increases as the level of LMA increases.
- Increasing the LMA to 15% in mixes containing 30% GGBS does increase the rate of chloride migration, but by less than 1.5×10-12 m2/s at 35 days and 0.6×10-12 m2/s at 1-year, which could be considered insignificant.
- Increasing the LMA to 15% with a 20% FA replacement of the cement does not increase the rate of chloride ion ingress.
- The inclusion of 5% CKD does not significantly alter the rate of chloride ion migration for a given level of LMA, irrespective of the type of binder.

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