

Physical and Chemical Characteristics Affecting the Durability of Condensed Silica Fume (CSF) Blended Cement Concrete

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Abstract: The durability performance of reinforced concrete is influenced by those physical characteristics of concrete that controls the diffusion of gases and or liquid through its pores. These physical characteristics depend on the chemical composition and the microclimatic condition of the environment. In the present paper, properties of concrete made with Condensed Silica Fume (CSF) blended cement were characterized in terms of physical and chemical composition at early-age. In addition, the effects of inland exposure condition on the durability performance of the concrete were also investigated. Concrete cubes were made using various concrete mixtures of water-binder ratios (w/b) = 0.40, 0.50, 0.60, 0.75 and binder contents = 300, 350, 400, 450 kg/m³. Concrete cube of 100 mm size were cast and cure d in water for 3, 7, or 28 days, then characterized at early-ages in terms of its physical and chemical properties. Companion concrete samples were exposed indoors or outdoors to undergo carbonation under natural environment. At the end of the varying exposure period, the concrete cube samples were characterized in terms of carbonation depths. The results of the concrete early-age properties and medium-term durability characterisation were analyzed. The results show that, increased knowledge of concrete materials and concrete early-age properties is vital in durability considerations for reinforced concrete structures.

Keywords: Reinforced concrete; Concrete durability; Pore structure; Pozzolans; carbonation; condensed silica fume; inland environment.

Date of Submission: 21 -06-2017

Date of acceptance: 20-07-2017

I. Introduction

It has been established that the perviousness of concrete as well as its chemical composition plays an important role in the control of concrete durability performance [1]. Accordingly, testing of transport parameters for concrete, such as permeability, absorption or diffusivity behaviour in most cases has been done on the basis of durability consideration. Similarly, the control of concrete durability especially chloride diffusion and carbonation is also affected by binder chemistry [2, 3]. The rate of carbonation in concrete and thus its durability performance is controlled by the proportion of the binder phase (hardened cement paste (hcp) and Interfacial Transition Zone (ITZ)), as well as its properties. The properties of the binder phase in concrete depend on its microstructure that is the type, amount and distribution of solids and voids in the hcp and ITZ . The size and continuity of the pores in the hcp and ITZ in concrete would determine its fluid transport properties and this is closely related to the capillary porosity and the solid-space ratio [4]. The capillary pores and its degree of interconnectivity as well as the entrapped air influences the pore structure of the concrete. While the composition of the solid phase in the hcp and ITZ determine the concrete chemical interaction [4]. In determining the resistance to degradation of concrete, not only should the total capillary porosity, size and interconnectivity of the capillary pores be considered, but also the composition of the calcium silicate hydrates - CSH , and calcium hydroxide - CH that exist in the hcp and ITZ .

The most common cause of deterioration in reinforced concrete is the corrosion of the steel reinforcement. The steel is susceptible to corrosion in the presence of chloride ions or if it becomes depassivated when the alkalinity of the concrete at the location of steel is reduced by carbonation. The transport of aggressive gases and/or liquids into concrete depends on its permeation characteristics. As the permeation of concrete decreases, its durability performance, in terms of physicochemical degradation, increases. Permeation controls the ingress of moisture, ionic and gaseous species into concrete. The use of blended cements or supplementary cementing materials increases the permeability of concrete, thereby decreasing the resistance of concrete to deterioration by aggressive chemicals [5, 6, 7, 8]. Thus, the incorporation of pozzolanic materials such as pulverised fuel ash (PFA), GGBS and silica fume (SF) has become an increasingly accepted practice in concrete structures exposed to harsh environments. Carbonation rate in concrete apart from being influenced by the concrete materials, and its processing, the exposure condition of the concrete in terms of its micro climate also

influences the rate of carbonation in RC. Thus, it is important to quantify the effects of this blended cement concrete in the natural environment.

In this study, condensed silica fume blended cement concrete were characterized at early-age in terms of its pore structure and chemical composition via the durability index tests and the thermogravimetric analysis test respectively. Additionally, the influence of exposure conditions micro climate on the rate of carbonation of these blended cement concretes were also investigated.

1.1 Research significance

The partial replacement of plain cement (PC) with CSF result in significant benefits in terms of dense pore structure for the concrete, while the reduction in calcium hydroxide contents due to pozzolanic reaction affect the ingress of CO₂ into such blended cement concrete. Exposure condition of the concrete influence the efficacy of such blended cement concrete in terms of its durability performance thus an understanding of the effects of CSF blended cement on the physical characteristics and chemical composition of the concrete thus, the durability performance is important, and can help in the development of more durable concretes. Additionally, since micro-climatic condition of the exposure plays a major role in the durability performance of concrete, it is important that concrete durability performance is investigated for the different exposure sites so that the extent of the carbonation is known for CSF blended cement concretes. This is particularly important for mix proportioning and cover depth determination.

II. Experimental Procedure

2.1 Materials

A single source of CEM I complying with SANS was used throughout this study and its chemical and Bogue compositions are given in Table 1. The physical and chemical properties of the CSF can also be found in Table 1. Granite crusher sand and 19 mm granite coarse aggregate were the fine and coarse aggregate used in the concrete [9, 10]. A commercially available plasticizer, was used in some of the mixtures.

Table 1 Oxide analysis of binders (%) used

Oxides	LOI	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	TiO ₂	Mn ₂ O ₃	Na ₂ O	K ₂ O	P ₂ O
CEM I	3.04	21.85	5.10	62.84	3.41	2.28	0.43	0.45	0.07	0.32	0.09
CSF	4.00	88.73	0.60	1.35	2.61	0.86	0.01	0.09	0.20	2.25	0.14

2.2 Concrete mixes

The concrete were made from two binders, 100% CEM I and 90/10 CEM I/FA blend, reflecting the standard blend ratio in South Africa. The concrete mixtures consisted of w/b = 0.40, 0.50, 0.60, 0.75 and binder contents = 300, 350, 400, 450 kg/m³. Slumps in the range of 25 to 140 mm were achieved. Table 2 gives the concrete mixtures proportion and their respective slumps.

Table 2 Concrete mixtures used in the investigation

Binder combination	Mixture ID	CEM I (kg/m ³)	CSF (kg/m ³)	Coarse aggregate (kg/m ³)	Fine aggregate (kg/m ³)	Water content (kg/m ³)	Water/binder ratio	Admixture (l)	Slump (mm)
100% CEM I	PC-40	450	-	990	800	180	0.4	4.00	30
	PC-50	400	-	990	877	200	0.5	3.25	60
	PC-60	350	-	1015	869	210	0.6	-	100
	PC-75	300	-	1050	810	225	0.75	-	140
90/10 CEM I-CSF	SF-40	405	45	990	800	180	0.4	4.25	25
	SF-50	360	40	990	877	200	0.5	3.50	45
	SF-60	315	35	1015	869	210	0.6	-	85
	SF-75	270	30	1050	950	225	0.75	-	115

2.3 Mixing and casting

Concrete was produced in 50 litres batch mixes using a horizontal forced action pan mixer of 0.05 m³ capacity. The mixer was charged with coarse aggregate, fine aggregate and binder respectively and the constituents dry mixed for approximately 30s. Water mixed with plasticiser was slowly added for some mix until the mixture was visually consistent. Plasticiser was added in concrete mixes of 0.40 and 0.50 w/b, achieving slumps of 30 and 60 mm for CEM I and 25 and 45 mm for CEM I/CSF respectively. The dosage of the admixture as shown in Table 2 is given as a percentage of total binder content by mass. The total mixing time was about 3 min. After testing for slump, 100 mm cube moulds were filled, compacted on a vibrating table, and covered with polythene sheets for 24 hours at room temperature before demoulding.

2.4 Curing and subsequent storage

The concrete cubes were thereafter placed in a water bath maintained at 23 ± 2 °C for continuous water curing until their respective test ages of 3, 7 or 28 days. For each mixture, 54 concrete cubes were cast, with a set of three cubes, each set having been cured for 3, 7 or 28 days were tested for compressive strength, two concrete cubes, each set having been cured for 3, 7 or 28 days were tested between 28 and 32 days after casting for permeability via the oxygen permeability and water sorptivity tests. One cube, each having been cured for 3, 7 or 28 days were tested for chemical composition of the concrete mixtures in terms of $\text{Ca}(\text{OH})_2$ content. Additionally, for durability characterisation of the concrete mixture in terms of carbonation, a set of four concrete cubes, each set having been cured for 3, 7, or 28 days and exposed to the natural environment. Prior to exposure of samples to designated sites, all four sides running from the finishing surface through to its opposite end surface, were coated with two layers of epoxy coating, leaving the two adjacent opposite sides uncoated. After sufficient drying of surface coatings over a 24 hour period, samples were taken to their respective exposure sites and placed at a spacing of at least 50 mm from each other such that the two non-coated surfaces were exposed to air. The natural exposure conditions were chosen based on the EN Classification *XC3* and *XC4* [11] as indoor, outdoor sheltered, outdoor exposed. These exposure conditions are the higher aggressiveness classification for carbonation-induced corrosion of reinforced concrete in the inland environment. In order to replicate these conditions, concrete cube samples were stored in a basement parking garage (indoor exposure), under a bridge alongside a busy highway (outdoor sheltered) and on the roof top of a building in an urban setting (outdoor exposed).

2.5 Testing concrete

At the end of the various curing periods and air storage, the concrete cubes were characterized as follows:

2.5.1 Strength:

To characterise the concrete mixtures in compressive strength terms, 100 mm concrete cube samples were tested at 3, 7 and 28 days after casting under standard moist curing conditions. The compressive strength test were carried out in accordance with the SANS 5863 [12] method using an Amsler compression testing machine with a capacity of 2000 kN. All samples were weighed in a saturated, surface dry condition to an accuracy of 5 g before being tested;

2.5.2 Pore structure:

The durability index test for micro structure characterisation of the concrete mixtures were carried out on specimen obtained from concrete cube samples at between 28 and 32 days after casting and under saturated moist curing conditions. The oxygen permeability and water sorptivity test were performed in a temperature and humidity controlled room following the procedures outlined in the SANS test specification of 2010 [13], it is a revised version of the Durability Index Testing Manual originally developed by Ballim [14] (1994). At the appropriate time after moist curing concrete cube samples were removed from the laboratory room for the coring operation. The discs (70 ± 2 mm \varnothing and 30 ± 2 mm thick) obtained were then transferred to a 50 ± 2 °C ventilated oven to be dried for a minimum of 7 days \pm 4 hours before being tested for oxygen permeability and water sorptivity. The discs permeability and water sorptivity measurement were carried out using the falling head permeameter and water sorptivity tests respectively as described in the Durability Index Testing Manual.

2.5.3 Portlandite contents:

The portlandite content of the concrete mixtures was determined from specimen obtained from concrete cube samples after 3, 7 and 28 days of standard moist curing periods after casting. Thermogravimetric analysis (*TGA*) was performed on powdered specimens. The powdered specimens are obtained following the procedure described in Alhassan [5]. The procedure involve drilling to a depth of 20 ± 5 mm with a 7 mm diameter drill bit on the interior surface of a cube after it has been oven dried. The samples obtained are then sieved through a 63 μm sieve size and preserved under vacuum in airtight bottles until tested. The chemical composition of the specimens was determined in terms of the $\text{Ca}(\text{OH})_2$ content using the *TGA* test. The thermal decomposition behaviour of the powder specimens were studied by tracing mass and heat changes using a Perkin Elmer TG400 thermogravimetric analyser coupled to a Setaram differential analyser. The thermal analyser used in this study, enables the thermogravimetric (*TG*) curve and the derivative thermogravimetric (*DTG*) curve to be obtained simultaneously on the specimen. A plot of temperature against mass loss (*TG* curve) and derivative mass loss (*DTG* curve) is obtained automatically. The weight loss obtained from the *TG* and the *DTG* curves were used to estimate the amounts of calcium hydroxide ($\text{Ca}(\text{OH})_2$) and calcium carbonate (CaCO_3) present in the specimen;

2.5.4 Durability characterisation:

For the durability performance of the concrete mixture, the set of four cubes, exposed to each of the three natural sites after 14 days of preconditioning and taken to the laboratory after varying ages for carbonation testing. Carbonation measurements were done at ages of 6, 12, 18, and 24 months after exposure. A solution of 1 % phenolphthalein in ethanol was sprayed onto the freshly broken surface of the concrete and average of the depth of the colourless section was taken as the carbonation depth.

2.5.5 Exposure condition measurement:

The microclimatic conditions of the exposure sites were monitored using a 24-hour, automated data logger set up on each of the site to record the micro-climate conditions. The CO₂ concentration, relative humidity (RH) and temperature were monitored throughout the period of exposure. Data was recorded every one minute and downloaded monthly into a computer hard-drive. These micro-climate data were analysed for each exposure sites in terms of the mean value of CO₂ concentration, RH and temperature.

III. Experimental Results and Discussion

Results of tests and discussion on early-age characterisation of the concrete are given in Figures 1-4 for the compressive strength, permeability, sorptivity and chemical composition respectively. While, results for the micro-climatic variations for the exposure sites, where the concretes samples were kept in terms of CO₂, RH, temperature and precipitation as well as the results of the carbonation depth for the concrete cubes are presented in Figure 5 - 9. The results presented on the carbonation depths/rates are discussed in terms of the concrete physical and chemical properties while the influence of the exposure condition on the depth of carbonation was also examined and discussed

3.1 The development in strength for the different mixtures as influenced by w/b, and curing time is presented in Figure 1. As expected, compressive strengths increase steadily with decrease in w/b for any given mixture. Similarly, strengths increase progressively with age, as is typical of concrete mixtures under moist curing. Note that as the moist curing age of the concrete increases, hence the hydration of its binder components, the gain in compressive strength with CSF blended concretes becomes more apparent especially at low w/b. It may be recalled that while 28-day strength is a standard value, it is the 3 or 7 day strengths that are realistically representative of site curing practice. Analyzing the ratios of the 28 days and 7 days compressive strengths for all the concrete mixtures tested, the *CSF* concretes presented higher values compared to the *CEM I* concretes. Increased compressive strength gain at later ages for this *CSF concrete* can be attributed to its slower hydration rate and chemical pozzolanic effect, as well as to the physical effect of the generally tiny *SCM* particles. Detwiler and Mehta [15] and Goldman and Bentur [16] suggested in their work that the improvement in compressive strength in blended cement concrete is primarily dependent on the micro filler effect of the *SCMs*.

3.2 The permeability results for the concrete mixtures are presented in Figure 2. This result shows that, increasing w/b and decrease extent of initial moist curing of concrete resulted in decreased oxygen permeability index (OPI) values with the CSF blended concretes presenting higher OPI values. Although the influence of moist curing on the permeation properties of the concrete can be seen clearly in some of the mixtures, it is clustered for the others. However, there are distinct trends of improvement in concrete permeation as the w/b decreases. This is due to the increasing volume and degree of interconnection of capillary pores within the cement paste. In addition, as w/b decreases or as curing time increases (therefore the degree of hydration increases), the reduction of concrete paste porosity is mainly due to the reduction in pores of larger dimensions that have been filled or are connected by *C-S-H* gel pores. Graf and Grube [17] state that as hydration proceeds, the initially water filled and fully interconnected pores within the cement paste are filled with hydration products and become discontinuous. The increased gain in impermeability at later ages for the *CSF* blended concretes can be attributed to a combination of the pozzolanic and micro filler effects of *CSF*. The *CSF* blended concretes however performed better than the *CEM I* concretes at all w/b under continuous initial moist curing.

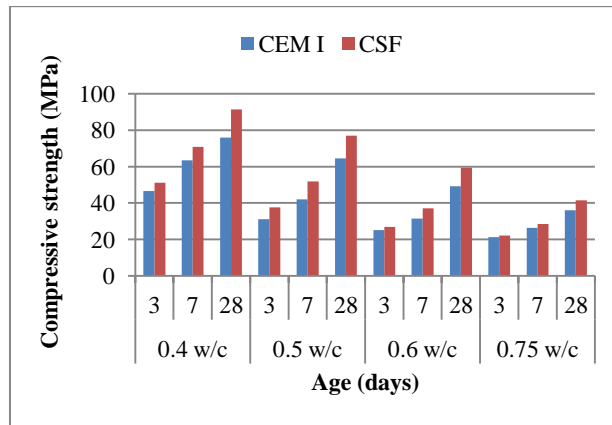


Fig.1 Compressive strength results for the concrete mixtures

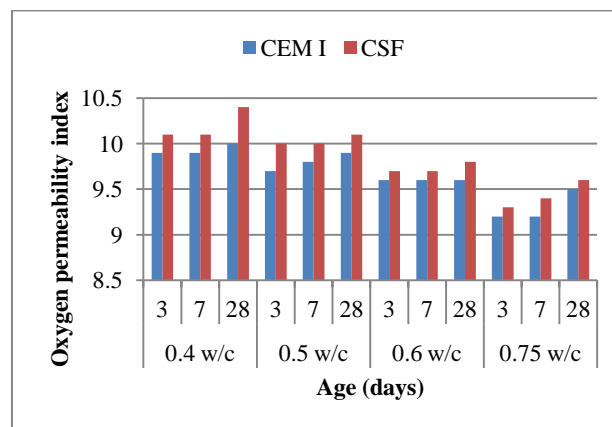


Fig. 2 Oxygen permeability results for the concrete mixture

3.3 Figure 3 present the sorptivity results for the concrete mixture. From the sorptivity plot presented, it can be seen that the sorptivity values decrease as the w/b reduces and the extent of initial moist curing period increases for all the concrete mixtures investigated. However, the decrease in sorptivity values as a result of the decrease in w/b are more marked than the increase in the extent of initial moist curing. Similar lower sensitivity of the concretes to the extent of extended initial moist curing was noted with the permeability results. The improved sorptivity for all the concretes at low w/b and prolonged duration of initial moist curing are attributed to the reduction in the volume and size of the pores within the concrete microstructures as well as the interconnections. The beneficial effect of SCMs in improving concrete pore structure can be seen for the CSF blended concretes in comparison to the CEM I concretes. CSF blended concretes presented better sorptivity values at lower w/b and extended initial moist curing periods. Improvement in sorptivity for these blended concretes can be attributed to the pozzolanic effect of the SCMs at later ages and also to their pore filling effect.

3.4 The portlandite contents for the concrete mixtures are given in Figure 4. The use of different binder type and content influenced the $Ca(OH)_2$ content of the concretes, with increasing w/b and decreasing binder contents resulting in decreased $Ca(OH)_2$ content for all the concrete mixtures investigated. From the $Ca(OH)_2$ plots presented, it can be noted that the $Ca(OH)_2$ content increases as the concrete moist curing ages increases for the plain concretes while a decrease in $Ca(OH)_2$ content were noted for the CSF blended concretes. The reason for the increase in portlandite for the plain concretes is that, CEM I concrete produces $Ca(OH)_2$ during hydration and its content increases with hydration time. While, the decrease in $Ca(OH)_2$ content for the blended cement concrete can be attributed to both the high dilution effect related to the fact that $Ca(OH)_2$ results from cement hydration which in turn is directly related to the cement proportion in the mixture, as well as the pozzolanic reaction between $Ca(OH)_2$ and CSF which consumes some $Ca(OH)_2$, and may extend over a long time period [18]. It can also be noted from the figure that the $Ca(OH)_2$ content for both the blended and unblended concretes are not significantly different at 3 and 7 days. This shows that CSF have little effects on the normal hydration process of CEM I concretes at early ages. This behaviour is in agreement with earlier works by Marsh and Bonnery [19] and Marsh and Day [20].

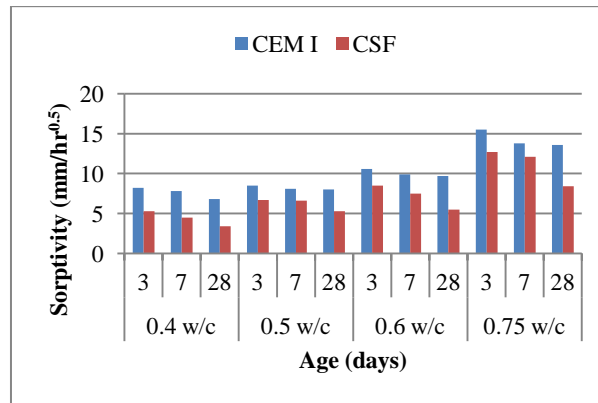


Fig. 3 Sorptivity results for the concrete mixtures

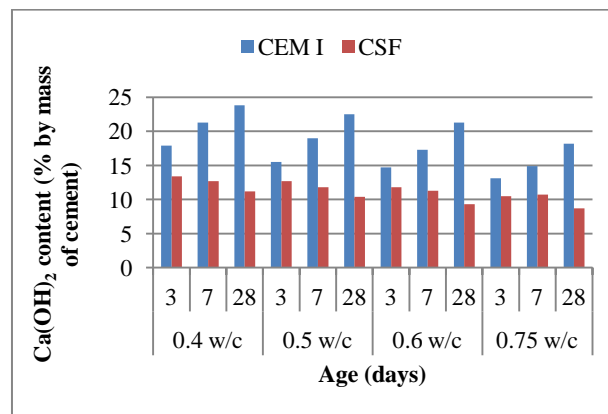


Fig. 4 Ca(OH)₂ content results for the concrete mixtures

3.5 The micro climate results for the exposure sites in terms of CO₂ concentration, relative humidity and temperature are presented in Figures 5 to 7, while Figure 8 shows the annual precipitation results for the town as obtained from the literature. Overall, the average annual micro- climatic conditions for indoor exposure site are 291 ppm CO₂ /34% RH/24°C temperature while for the outdoor exposure sites are 248 ppm CO₂ /49% RH/19°C temperature. From the figures, it can be observed that the variability in CO₂ concentration between the exposure sites is not significant. However, slightly higher CO₂ values are observed for the indoor exposure site in most months and this can be attributed to the enclosed nature of the indoor site and the lack of free flow of air. Note also that the indoor exposure site is a basement parking garage and would be influenced by CO₂ from the exhaust fumes of cars. The outdoor exposure condition, unsheltered from rain and sun presented the least CO₂ concentration. Based on the fact that external RH influences the internal humidity of the exposure. Concrete exposed on this site will have lower carbonation depth. Reasons are that these concrete will have better pore structure because of the continued hydration. Additionally, the pores will be saturated during the summer months and dry out faster during the winter months. The RH result presented shows that concrete cube samples exposed outdoor but sheltered from rain and sun will have the highest carbonation rates because the RH profile mostly lies in the zone (50 – 70%) for maximum carbonation rate.

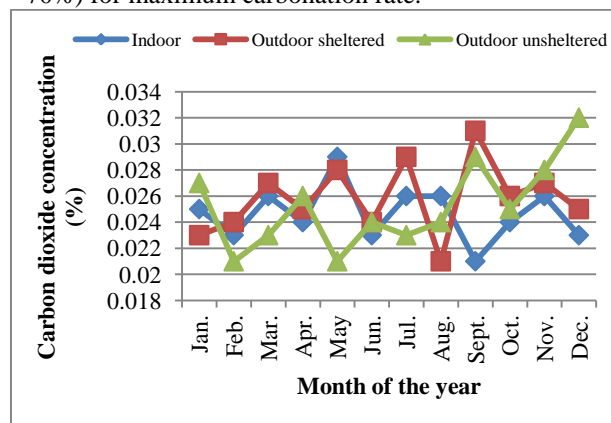


Figure 5 Variation in atmospheric CO₂ content at the different exposure sites [5]

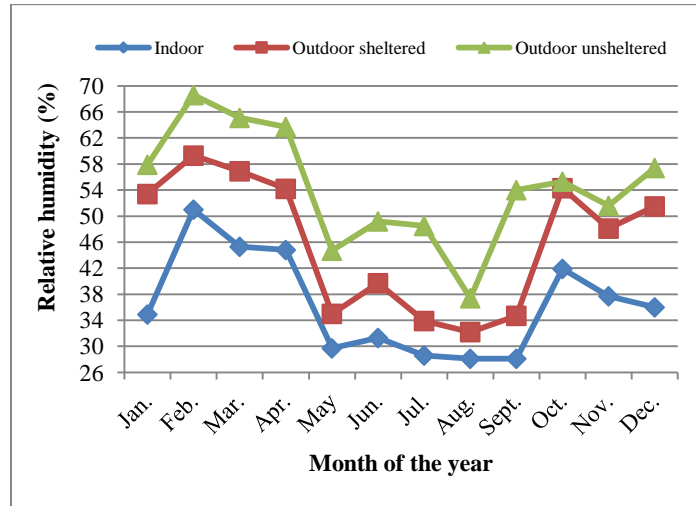


Figure 6 Variation in relative humidity at the different exposure sites [5]

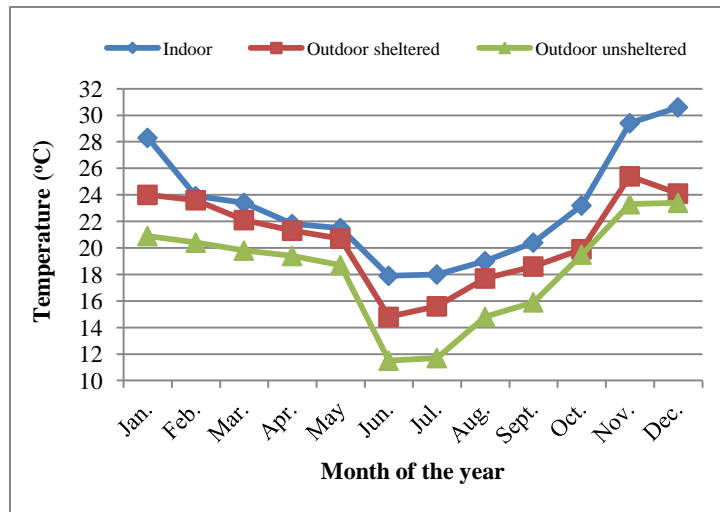


Figure 7 Variation in atmospheric temperature at the different exposure sites [5]

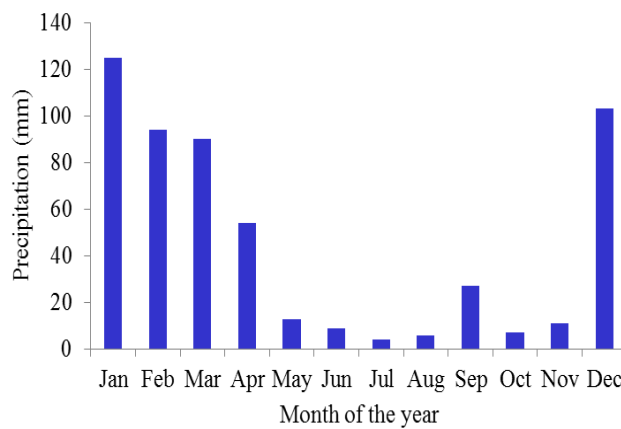


Fig. 8 Annual average precipitations in Johannesburg [21]

The RH of the exposure sites is influenced by precipitation and the influence is higher for samples outdoor and unsheltered. The influence of temperature variations is not very significant for concrete carbonation as noted from this investigation, and from the literature [22]. However, results of the temperature variation shows that carbonation rates should be lower for concrete cube samples exposed indoors, while it may be similar for the other two exposure sites since their temperature profiles are similar.

3.6 From the carbonation coefficients results for the different concrete types, it is evident that the carbonation rate of the concretes followed similar pattern for the different w/b investigated. Hence, it is only the 0.5 w/b concretes carbonation rates results that is presented for the different exposure sites (see Figure 9). Generally, the trends observed were distinctly influenced by the concrete physical and chemical characteristics as well as the micro climate condition of the exposure sites. Thus, variations of the carbonation rates for the concretes will be explained in relation to each of these influencing factors. The general carbonation behaviors of the mixtures under indoor, sheltered and unsheltered exposure conditions are compared in Figure 9. Clearly, the concretes stored under indoor and sheltered exposure give higher carbonation rates than those kept under the exposed conditions. The differences in carbonation are related to effect of rain for the exposed samples, which tends to temporarily block the pores of surface concrete, thereby inhibiting CO₂ ingress during periods of precipitation. Again the wetting and drying period experienced by samples on the exposed site is probably the reason for the reduced carbonation rate, since both the outdoor sheltered and unsheltered exposure conditions have similar micro-climates in terms of CO₂ content, RH and temperature. In addition, concrete cube samples exposed outdoor to rain and sun have higher internal humidity during the summer months from precipitation effects, thus reducing CO₂ ingress during this period. Furthermore, these concretes had improved pore structures from rapid hydration effects as a result of its high internal moisture content during the summer months.

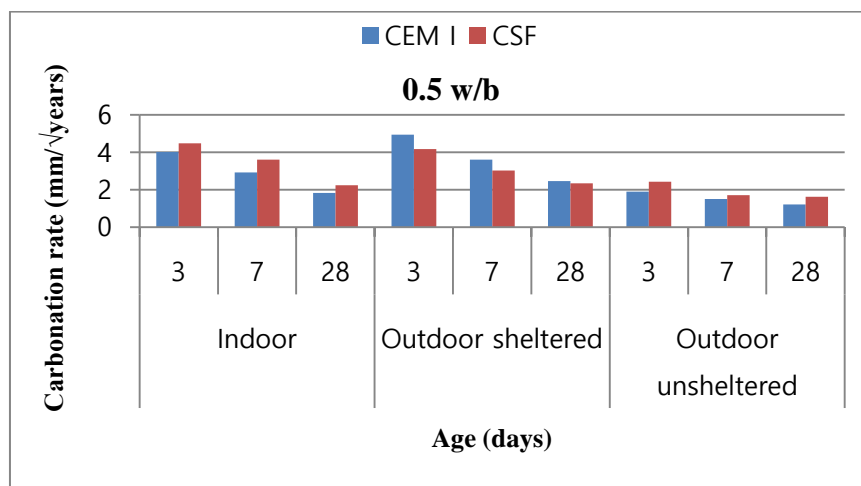


Fig. 9 Carbonation rate results for the various concrete mixtures

The influence of CSF is clearly evident in the concrete, generally showing increase in carbonation. Under sheltered exposure, CSF behaved similarly as CEM I, with both mixtures giving relatively high carbonation progressions compared to the other exposure and under prolonged curing. The different behavior of CSF mixes under unsheltered exposure may be related to availability of precipitation which in turn promotes pozzolanic activity in a manner not attainable under the partial saturation conditions of sheltered exposures. From Figure 9, It can be seen that CSF addition led to a greater carbonation rate, which is consistent with the trend observed by Osborne, [23] and Papadaki, [24]. The changes in pore structure were not adequate in CSF concrete to counteract the reduction in CH content, and hence the carbonation rate increased. In the presence of CSF, the amount of CH available to react with CO₂ is lower, for two reasons; first, less CaO is added to the concrete. Second, some of the CH reacts with the added CSF. Thus, if the concrete pore structure modification does not govern over the reduction in CH, the carbonation may proceed faster due to the fewer amounts of carbonatable materials available per unit area of CO₂ to react with. This explains why carbonation tends to proceed faster in concrete containing mineral admixture. However, due to the slower pozzolanic reaction, porosity can be expected to be higher during the initial stage, thus allowing more rapid diffusion of CO₂.

Generally, carbonation rate increases with increase in w/b irrespective of the concrete initial moist curing periods. This is attributed to increased pore connectivity of the concrete since w/b ratio primarily determines the gel/space ratio, the capillary porosity and thus the permeability of the concrete. Concretes of lower w/b had better resistance against the diffusion of CO₂, possibly due to the denser pore structure and higher Ca(OH)₂ content. Similar findings in relation to the effect of low w/b ratio on carbonation have also been reported in the literature [25, 26, 27]. The decrease in the carbonation rates of the concretes at lower w/b can be attributed to the fact that the cumulative pore volume and the amount of pores are lower compared to a higher w/b concrete. This fact was also evident from the permeation test results of the concretes (see Figures 2 and 3). Hence, the lower w/b concretes, showed superior resistance against carbonation.

IV. Conclusions

From this investigation and based on the mixture proportion, cementitious materials, and test conditions adopted in this study, the following conclusion can be drawn: A lower w/b and prolonged curing age of concrete in water generally led to a slower rate of carbonation, mainly due to pore structure densification, as indicated by a decrease in cumulative and differential pore volume;

For concrete with the same compressive strength, there is no significant difference between the rate of carbonation between plain and blended cement concrete, because the increased tendency to carbonate by blended cement concrete is offset by its increased permeability;

Structural elements exposed outdoor but sheltered will present the highest carbonation rate. Thus, such elements should be more carefully designed and constructed if its design service life in terms of the initiation limit state is to be achieved;

It is evident that concrete elements exposed outdoor to rain and sun are at risk of corrosion due to the high RH as a results of the occasional precipitation during summer months. However, the risk of carbonation may be very low for such concrete elements because of the low or high saturated pore structure, depending on the period of the year; The cumulative pore volume for CSF blended cement concrete was smaller than 100% plain cement concrete. The pore size modification due to pozzolanic reaction of CSF had better pore volume and hence the densest pore structure. Thus, the carbonation coefficients of these CSF mixtures were lower than that of the plain concrete.

Acknowledgements

This work is based on the research carried out at the University of the Witwatersrand, South Africa. The first author wishes to thank Prof Yunus Ballim, of the School of Civil and Environmental Engineering, University of the Witwatersrand, South Africa, for supervision and financially supporting the research.

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Alhassan. "Physical and Chemical Characteristics Affecting the Durability of Condensed Silica Fume (CSF) Blended Cement Concrete." IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE) 14.4 (2017): 01-10.