PROPERTIES OF METAKAOLIN CONCRETE – A REVIEW

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Abstract

The use of cement supplementary materials in structural concrete is widely accepted by the construction industry for technical, economical and environmental reasons. Metakaolin (MK), produced by calcining kaolinite at high temperature is suitable for concrete production due to its pozzolanic property. This paper reviews the some of the research published on effects of using MK on engineering properties of structural concrete as a cement replacement material. The review shows that the use of relatively finer MK to partially replace cement reduces the consistency of concrete and enhanced the strengths, deformational and durability properties of concrete. MK is most effective in enhancing compressive strength (particularly at early ages) compared to other strengths and modulus of elasticity was least improved. Drying shrinkage and creep of MK concretes are lower than those for the control concrete. The high pozzolanic reactivity of MK with calcium hydroxide contributes to both porosity reduction and pore-structure refinement in the pastes and concrete. As the consequence, the durability of concrete is improved through increased resistance to chloride penetration and controlled expansion, due to alkali-silica reaction and sodium sulphate attack.

1. INTRODUCTION

Minimising environmental impact and energy requirement is becoming increasingly important for sustainable concrete production, in addition to achieving reductions in the use of natural materials and greenhouse gas emission. The use of supplementary cementitious materials (SCM) in concrete mixes not only reduces the cement content but also increases the use of the industrial waste materials in an effective way. Fly ash, ground granulated blast furnace slag, silica fume, rice husk ash and metakaolin (MK) are pozzolanic materials which are used as effective cement replacement materials in concrete mixes.

Clay minerals are known to produce amorphous mineralogy prior to recrystallization at high temperature [1, 2]. The calcination of high-purity kaolinite at temperatures (700 to 850^{o}C) produces metakaolin (MK) which consist of silica and alumina in an active form which reacts with calcium hydroxide (CH) at room temperature. The use of MK as a partial replacement to cement in concrete is increasing due to its high pozzolanic reactivity. This paper reviews the some of the published research on the engineering properties of concrete containing metakaolin as a cement replacement material with increasing amount.

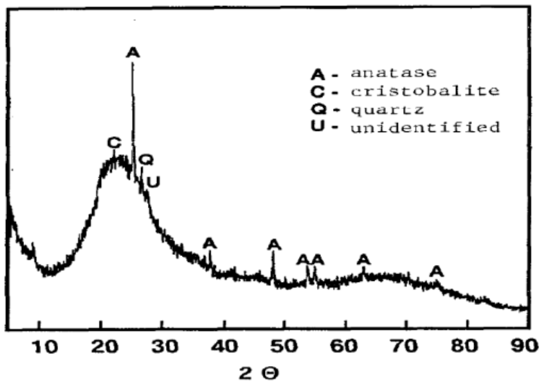

Figure 1: X-ray spectrum of metakaolin (adapted from [1])

2. PRODUCTION AND PROPERTIES OF METAKAOLIN

Thermal activation of kaolinite at 700 to 850°C by dehydroxylation causes partial or complete breakdown of crystal lattice structure forming a transition phase amorphous structure with high pozzolanic reactivity [2, 3]. Carefully controlled production process refines it colour by removing the impurities and tailors the particle size. High purity whitish powder of MK produced has the following (typical) physical properties: specific surface area of 16.8m^2/g; median particle size of 1.3µm which is smaller than that of cement (appro. 10µm) and larger than that of silica fume (appro. 0.1µm) and the specific gravity of 2.50 compared to 3.15 for cement.

Typical chemical compositions of MK are shown in Table 1. The combined amount reactive silica and alumina in MK is over 90% and the major chemical compound is $Al_2O_3.2SiO_2$ (or AS_2). MK is mainly an amorphous material with a small quantity of crystallized phases as shown in Figure 1.

Table 1: Typical chemical composition of metakaolin (adapted from [2])

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	L.O.I
% by mass	51.5	40.2	1.23	2.0	0.12	0.08	0.53	2.27	2.01

3. POZZOLANIC REACTIVITY OF METAKAOLIN

Pozzolanic reaction of AS_2 with CH liberated from cement hydration produces: (i) C-S-H gel; (ii) crystalline calcium aluminate hydrates(C_4AH_{13} and C_3AH_6); and (iii) crystalline calcium alumino-silicate (C_2ASH_8). The crystalline products depend principally on the AS_2/CH ratio and calcining temperature [3-5]. Murat [3] suggested the following competitive equations for pozzolanic reaction of MK:

$$AS_2 + 6CH + 9H = C_4AH_{13} + 2C\text{-}S\text{-}H \quad (1)$$

$$AS_2 + 5CH + 3H = C_3AH_6 + 2C\text{-}S\text{-}H \quad (2)$$

$$AS_2 + 3CH + 6H = C_2ASH_8 + C\text{-}S\text{-}H \quad (3)$$

Ambroise et. al. [2] observed the following changes during the hydration of cement-metakaolin blended paste: (i) calcium hydroxide was quickly consumed; (ii) microstructure became rich with C-S-H gel and crystalline $CASH_8$; and (iii) pore sizes were reduced with pore structure refinement. MK acted as an accelerating admixture when cement was replaced with MK up to 30%, by weight. XRD studies by Kim et. al. [6] showed that pozzolanic reactivity of MK reduced the CH content depending on the MK content, while the weak peaks of C–A–H was slightly increased. Metakaolin is superior to silica fume in relation to strength enhancement of concrete at early ages due to the increased cement hydration in the presence of metakaolin [7]. Taylor-Lange et. al. [8] observed increased reactivity of metakaolin-cement systems during first 24 hours with the addition of zinc oxide.

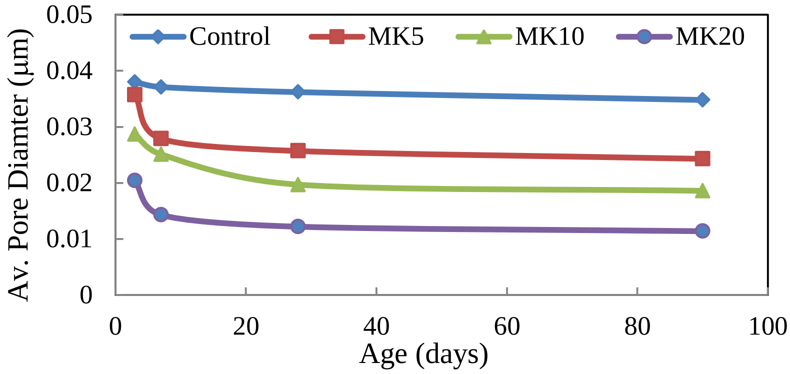

Figure 2: Average pore diameter of blended cement pastes (adapted from [7])

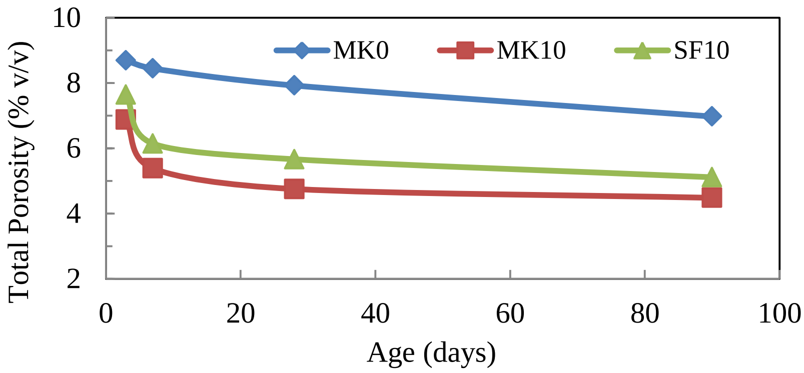

Figure 3: Total porosity of high performance concrete (adopted from [9])

4. POROSITY AND PORE SIZE DISTRIBUTION IN PASTES AND CONCRETE

Khatib and Wild [10] studied the porosity and pore size distribution of hydrating of pastes with up to 15% cement replacement with MK, at a w/b ratio of 0.55. Their reported that MK produced pore structure refinement in pastes with an increase in the proportion of pores, below 20µm in radius. The volume of pores, below 20µm in radius, in an year old paste with 15% MK was 55% compared to 35% for the cement paste (0% MK), confirming pore size refinement.

Poon et. al. [7] observed significant drop in the average pore diameter with the increase in MK as the cement replacement material and the results are shown in Figure 2. For 90-day old blended paste with 20% MK, the average pore diameter was 0.0114μm compared to 0.0348μm for the cement paste at the same age. At the same time, the total porosity was 9.21% for blended paste compared to 14.0% for cement paste.

Poon et. al [9] reported the variation of total porosity with age for high performance concretes (Figure 3), having the water to binder ratio of 0.30 and 10% cement replacement with either metakaolin or silica fume. The total porosity of concrete has dropped with increasing age due the combined effects of cement hydration and pozzolanic reaction. MK is found to be superior to silica fume in reducing the total porosity.

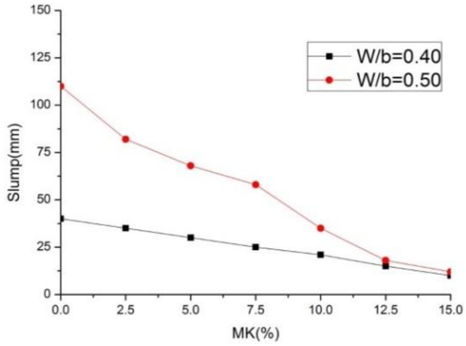

Figure 4: Effect of MK content on the consistency of concretes (adapted from [11])

5. CONSISTENCY AND SEETING OF METAKAOLIN CONCRETE

Bai et. al [11] and Johari [12] reported that the slump of concrete with given water content was significantly reduced with the increase of MK content. Figure 4 shows that the high slump concrete experienced a relatively rapid loss in its consistency with the increase of MK content compared to low slump concrete. The reduction in consistency may be attributed to the increase in the surface area of the binder materials, considering the high fineness and low density of MK compared to those for cement. Although MK concretes exhibited reduced slump, Bai et. al. [11] experienced that full compaction of MK concretes was achieved due to the ability of MK to impart some degree of thixotrophy to concrete.

Brooks et. al. [13] reported that cement replacement with MK had increased the setting times of high strength concrete with high dosage of superplasticiser. Their results showed that the initial and final setting times in concrete with 15% MK were increased by 29% and 21%, respectively. The difference between final and initial setting times was increased marginally from 2.70h to 2.86h. However, Ambroise et. al. [2] reported the reduction in setting time of paste with MK and concluded that MK performed similar to an accelerating admixture. Therefore, it is possible that the superplasticiser has the capability of nullified the accelerating effect of MK in high strength concrete.

6. MECHANICAL PROPERTIES OF METAKAOLIN CONCRETE

6.1 Compressive strength

Pozzolanic material in concrete contributes to the strength improvement in concrete mainly in three main ways: (i) filler effect (immediate); (ii) accelerating effect (within 24h), and (iii) pozzolanic effect (time dependent). Quin and Li [14] studied the mechanical properties of concrete with increasing MK content at a w/b ratio of 0.38. Table 2 summarises the results and Figure 5 compares the hardened concrete properties of MK concrete at 28 days. The compressive, tensile and flexural strengths and modulus of elasticity of concrete are found to increase with the increase in the MK content at all ages.

Table 2: Effect of metakaolin content on tensile and flexural strength of concrete [14]

Age (days)	Property	Control	Cement replacement with MK		
			5%	10%	15%
3	Compressive Strength	27.9 MPa	1.29	1.40	1.51
28		37.8 MPa	1.21	1.68	1.84
60		58.0 MPa	1.08	1.15	1.34
28	Tensile Strength	3.35 MPa	1.07	1.16	1.28
28	Flexural Strength	4.65 MPa	1.02	1.32	1.38
80		5.70 MPa	1.02	1.13	1.24
3	Modulus of Elasticity	24.1 GPa	1.06	1.05	1.09
28		30.0 GPa	1.08	1.11	1.13
60		30.4 GPa	1.09	1.11	1.14

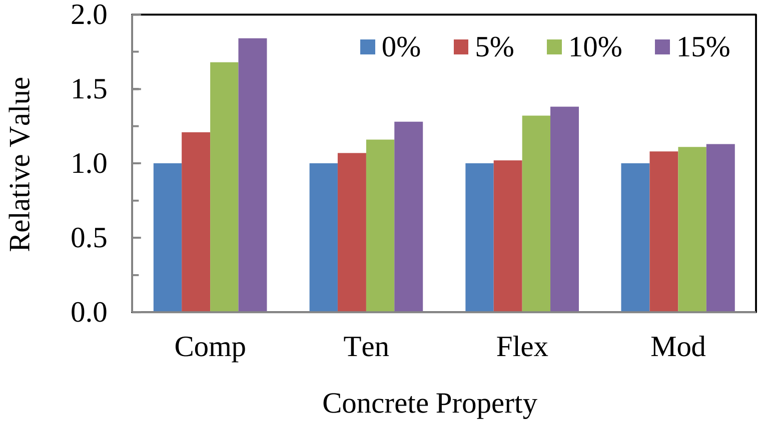

Figure 5: Effectiveness of MK on 28-day properties of concrete (adapted from [14])

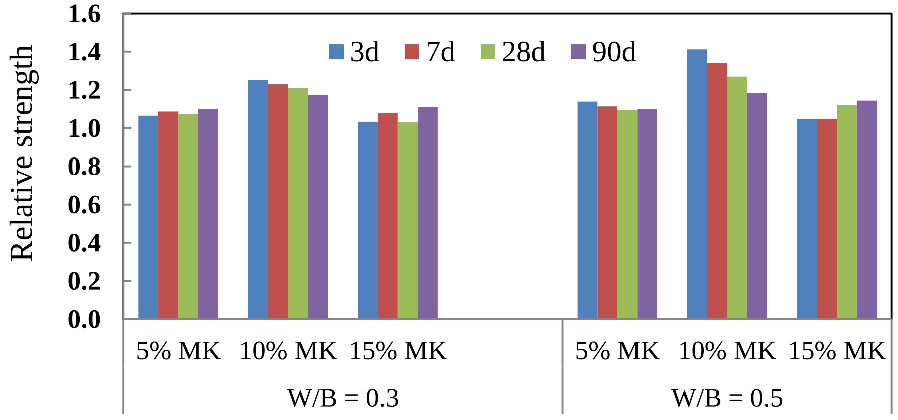

Figure 6: Effectiveness of MK in concrete - function of age and w/b ratio (adapted from [9])

The results show that compressive strength is the most enhanced property of concrete while the modulus of elasticity was least improved. With 15% MK, the compressive strength was increased by 84% and 34% at 28 and 90 days, respectively. Therefore, the effectiveness of MK in improving the compressive strength is reduced with the increase of age of concrete. The early age strength increases could be due to the aluminium content (45%) and finer particle size (3.5μm) of MK, which accelerates the hydration reaction and packs into cement particles gaps. In the other hand, the long term strength of concrete is increased through pozzolanic effect.

Poon et. al. [9] studied the effectiveness of MK content up to 15% on the compressive strength of concrete over 90 days at the w/b ratios of 0.30 and 0.50. The results shown in Figure 6 indicate that the effectiveness of MK was optimum at 10% MK content and increased with the increase in the w/b ratio. Guneyisi et. al. [15] observed the compressive strength improvements of 44% and 29% at 28 days for 15% MK concrete at the w/b ratios of 0.35 and 0.25, respectively. Zhang and Malhotra [1] and Jin and Li [16] were reported similar results.

6.2 Tensile and flexural strengths

Qian and Li [14] observed that MK is less effective in improving the tensile and flexural strengths compared to compressive strength (Figure 5). 5% MK had 7% and 2% improvement on the tensile and flexural strengths of concrete at 28 days (Table 2). At 10% MK, the tensile and flexural strengths were increased by 16% and 32% and further improvements of 28% and 38% were recorded at 15% MK. The results also showed that with the increase of age from 28 to 80 days, the flexural strength improvement had dropped from 32% to 13%.

6.3 Modulus of elasticity, shrinkage and creep

The modulus of elasticity of concrete at 28 days had moderate improvements of 11% and 13% at 10 and 15% MK (Table 2 and Figure 5). Unlike compressive and tensile strengths, effectiveness of MK had not dropped with the increase in age.

Table 3: Shrinkage and creep of metakaolin concrete (adapted from [17])

Concrete mix	200d auto. shrinkage (10^{-6})	200d drying shrinkage (10^{-6})	Creep (10^{-6})		Specific creep (10^{-6} / MPa)		
			Total	Basic	Dry	Basic	Drying
MK0	445	416	358	285	25.8	20.5	5.3
MK5	485	228	312	235	18.5	14.0	4.5
MK10	419	199	201	126	11.3	7.2	4.1
MK15	327	189	171	115	9.5	6.4	3.1

Brooks and Johari [17] investigated the effectiveness of MK on autogenous shrinkage, drying shrinkage and creep of high strength concrete. The results summarised in Table 3 show that early age autogenous shrinkage of concrete was reduced with the increase of cement replacement level with MK. The autogenous shrinkage after 24h for 15% MK concrete was 35% of that for the control concrete due to the reduction in the cement content. A rapid increase in autogenous shrinkage for MK concretes was noted when the autogenous shrinkage was measured from 24h to 14 days. This could be attributed to the combined effect of acceleration of cement hydration and the pozzolanic reaction. From 14 days to 4 months, a relatively small increase in autogenous shrinkage was recorded. For 10% and 15% MK concretes, 200-day autogenous shrinkage was reduced by 6% and 27% compared to the control concrete.

200-day drying shrinkage of concrete had dropped from 416 microstrain (for control concrete) to 228, 199 and 189 microstrains for concretes with to 5%, 10% and 15% MK content, respectively. This could be due to the reduced moisture loss from MK concrete.

Table 3 also shows that both total and basic creep of concrete was decreased with the increase of MK content. This could be due to a combination of the following in MK concretes: (i) denser pore structure; (ii) stronger paste matrix; and (iii) improved paste aggregate interface.

7. DURABILITY PROPERTIES OF METAKAOLIN CONCRETE

7.1 Carbonation

Kim et al. [6] conducted accelerated carbonation test (5% CO_2, 60% RH and 30^oC) on high-strength concretes having increasing MK content. The carbonation depth of concrete was found to increase with the increase in MK content. The carbonation depth after 56 days for 20% MK concrete was 14mm compared to 4mm for the control concrete (0% MK). Kim et. al. [6[suggested that the reduced amount of portlandite in hydrate products is probably responsible for the increased carbonation in MK concretes. In dense and compacted concrete, the carbonation initially produces calcite having 11% to 12% higher volume than CH, resulting in reduced total porosity and permeability [18]. In general, concrete with a low hydration rate will carbonate faster than that with high hydration rate, when exposed to the environment at an early stage.

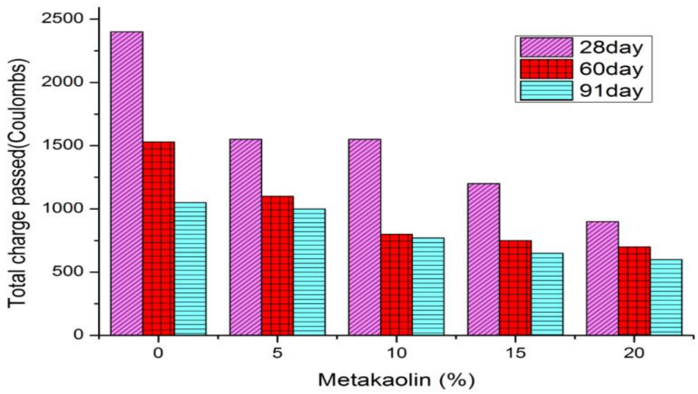

Figure 7: Chloride permeability of MK concretes (adapted from [6])

7.2. Chloride diffusion

Kim et. al. [6] studied the chloride diffusion in high-strength MK concretes up to 91 days, using rapid chloride permeability test. The results, shown in Figure 7, indicate that the chloride permeability in concrete was decreased with the increase of age and MK content. The decrease in the porosity and refinement in pore size, due to cement hydration and pozzolanic reaction, contribute to the reduction in the permeability in concrete.

Poon et. al. [9] reported similar results as shown in Figure 8 for control concrete and 10% MK concrete at the w/b ratios of 0.30 and 0.50. The best performance for MK concrete was noted with 10% and 20% MK content at the w/b ratios of 0.30 and 0.50, respectively. This indicates the increased MK content to decrease the permeability of MK concrete having high initial porosity (or w/b ratio).

Bai et. al. [19] observed significant reductions in chloride penetration depth in fly ash concrete, exposed to sea water, when fly ash was partially replaced with MK. The results showed that the resistance to chloride penetration was increased with the increase of both MK content and curing time. This was attributed to the relative changes in intrinsic diffusivity and chloride binding capacity with age exhibited by the different binder compositions.

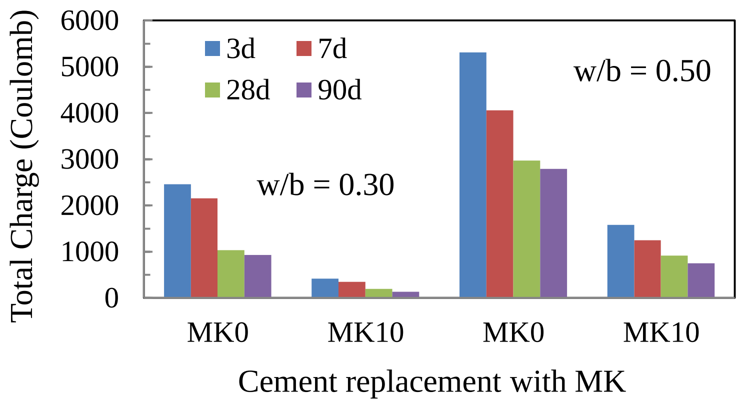

Figure 8: Chloride permeability of control and MK concretes (adapted from [9])

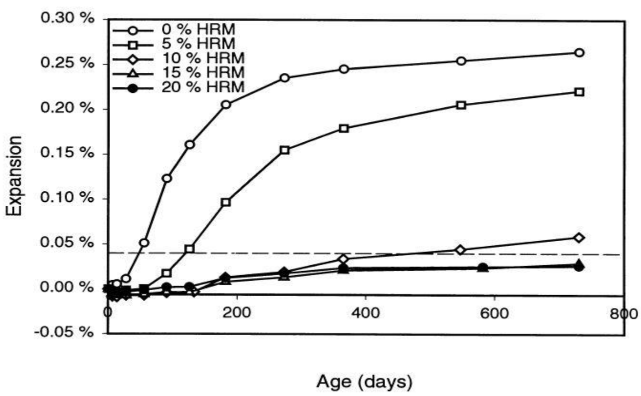

Figure 9: Alkai-silica expansion in MK concrete prisms (adapted from [20])

7.3. Alkali–Silica resistance (ASR)

Ramlochan et. al. [20] studied the efficacy of MK in controlling expansion due to alkali-silica reaction with a reactive aggregate by using concrete prism method and the results are shown in Figure 9. MK as a partial cement replacement between 10% and 15% was found to control deleterious expansion below 0.04 after 2 years, depending on aggregate type. MK appears to control the ASR expansion through entrapment of alkalis by the supplementary hydrates and a consequent decrease in the pH of pore solutions.

Walters et. al. [21] determined the effectiveness of MK in suppressing ASR in concrete using mixtures containing inert limestone aggregate and chert/flint reactive sand. The results showed that control concrete (0% MK) exhibited an expansion of 0.45% after 6 to 9 months while the concrete with 10% to 15% MK had reduced the expansion to less than 0.01% during the same period. The cracks and surface deterioration, which were found in control concrete, were virtually eliminated in MK concretes. Kostuch et. al. [22] reported 15% MK had completely eliminated ASR expansion.

The expansive product in control concrete was "high Ca^{2+} gel" containing low levels of Na^+ and K^+ and the amount formed was dependant on the CH availability relative to active silica content. MK reduces CH content and CH/SiO_2 (active) ratio which consequently preventing the formation of swelling gel and reducing the ASR expansion.

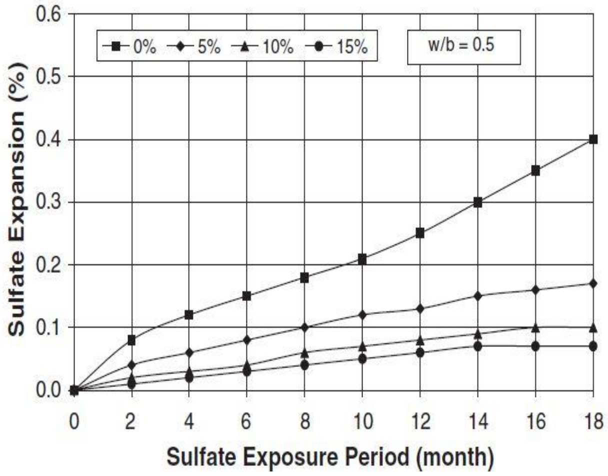

Figure 10: Sulfate expansion of MK concretes (adapted from [23])

7.4. Sulfate resistance

Al-Akhras [23] investigated the behaviour of MK concretes (w/b of 0.50), with increasing MK content up to 15%, exposed to sodium sulfate solution over 18 months. The sulphate attack was assessed through by a number of ways: (i) expansion measurements in concrete prisms; (ii) compressive cube strength; and (iii) visual inspection of concrete prisms. Figure 10 shows the sulfate expansion in concrete prisms as a function of age and MK content. The sulfate resistance of concrete was increase with the increase of cement replacement with MK. Control concrete experienced the maximum expansion of 0.4% in 18 months while MK concretes with 10% and 15% MK showed the expansion of 0.10% and 0.07% after the same exposure period. The improved sulfate resistance of MK concrete could be due to the following reasons: (i) cement replacement reduces the total amount of tricalcium aluminate hydrate in the hardened paste; (ii) pozzolanic reactivity of MK partially consumes calcium hydroxide released by cement hydration which controls the formation of expansive gypsum; and (iii) permeability of concrete is reduced through pore structure refinement in MK concrete. Lee et. al. [24] investigated the sulfate resistance of MK mortars, incorporating 5, 10, and 15% MK as partial replacement of cement exposed to magnesium sulfate solution. The degree of deterioration of MK mortar was found to be more severe with an increase of the solution concentration of magnesium sulfate due to increased expansive gypsum formation.

7.5. Freeze –Thaw resistance

Kim et. al. [6] studied the effectiveness of MK and silica fume (SF) on the freezing–thawing characteristics of air-entrained (3% air content) high-strength (w/b of 0.25) concretes. Tests were conducted on five concrete mixes, control concrete and concretes with 5% and 10% of MK or SF as cement replacements. The relative dynamic modulus of elasticity was found to remain quasi-constant for all concretes until 300 cycles due to low w/b ratio and the air entrainment. Both MK and SF concretes showed comparable freeze-thaw resistance similar to the control concrete.

8. CONCLUSIONS

The following conclusions are made from the review of the properties and performance of concrete containing metakaolin as partial replacement to cement use of metakaolin.

- Consistency of concrete was decreased with the increase in MK content without affecting the compaction of concrete.
- MK acts as an accelerating admixture in promoting cement hydration in MK concrete.
- Relatively finer and highly pozzolanic metakaolin as a partial replacement of cement produces pore structure modification, reduces porosity and pore size refinement in the hardening pastes and concretes.
- Metakaolin contributes through filler effect and acts as an accelerating admixture to enhance cement hydration at early ages. At later ages, through pozzolanic reactivity MK contributes to the improvements in the properties of hardened concrete.
- Partial cement replacement with metakaolin significantly increases the compressive strength and the degree of strength enhancement increase with the increase of MK content and w/b ratio. However, it was general found to reduce with the increase of age.
- Tensile and flexural strengths of concrete are less improved with the increase of MK content compared to compressive strength and modulus of elasticity was the least improved.
- Drying shrinkage and creep are reduced significantly with the increase in MK content.
- While chloride permeability, resistance to ASR and sulfate attack is reduced in MK concrete, the carbonation depth was increased. 10% to 15% cement replacement with MK is sufficient to control ASR related expansion and sulfate attack.

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