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Properties of Western Cape Concretes with Metakaolin

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Abstract. A global demand for affordable, sustainable, and durable concrete has resulted in growing use of Supplementary Cementitious Materials (SCMs). In the Western Cape Province of South Africa, the readily available SCM is Granulated Ground Corex Slag (GGCS), although fly ash can also be obtained. However, the availability of these SCMs, particularly GGCS, is subject to market and other extraneous factors, and this may render them vulnerable as sources of SCM for concrete. This points to the need for innovation and investigating other potential SCMs which are economically and environmentally effective. Metakaolin, a high-grade type of calcined clay, emerges as a possible potential future SCM in the Western Cape. This study aimed at investigating the influence of a locally available metakaolin on mechanical (compressive and tensile strength), and durability (concrete penetrability and potential to mitigate Alkali Silica Reaction (ASR)) properties of Western Cape concrete. In comparison to GGCS, concretes with metakaolin showed superior performance in both mechanical and durability properties. This was attributed to its role in concrete in terms of accelerating hydration reactions, pozzolanic activity, and dilution effect. Metakaolin can therefore be regarded as a beneficial substitute for GGCS in Western Cape concrete. However, questions that remain include cost-effectiveness, and the awareness and willingness of industry to incorporate this material.

1 Introduction

Approximately 12 billion tons of concrete produced in the construction industry uses 1.6 billion tons of Portland cement, annually [1]. This contributes to high kiln fuel expenditure which affects production costs, and high carbon dioxide (CO_2) emissions to the atmosphere, hence greenhouse gas effects. Manufacture of cement clinker produces 0.8 tons of CO_2 per ton of cement [2], accounting for 5-8% of worldwide anthropogenic CO_2 emissions [3]. This has led to global adoption of the use of Supplementary Cementitious Materials (SCMs) in concrete, for purposes of not only reducing the cement content, but also sustainable use of industrial by-products such as silica fume, fly ash, and slag. This results in reduction of clinker production and a consequent decrease in CO_2 emissions and production costs [4].

Use of these SCMs has generally proven to improve strength and enhance durability of concrete [5]. This has caused a global demand for SCMs, raising the need for innovation and investigating other potential SCMs which are economically and environmentally effective. For instance, in the Western Cape Province of South Africa, Corex slag is currently used as an SCM. It is difficult to guarantee its availability since it is a by-product of the iron and steel production industry (Saldanha Steel). It is possible that the decline of steel production or other global factors may lead to scarcity of this material. On the other hand, fly ash produced at electrical power stations may compensate during any scarcity of Corex slag. However, fly ash itself has negative environmental impacts: its production involves burning coal which produces greenhouse gases [3], and it is produced remotely from the Western Cape.

Therefore, an alternative possible candidate SCM for use in Western Cape concrete could be metakaolin. Metakaolin is a highly reactive pozzolanic material which is obtained by calcining kaolin clay in a dehydroxylation process at a temperature range between 600°C and 900°C, see equation 1[2]. However, an optimum temperature appears different in the literature because it mostly depends on the structural layer of stacking order of kaolin clay the calcination techniques (traditional or thermal calcination) which are associated with metakaolin reactivity, and. Some literature, specify that at the temperature range between 550°C and 600°C, disordered kaolin clay is dehydroxylated to produce a more reactive metakaolin, while the temperature above 600°C, ordered kaolin clay is dehdroxylated to produce a less reactive metakaolin [6,7].

Kaolin clay is available in different locations in the Western Cape such as Cape Town (Atlantis, Noordhoek-Fish Hoek Valley, Brackenfell-Kuils River area, and Stellenbosch-Somerset West area), Vredenburg peninsula, and southern Namaqualand areas [8]. Exploitation of these clays and converting them into Metakaolin would assist in the concrete construction industry and help solve scarcity of SCMs.

This study focuses on investigating properties of concrete containing a locally available metakaolin for use as an alternative SCM in the Western Cape concrete. The study concentrates on assessing the influence of metakaolin on mechanical and durability properties of concrete and comparing its performance with the properties of concrete with corex slag.

$$\overset{\text{Kaolin clay}}{Al_2Si_2O_5(OH)_4} \xrightarrow{600^{o}C\text{-}900^{o}C} \overset{\text{Metakaolin}}{Al_2O_3.2SiO_2} + 2H_2O \quad (1)$$

2 Materials and methods

Portland limestone cement, CEM II/A-L 52.5N from PPC Cement, was used to prepare the concrete. Metakaolin manufactured by the Kaolin Group and supplied by Serina Trading Company was used to partially replace cement at rates of 10%, 15% and 20%. Ground Granulated Corex Slag (GGCS) from PPC Cement, at 50% replacement level, was also used for the purpose of comparing its performance with concrete containing metakaolin. Table 1 shows chemical properties of the cement, metakaolin, and GGCS.

The locally available greywacke aggregates and sands i.e. dune sand and greywacke crusher sand at a ratio of 60:40, were used to prepare concrete mixes. A total of fifteen mixes were prepared as shown in Table 2. Three water/binder (w/b) ratios, i.e. 0.4, 0.5, and 0.6, were used to assess the influence of metakaolin at different w/b ratios. Water and aggregate contents were kept constant in all mixes, at

185 kg/m^3 and 1000 kg/m^3, respectively. Concrete slump was regulated at 100 mm by use of a superplasticizer (CHRYSO® Plast Omega 103).

Mechanical properties tested were compressive strength, tensile splitting strength, and static elastic modulus. Strength tests involved casting six concrete cubes of 100 mm for each age (7, 28, and 56 days) per mix, with three cubes per test. The tests were conducted according to SANS 5863 and SANS 6253 [9,10], respectively. Static elastic modulus tests involved casting three concrete cylinders of 100 mm diam. x 200 mm per mix. At the age of 28 days, the cylinders were tested for elastic modulus according to BS 1881: Part 121[11].

Durability properties of concrete were assessed in terms of concrete penetrability, and the potential for metakaolin to mitigate Alkali Silica Reaction (ASR). Concrete penetrability was assessed by Durability Index (DI) tests i.e. Oxygen Permeability Index (OPI), Water Sorptivity Index (WSI), and Chloride Conductivity Index (CCI), as per SANS 3001-CO3-1&2 and the Durability Index Manual [12-14].

Table 1: Chemical composition of cement, GGCS and metakaolin

Chemical formula	Chemical composition, %		
	CEM II A-L 52.5N	GGCS	Metakaolin
SiO_2	19.77	31.32	52.81
Al_2O_3	3.24	17.04	42.02
Fe_2O_3	3.11	1.00	0.32
Mn_2O_3	0.06	0.05	0.03
TiO_2	0.19	0.58	1.30
CaO	63.84	35.15	0.02
MgO	1.28	11.76	0.07
P_2O_5	0.14	0.03	0.09
SO_3	2.55	3.04	0.00
K_2O	0.61	0.63	0.06
Na_2O	0.22	0.00	0.00
SrO	0.26	0.00	0.00
LOI	4.63	-	1.16
Total	99.9	99.3	97.9

Table 2: Concrete mix proportions

w/b	Mix	Variables	Cement	Metakaolin & Corex slag		Water	Aggregate		
			CEM II	mk	GGCS		Grey-wacke	Dune sand	Crusher sand
		SI unit				kg/m^3			
0.4	1	0% mk	463	-	-	185	1000	544	305
	2	10% mk	416	46	-	185	1000	534	300
	3	15% mk	393	69	-	185	1000	529	297
	4	20% mk	370	93	-	185	1000	524	294
	5	50% GGCS	231	-	231	185	1000	533	299
0.5	1	0% mk	370	-	-	185	1000	597	335
	2	10% mk	333	37	-	185	1000	590	331
	3	15% mk	315	56	-	185	1000	586	328
	4	20% mk	296	74	-	185	1000	582	326
	5	50% GGCS	185	-	185	185	1000	589	330
0.6	1	0% mk	308	-	-	185	1000	633	355
	2	10% mk	278	31	-	185	1000	626	351
	3	15% mk	262	46	-	185	1000	623	350
	4	20% mk	247	62	-	185	1000	620	348
	5	50% GGCS	154	-	154	185	1000	626	351

The accelerated mortar bar test as per ASTM C 1567-13 [15] was performed to determine the potential for metakaolin to suppress ASR expansion of a deleteriously reactive aggregate, namely the local greywacke.

3 Results and Discussion

The results are discussed below, regarding the concrete properties indicated previously. Dashed lines in the figures are used to distinguish concretes that were cast with a different batch of aggregates, which however, were of the same type and source. The points connected by solid lines represent concretes cast with same aggregate batches.

3.1. Compressive strength results

Figure 1 shows results for compressive strength of the fifteen concrete mixes, at three w/b ratios. The strength decreased with increase in w/b ratio, while it increased with curing age for all concretes. Metakaolin concretes had higher strength than the control (0% mk) and GGCS mixes. It was observed that as metakaolin content increased to 20%, the strength also increased. This was due to the high pozzolanic reactivity of metakaolin, primarily calcium hydroxide (CH) reacting with the components of metakaolin (silica and alumina). The reaction results in the production of secondary calcium silicate hydrates (CSH) which assisted in increasing the strength of concrete. Similar trends of strength increase with metakaolin content were observed by Mermerdas *et al* and Wild, Khatib and Jones [14,15], while Badogiannis *et al.* and El-Diadamony *et al.* [18,19] indicated that 20% replacement decreased the strength due to the dilution effect.

Figure 2 shows relative compressive strength of the concretes. Compressive strengths of the control were used as the references to study the influence of partial replacement of metakaolin and GGCS in concrete. It was observed that metakaolin concretes had the highest influence on boosting strength. GGCS concrete had less strength development especially at the early age.

Metakaolin showed an excellent potential to improve compressive strength, with strength increasing with metakaolin content. The maximum strength increases were achieved at 20% replacement level, with magnitudes of 36%, 42% and 47% for 0.4, 0.5, and 0.6 w/b, respectively. For 0.4 and 0.5 w/b concretes, the highest relative strengths were achieved at the age of 28 days, while for 0.6 w/b, it was at 7 days. The maximum strength increases were achieved at the early age for 0.6 w/b concretes. This was deduced as the influence of high water content in relation to cement content that accelerated hydration reactions, consequently, pozzolanic reaction started to take place earlier. However, a decreased rate in strength development at 0.6 w/b at 28 and 56 days was likely associated with depletion of CH content that led to decline of the metakaolin pozzolanic potential.

3.2. Tensile splitting strength

Figure 3 presents tensile splitting strength of concrete with metakaolin at different proportions, and with GGCS. As expected, the strength decreased with increase in w/b ratio. It was observed that metakaolin concretes had higher strength than control and GGCS concretes. As metakaolin replacement level increased, the strength also increased, although with minor increments. The highest strength was achieved at 20% replacement rate for all concretes. This opposed the results obtained by Qian and Li, and Shehab El-Din *et al.*[20,21] who claimed that the maximum tensile splitting strength of concrete was achieved between 10% and 15 %

replacement rate, and above that, the strength decreased.

Figure 4 shows relative tensile splitting strength of concretes at all w/b ratios. The higher strength development was more pronounced in metakaolin concretes than in GGCS concretes. Strength also increased with metakaolin content. The maximum increases were achieved at 20% replacement, with magnitudes of 39.5%, 39.6%, and 40.9% at 7 days for 0.4, 0.5, and 0.6 w/b, respectively. Further, as hydration time increased, the rate of strength development decreased. It was observed that almost all metakaolin concretes experienced the highest strength developments at 7 days, which decreased at 28 days, while in some concretes, there was a slight increase at 56 days.

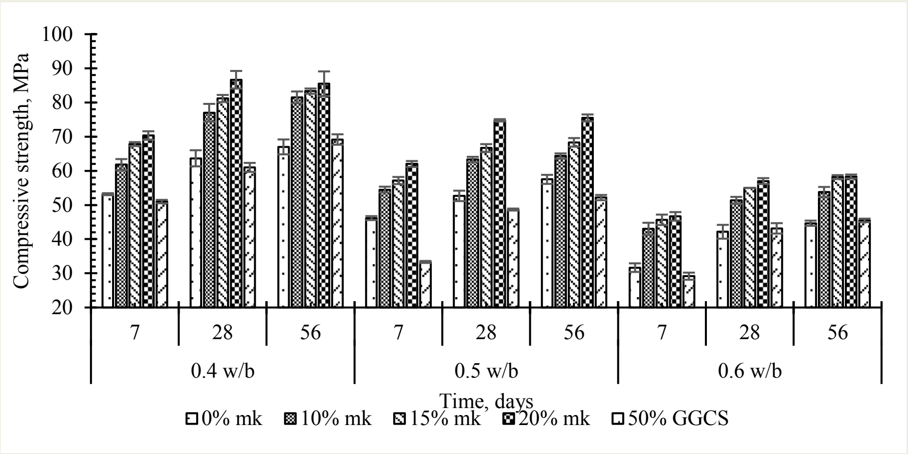

Figure 1: Compressive strength of concrete containing metakaolin and GGCS at three w/b ratios and three ages

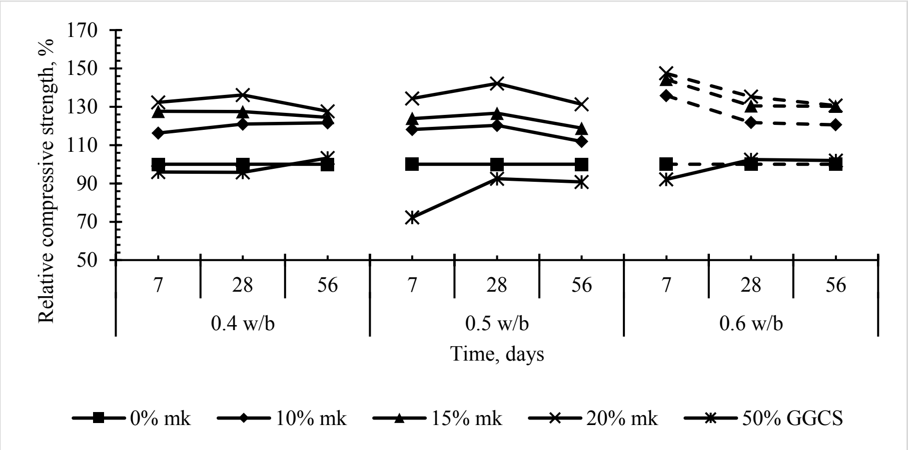

Figure 2: Relative compressive strength of concrete with metakaolin and GGCS at three w/b ratios

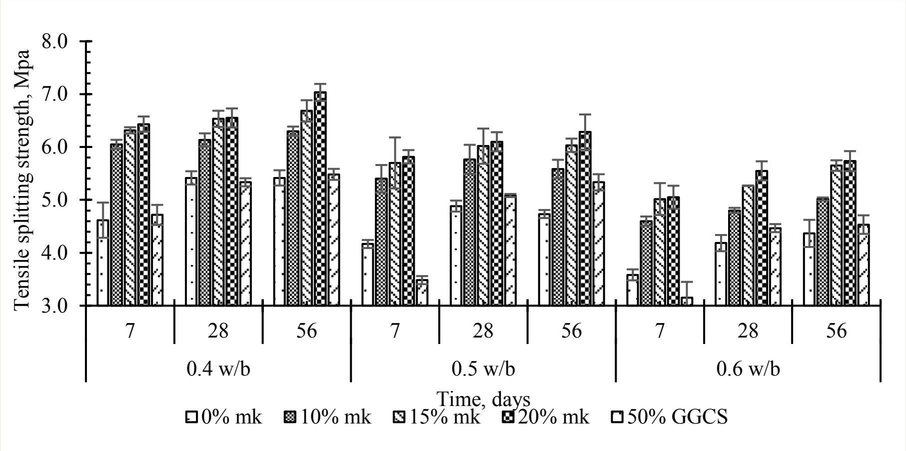

Figure 3: Tensile splitting strength of concrete containing metakaolin and GGCS at three w/b ratios

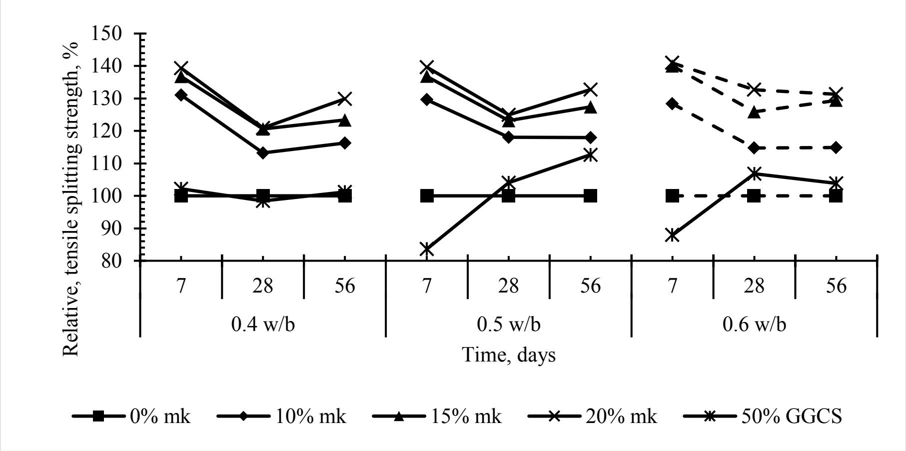

Figure 4: Relative tensile splitting strength of concrete with metakaolin and GGCS at three w/b ratios

3.3. Static elastic modulus results

Static elastic moduli of concretes were studied at 28 days for the range of concretes shown in Figure 5. Elastic modulus of concrete decreased with increase in w/b ratio. The highest values were obtained at 0.4 w/b, and the lowest at 0.6 w/b. Metakaolin improved the elastic modulus of concrete, although there was no definite trend between the improvement of elastic modulus and increase in metakaolin content. At 0.4 w/b, 10% mk had the highest value followed by elastic modulus decrease with increase in metakaolin. At 0.5 w/b, elastic moduli were almost similar, while at 0.6 w/b, 10% and 15% mk showed higher values. These results differed from those found by Qian and Li [21] who observed increasing elastic modulus with increase in metakaolin content. They explained this was due to the influence of metakaolin in increasing compressive strength and improving the interfacial transition zone.

By comparing the influence of metakaolin and GGCS, at 0.4 and 0.5 w/b, both SCMs proved to have similar influence on elastic modulus. However, at 0.6 w/b, GGCS seemed to have higher values. Generally, both SCMs played a good role in increasing the static elastic modulus of concrete.

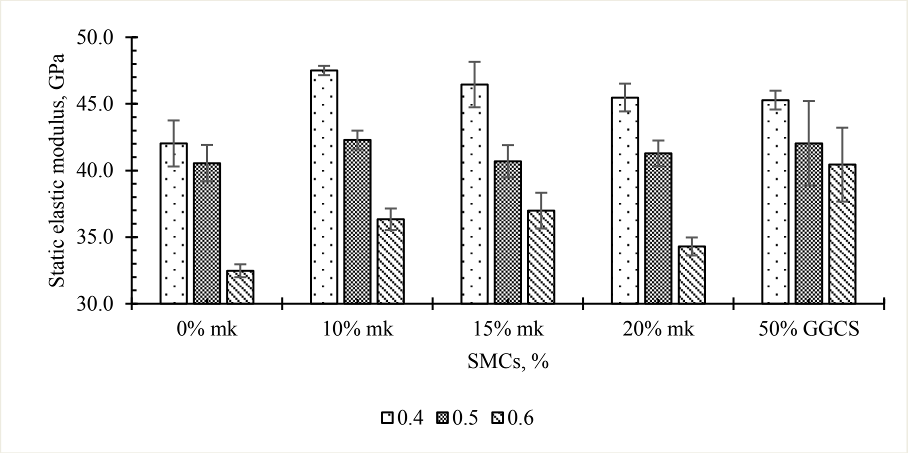

Figure 5: Static elastic modulus of concrete with metakaolin and GGCS at 28 days of curing

3.4. Durability index (DI) results

A durability index (DI) is a concrete quality parameter which characterises the concrete at an early age according to relevant transport mechanisms, i.e. oxygen permeability for permeation, water sorptivity for absorption, and chloride conductivity for diffusion. DIs give information about the microstructure of concrete and are also used to predict durability performance of concrete [23, 24]. In this study, the influence of metakaolin in concrete in altering the microstructure and improving durability performance were studied. The results obtained are discussed according to criteria for the quality of concrete, see Table 3, then, compared with the performance of control and GGCS concretes.

Table 3: Criteria for the quality of concrete using Durability Index (DI) values [25]

Quality of concrete	Oxygen permeability Index (OPI) log scale	Water sorptivity index (WSI) mm/h$^{0.5}$	Chloride conductivity index (CCI) mS/cm
Excellent	>10	< 6	< 0.75
Good	9.5 - 10	6 - 10	0.75 - 1.50
Poor	9.0 - 9.5	10 - 15	1.50 - 2.50
Very Poor	< 9	> 15	> 2.50

3.4.1.Oxygen Permeability Index (OPI) results

Figure 6 shows results for OPI of concrete with metakaolin and GGCS at different w/b ratios, while Figure 7 shows their corresponding D'Arcy coefficients of permeability (k). OPI is the negative log of the D'Arcy coefficient of permeability of concrete. According to Table 3, all concretes showed 'excellent' quality despite having different quantities and types of constituents in terms of metakaolin, GGCS, and w/b ratios.

At all w/b ratios, the OPI values increased with metakaolin content in the order of 0%, 10%, 15%, and 20% mk. The reduction of permeability coefficient with increase in metakaolin content was as follow; 32%, 37%, and 38% at 0.4 w/b; 8%, 21%, and 23% at 0.5 w/b; and 40%, 42%, and 54% at 0.6 w/b, for 10%, 15%, and 20% mk, respectively. The highest reductions in permeability were observed at 0.6 w/b. This implies that metakaolin content had the highest influence in reducing permeability at a high w/b. This might be associated with the fact that, at 0.6 w/b, there was enough space to accommodate pozzolanic reactions. However, permeability reduction was higher at 0.4 than at 0.5 w/b, because of the influence of low w/b ratio. This was caused by the lower production of capillary pores during hydration, plus the effect of metakaolin. These results were supported by Mobasher et al., Güneyisi *et al* and Badogiannis and Tsivilis [22–24]. They reasoned that a high potential of metakaolin in reducing gas permeability was associated with its ability in refining pores and reducing pore connectivity in concrete.

The influence of GGCS in comparison to metakaolin was also studied. It was found that GGCS had higher OPI values than the control, with values similar to those at 10% mk. GGCS reduced the coefficient of permeability in comparison to the control by 25%, 14%, and 23% at 0.4, 0.5, and 0.6 w/b, respectively. Generally, the inclusion of SCMs improved the microstructure of concrete by reducing its permeability, with metakaolin showing the highest potential especially at higher replacement levels.

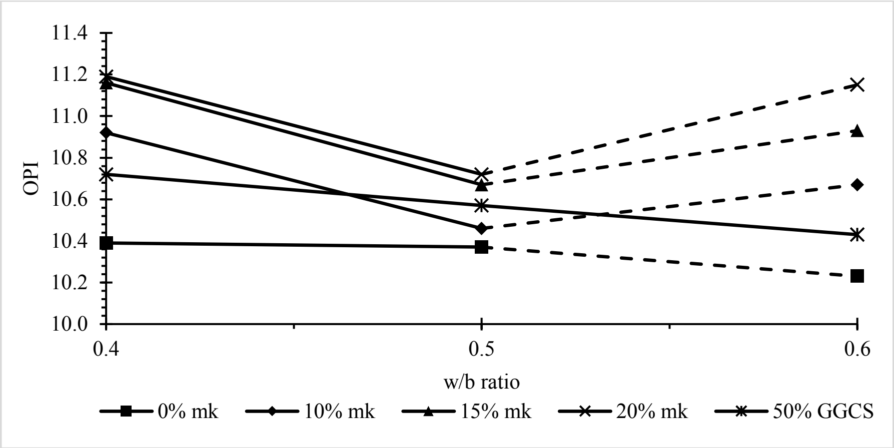

Figure 6: Oxygen permeability index (OPI) of concrete at different w/b ratios

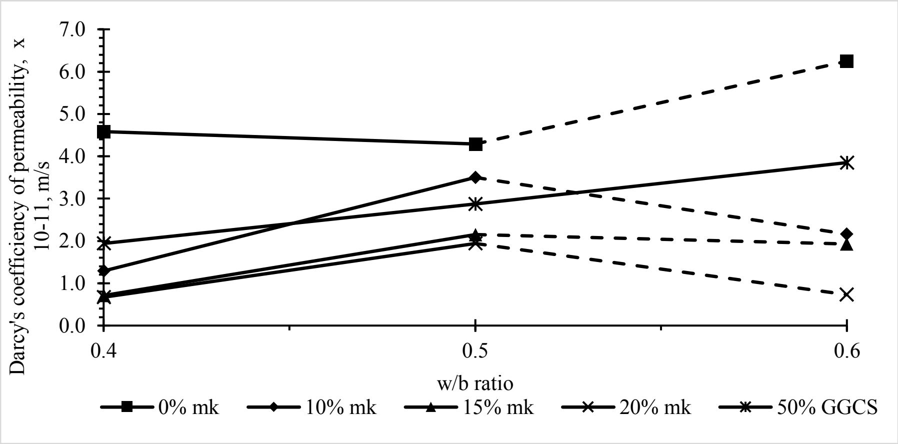

Figure 7: D'Arcy coefficient of permeability of concrete at different w/b ratio

3.4.2.Water Sorptivity Index results

Results for water sorptivity index (WSI) of concrete containing different proportions of metakaolin and 50% GGCS are shown in Figure 8. WSI represents the ability of concrete to absorb water by capillary suction. The results showed that all WSI values were below 10 mm/h$^{0.5}$ that represented ‘good’ quality concrete according to Table 3

The influence of metakaolin is clearly seen in Figure 8, with sorptivity decreasing (i.e. improving) as metakaolin content increased. Sorptivity decreased with metakaolin content for mixes in the order of 0%, 10%, 15%, and 20% metakaolin content, at all w/b ratios except at 0.4 w/b where 10% mk seemed to have a slightly higher value than the control. This implies that the capillary suction capacity of concrete was lower when a higher replacement of metakaolin was used in concrete. This was also associated with the capacity of metakaolin in reducing concrete porosity as is shown in Figure 9.

The inclusion of metakaolin in concrete showed the highest potential in the reduction of porosity in concrete. Firstly, it was observed that with increase in w/b ratio, porosity also increased. The highest influence was observed at 0.6 w/b ratio, where the porosity of control concrete was reduced by about 40% at 20% metakaolin. Other porosity reductions were 13% and 25% for 10% and 15% mk, respectively. At 0.5 w/b. the same behaviour was observed with porosity reductions of 8%, 12%, and 24% for 10%, 15%, 20% mk, respectively. At 0.4 w/b, the porosities were close to each other with values less than 6%, this might be associated with the effect of low w/b ratio.

It was found that GGCS also decreased porosity, with porosities at 0.5 and 0.6 w/b ratios lying between those for 15% and 20% mk. GGCS performed well in reducing porosity. Generally, it was concluded that metakaolin at a higher replacement rate has a higher potential in decreasing both water sorptivity and porosity in concrete than GGCS

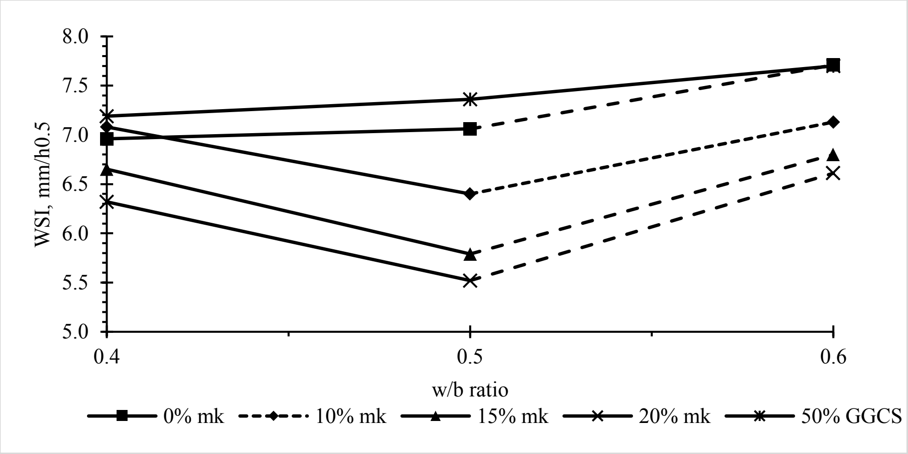

Figure 8: Water sorptivity index of concrete with w/b ratio

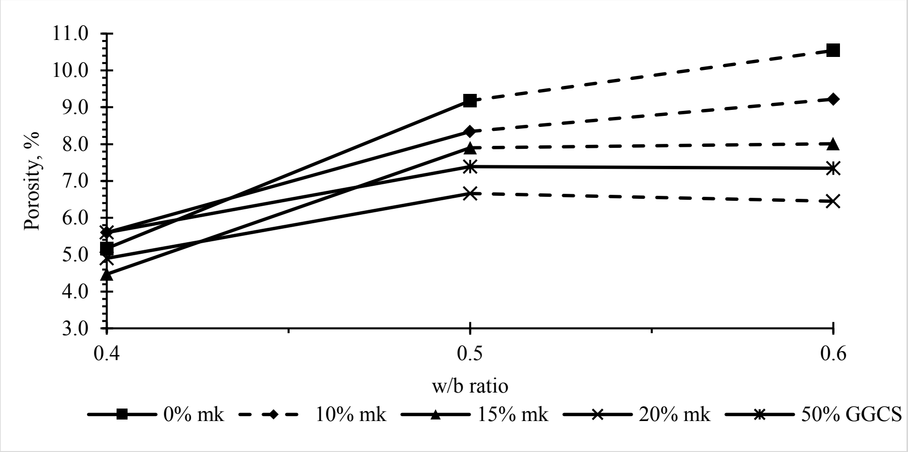

Figure 9: Porosity of concrete with metakaolin at different replacement levels and GGCS at 50% replacement level.

3.4.3. Chloride Conductivity Index results

The influence of metakaolin and GGCS in resisting transport of chloride ions in concrete was studied, using the chloride conductivity index (CCI). Results are shown in Figure 10. All concretes showed high resistance to chloride transport, with all CCI values below 0.75 mS/cm. These concretes were categorised as ‘excellent’ quality. The control at 0.6 w/b however showed values between 0.75 mS/cm and 1.5 mS/cm which was categorised as ‘good’ quality concrete.

The control concrete showed the highest values. With substitution of cement with SCMs, the chloride conductivity decreased markedly. All concretes with metakaolin showed excellent results. At 0.4 w/b, CCI values were very similar. At 0.5 w/b, the influence of metakaolin started to be seen. As metakaolin increased, the resistance to chloride ingress increased, with 15% and 20% metakaolin having similar low values. Metakaolin content influence was also clearly seen at 0.6 w/b. 20% mk showed the highest resistance followed by 15% and 10% mk.

Reasons for the influence of metakaolin on resisting chloride conductivity, were firstly, its potential to improve the microstructure of concrete by refining and reducing the pores and reducing pore interconnectivity, and secondly, due to its chloride binding capacity due to its high alumina content. Alumina that is a constituent of metakaolin tends to react with chloride ions to form calcium chloro-aluminate, commonly known as Friedel's salt, consequently reducing free chloride ions in the pore system for conductivity [29].

The influence of GGCS on CCI compared with metakaolin was also studied. GGCS was also effective in resisting chloride conductivity, but metakaolin was more effective at replacement levels above 10%. GGCS also resists chloride ingress by a similar mechanism to metakaolin, however, due to its lower alumina content, its influence was similar to that at 10% mk.

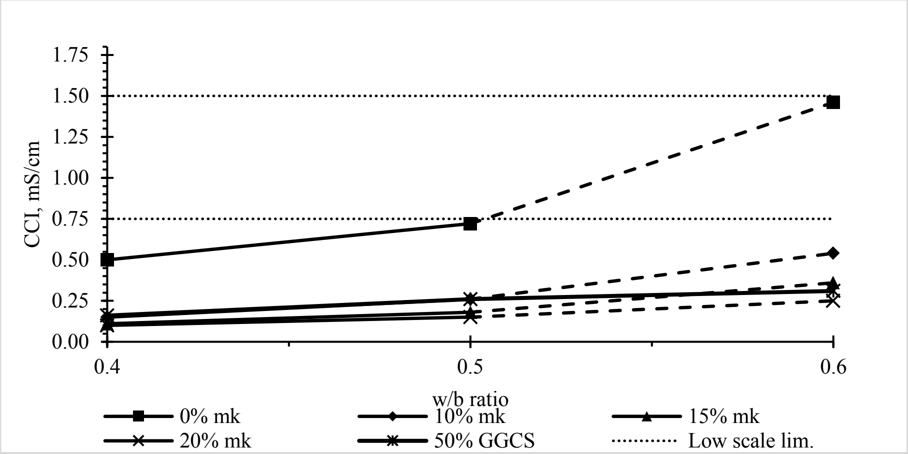

Figure 10: Chloride conductivity index (CCI) values of concrete at different w/b ratio

3.5. Alkali Silica Reaction (ASR)

In this study, metakaolin was used to supress ASR of a known deleterious reactive aggregate, greywacke. Different proportions of metakaolin were used to manufacture mortar bars. Their expansions were measured as per ASTM C 1567-13[15]. The replacement level at which the expansion was less than 0.10% after 16 days of testing (i.e. a total of 24 h of casting and demoulding, 24 h of curing in hot water at 80ºC, and 14 days of immersion in NaOH solution at 80ºC), was considered as an appropriate level at which metakaolin suppressed ASR expansion in concrete.

The results for ASR expansion are shown Figure 11. As expected, the control mortar showed the highest expansion which increased with time at the highest rate. The maximum expansion measured on the control mortar was 0.27 %. This result supported literature about the behaviour of greywacke as a deleterious reactive aggregate. The replacement by metakaolin showed excellent results; as its content increased, the expansion also decreased, with the lowest value being achieved at 20% mk.

The expansion reduction with metakaolin was 61%, 71%, and 89% for 10%, 15% and 20% mk, respectively. More than 50% of the expansion measured on the control mortar was reduced by the addition of metakaolin. This was a result of pozzolanic and dilution effects that reduced the quantity of alkalis to react with the silica phase in greywacke aggregate [30]. Therefore, it was concluded that metakaolin above 10% replacement level has a high potential to mitigate ASR expansion. However, an optimum replacement level has to be established for a specific aggregate since its potential is highly dependent on the nature and type of aggregates.

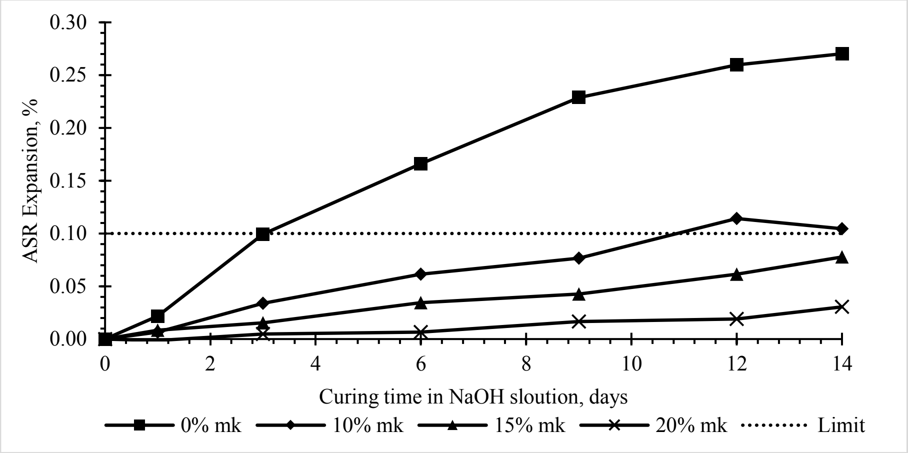

Figure 11: ASR expansion of mortar bars with metakaolin

4. Conclusion

Metakaolin that is locally available in the Western Cape has positive influences on enhancing properties of concrete over a range of mix proportions and replacement ratios;

1. It improves the compressive strength of concrete, with compressive strength increasing with increase in metakaolin content. The 28 day strength of the control was increased by approximately 40% by substituting 20% of cement with metakaolin.
2. It enhances tensile splitting strength of concrete, again with strength increase with increase in metakaolin content. The higher rate of strength increase is more observed at the early age, with an approximate 40% increase in tensile splitting strength of control at 20% metakaolin replacement rate.
3. It increases the static elastic modulus of concrete, the trend being independent of the replacement level.
4. The influence of metakaolin on reducing penetrability of concrete is excellent. Metakaolin decreases gas permeability and sorptivity of concrete with increase in its content. It also increases the resistance to chloride ion transport, with higher resistance being achieved at higher metakaolin contents.
5. Metakaolin shows a high potential in suppressing ASR expansion due to greywacke aggregate. Its potential is proven even at the lowest replacement level, however, increased content results in very low expansions.

By comparing metakaolin and GGCS, metakaolin generally exhibits better concrete properties than GGCS. Therefore, metakaolin can be used as a substitute for GGCS in Western Cape.

This study adds to the body of knowledge of metakaolin used as an SCM in comparison to GGCS. This leads to awareness on its potential in the Western Cape. Consequently, its utilization can assist in addressing possible scarcity of SCMs. However, questions that remain include cost-effectiveness (in terms of its production), and the awareness and willingness of industry to incorporate the material.

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