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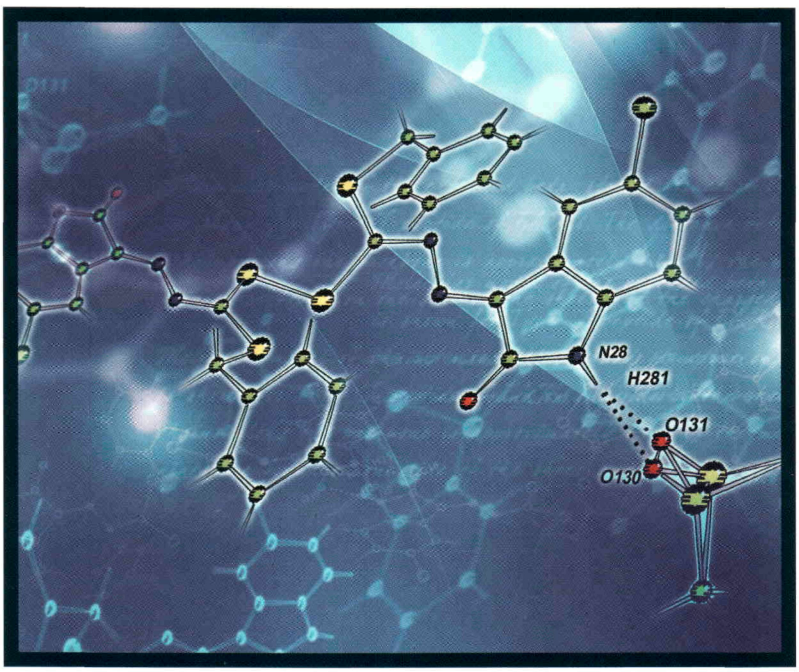

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Influence of Metakaolin as Partially Cement Replacement Minerals on the Properties of Cement and Concrete

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ABSTRACT

Metakaolin is a manufactured pozzolan produced by thermal processing of purified kaolinitic clay using electrical furnace. This study has examined the effect of Metakaolin on the properties of cement and concrete at a replacement level of 0%, 5%, 10% and 15%. The parameters studied were divided into two groups which are chemical compositions, water requirement, setting time and soundness test were carried out for cementitous properties. Workability, compressive strength and bending strength were test for concrete properties. Hardened concrete was cured under different type of curing conditions and tested. The result showed that the inclusions of Metakaolin as cement replacement minerals have change some of the cementitous and concrete properties. This research reveals, the optimum effect for cementitous and concrete properties for metakaolin was 10%.

***Keywords**: Metakaolin, cement paste, concrete, workability, setting times, soundness, strength*

INTRODUCTION

Pozzolanic materials such as silica fume, fly ash and ground granulated blast-furnace slag is known to improve the engineering and performance properties of concrete when they are used as a mineral additive or as partial cement replacement [1, 2]. In the recent years, there has been increased interest in the use of MK as a mineral admixture. Metakaolin (MK) is a thermally activated aluminosilicate material with high pozzolanic activity comparable to or exceeded the activity of silica fume (SF) [3]. MK is produced by calcining high purity kaolin clay at temperature of 600-800°C. Unlike other mineral admixtures that are mostly by-product pozzolans, which can have variable composition, purity and reactivity, the production of MK can be closely controlled and thus, a higher degree of purity and pozzolanic reactivity can be obtained [4, 5]. MK was reported to be useful in improving the concrete quality such as enhancing strength, decreasing autogenous shrinkage, reducing risk of chloride induced corrosion of embedded steel, controlling hydrate transformation of high alumina cement, and improving the durability of concrete [6].

Malaysia has an abundance of kaolin clay which is used in some industry such as ceramic industry, rubber product industry, cosmetic, paint etc. Used of kaolin clay as mineral admixture or mineral replacement materials in concrete industry was limited. Earlier research has shown Malaysian metakaolin named as MK7003 produced from locally available kaolin has the potential to improve the properties and performance of concrete. However, more research works are required to prove that this material could really improve the properties and performance of concrete. In addition, not much information is available on the effect of curing conditions on concrete containing MK7003.

EXPERIMENTAL METHOD

Production of Metakaolin

Metakaolin was produced by thermal processing of purified kaolinitic clay using electrical furnace at heating rate of 20°C per minute up to 700°C and maintained at this temperature for 3 hours and than allowed to cool down to room temperature. The kaolinitic clay was obtained from AKI Association which is based in Tapah, Perak.

Cementitous Properties

The chemical composition analysis on the prepared Metakaolin was carried out using XRF analysis apparatus in Universiti Sains Malaysia (USM). The properties of cement paste on water requirement, initial setting time, final setting time and soundness test were carried out using Vicat apparatus and Le-Chatelier apparatus respectively. The cement paste containing OPC and Metakaolin at a replacement level of 0%,5%, 10% and 15% from mass to mass basis named as OPC, MK5,MK10 and MK15 respectively.

Concrete Properties

In order to study the effect of Metakaolin as partial cement replacement mineral in concrete in a different curing conditions, the prepared Metakaolin was blended with an OPC at 0%,5%,10% and 15% replacement level. Ordinary Portland cement (OPC) used was produced by Kedah Cement Malaysia Bhd. The replacement was done on mass to mass basis. The concrete mixes are known as M1, M2, M3, and M4 respectively to the replacement level. Locally available Natural River sand (53 % passing 600μm sieve) and crushed blue granite stone aggregates (passing 14 mm sieve) were used as fine and coarse aggregate, respectively. The materials were proportioned so that the concrete mixes will achieve 28 days strength of greater than 40 MPa. A total binder content of 532 kg/m^3 and a constant water/binder ratio 0.47 were used. A superplasticizer known as Reobuilt 1100 was used at a dosage rate of 0.75% to maintain the same workability range (60 to 180mm). Details of the concrete mix proportions are shown in Table 1.0. A concrete mix without any cement replacement materials was prepared as a control mix. All the concrete materials were mixed in the concrete mixer for 3 minutes and were tested for slump to determine the effect of Metakaolin on the workability of concrete. The concrete mixes were than cast into steel cube moulds (100 x 100 x 100 mm) in three layers and each layer was compacted using a vibrating table. The total 144 concrete cubes were prepared and all the cubes have been cured in 3 different curing condition named as normal curing, air curing and high temperature curing. Normal curing is referring to a curing under water at controlled temperature in the range of $20 \pm 2°C$. Air curing is referring to a curing in a room temperature from 27 - 28° C. For high temperature curing the specimens where kept in elevated curing tank and the temperature is controlled at 55°C. All prepared specimens will be cure until a date of testing which is 1, 3, 7, 28, 84 and 168 days. At all testing ages, three cubes were tested for compressive strength. The total 48 concrete beam of 100 mm x 100mm x 500mm were also cast and tested for flexure strength on 28 and

168 days. All tested was carried out in accordance with British Standard specifications for concrete.

Table 1: Concrete Mix Proportions

Concrete Mixes	OPC (kg/m^3)	Metakaolin (kg/m^3)	Sand (kg/m^3)	Coarse Agg(kg/m3)	Water (l/m^3)
M1	532	-	658	909	250
M2	505.4	26.6	658	909	250
M3	478.8	53.2	658	909	250
M4	452.2	79.8	658	909	250

RESULT AND DISCUSSION

Chemical Composition

Table 2: Chemical Composition of Metakaolin and OPC

Composition	OPC (%)	Metakaolin (%)	PC:MK
SiO_2	20.0	55	26
Al_2O_3	5.70	38	13
Fe_2O_3	2.90	0.65	2.3
CaO	63.0	0.072	49
MgO	0.99	0.9	0.97
K_2O	1.2	2.5	1.3
SO_3	3.50	0.039	2.7
TiO_2	0.25	0.73	0.33
NiO	Trace	NIL	Trace
LOI	2.8	2.1	3.9

Table 2 shows a chemical composition of Metakaolin compared to OPC. Metakaolin contained 55% of SiO2 which is almost 60% more than OPC. Table 2.0 also shows that Metakaolin also containing 38% Al_2O_3 which is 85% more than OPC. In term of CaO content, Metakaolin containing less than 1% compared to OPC which contained about 63%. The inclusion of Metakaolin as partial cement replacement minerals at a mix proportion of 90:10 (PC:MK) shown in Table 2.0 have increase the content of SiO_2 and Al_2O_3 in OPC and in the same time reduced the content of CaO, Fe_2O and MgO of OPC. This shows that the chemical compositions of major component in cementitous systems for a hydration process have been balance up and the amount of $Ca(OH)_2$ will be reduce since the CaO will be fully utilised during the hydration process.

CEMENTITOUS PROPERTIES

Effects of MK on Water Demand

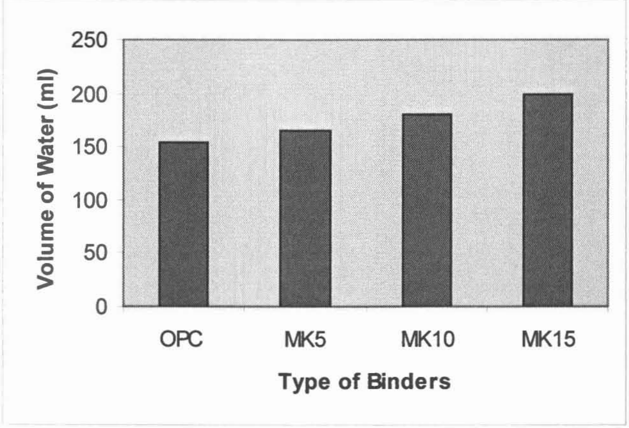

Figure 1: Water Requirement of the Different Binder Pastes to Give Standard Consistency

The effect of MK on the water demand of binder pastes and workability of concretes is shown in Figures 1. From Figure 1, it is obvious that the inclusion of MK increases the water demand of the binder pastes to obtain a standard consistency, with greater increases at higher MK replacement levels. This could be attributed to the greater fineness of the MK in comparison to OPC, hence, the higher the MK replacement level, the greater the water demand. Similar trend was observed by Brooks *et al.* [7] and Bai *et al.* [1].

Effects of MK on Setting Time

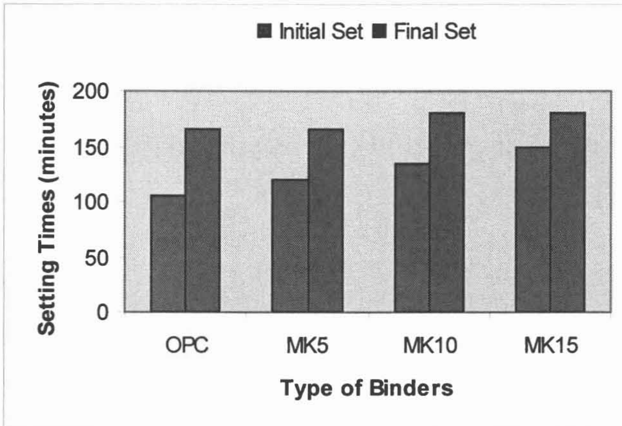

Figure 2: Setting Times of the Different Binder Pastes

Figure 2 shows the effect of MK inclusion on the setting times of the binder pastes measured using the Vicat apparatus on binder pastes with standard consistency. It is clear that the effect of MK is to increase the setting times of the binder pastes in particular the initial setting time. The observed retardation in setting times could be mainly attributed to the combined effect of a lower cement content and higher water/binder ratio for the binder pastes containing MK, since part of the cement was replaced by MK. Previously, it has been demonstrated (Figure 1) that for the binder pastes containing MK, the amount of water required to achieve standard consistency should be increased. Hence, these binder pastes should have higher water/binder ratio than the OPC control paste. Water/binder ratio has been recognized as one of the important factors that could influence the setting characteristics of cement and concrete [7, 8]. By taking a closer look at the result in Figure 2, it is clear that the final setting time is only marginally retarded in comparison to the initial set. For the initial set, the retardation ranges from 15 minutes to 45 minutes for MK5 and MK10, respectively. On the other hand, for the final set, it ranges from no retarding effect to 15 minutes for MK5 and MK15, respectively. Therefore, the retarding effect is only a short-term phenomenon and should not cause any undesirable delay in construction process.

Effects of MK on Soundness

The soundness of the binder pastes was assessed using the Le Chatelier apparatus and the results are given in Table 3.0. It is clear that the inclusion of MK tend to reduce the expansion of the binder pastes. The code of practice (BS 1881) put a limit on the potential expansion of neat cement paste. This is done to reduce any potential expansive reaction in concrete at later ages which could disrupt and damage the concrete. Hence, the use of MK is beneficial as it could reduce the potential future expansive reaction in concrete.

Table 3: Soundness of the Binder Pastes Measured Using the Le Chatelier Apparatus

	OPC		MK5		MK10		MK15	
Sample	A	B	A	B	A	B	A	B
Initial Reading (mm)	7	6.5	7	6.5	6	7	6	6.5
Reading After Boiling (mm)	8.5	8.5	8	7.5	7	8	7	7.5
Expansion (mm)	1.5	2	1	1	1	1	1	1

CONCRETE PROPERTIES

Effects of MK on Workability of Concrete

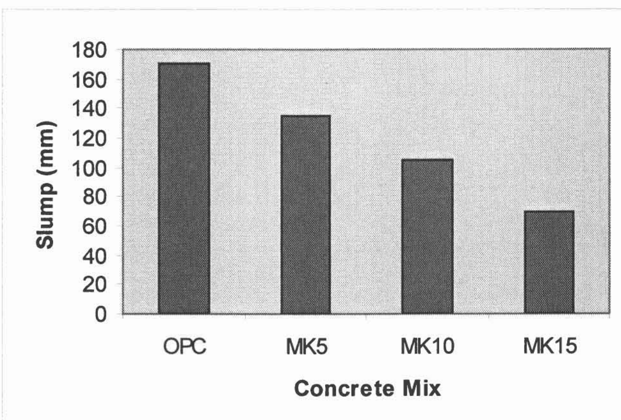

Figure 3: Slump Values of the Different Concrete Mixes

In the case of workability, a Figure 3 show that the effect of Metakaolin is to reduce the workability of the concrete mixes measured using the slump test, with greater reduction at higher replacement levels. The slump test results exhibited in Figure 3 were obtained from concrete mixes containing a constant dose of superplasticiser. This was done so that all concrete mixes achieve a target slump in the range of 60 – 180 mm to enable proper compaction of the concrete. Therefore, the previously observed effect of Metakaolin on water demand supports the later findings on workability. Due to the greater water demand of the Metakaolin, the workability reduces in particular at higher replacement levels. As been reported by Sabir *et al* [1], slump characteristics will be reduce at higher level replacement of metakaolin. This was supported by Megat Azmi *et al* [9].

Effects of MK on Compressive Strength

The general effect of Metakaolin is to improve the compressive strength of the concrete. The results as depicted in Figures 4, 5 and 6 shows that the use of Metakaolin as partial cement replacement enhances the compressive strength of the concrete in particular the strength at early ages. This could be attributed to the filler effect, acceleration in OPC hydration and pozzolanic reaction of Metakaolin. The fine particles of Metakaolin act as fillers, occupying the void spaces between the cement particles and aggregates. These enhance the quality of the binder matrix to become much denser as well as improve the quality of the transition zone between the aggregates and the binder matrix. According to Sabir *et al.* [8], the use of Metakaolin could also accelerate the rate of hydration reaction of OPC. The fine particles of Metakaolin provide greater surface area of OPC hydration products. As a result, the early age strength of the concrete is enhanced [10].

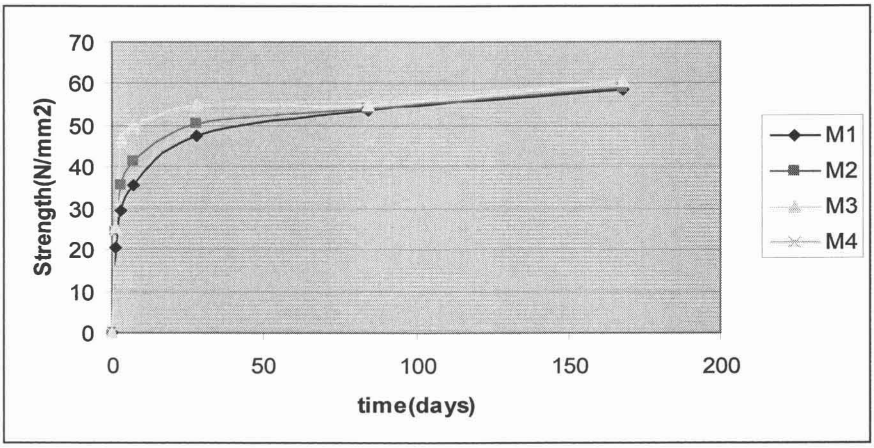

Figure 4: Effect of Metakaolin on Compressive Strength of Concrete Under Normal Curing

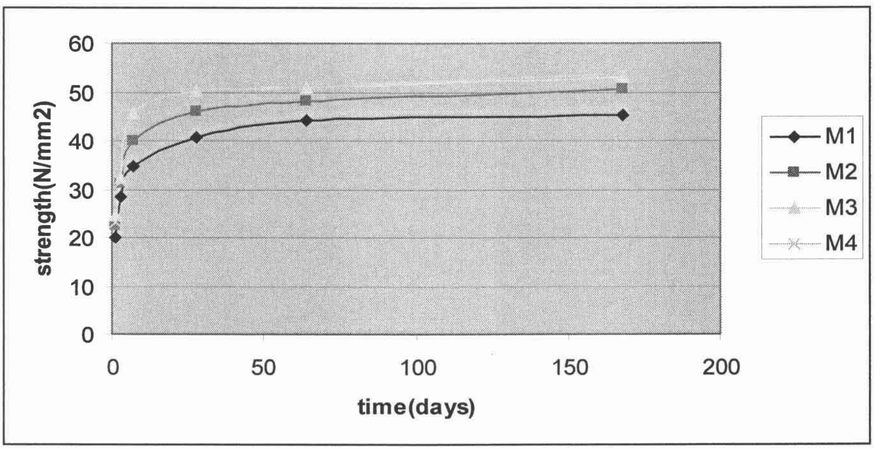

Figure 5: Effect of Metakaolin on Compressive Strength of Concrete Under Air Curing

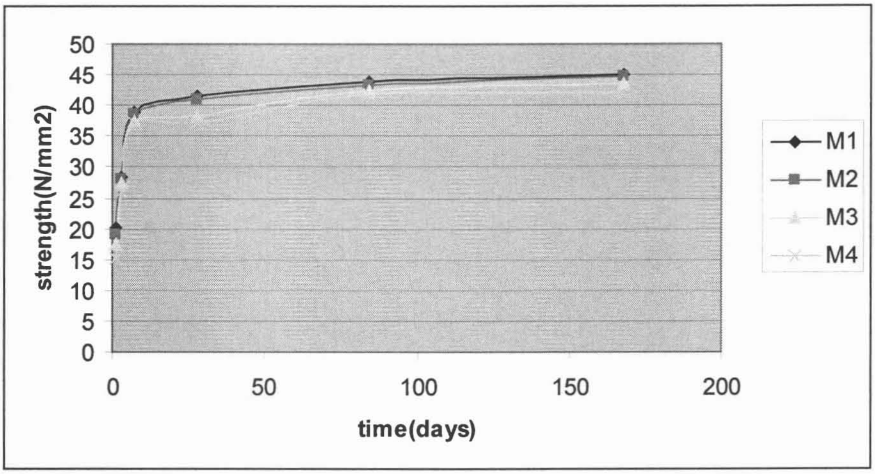

Figure 6: Effect of Metakaolin on Compressive Strength of Concrete Under High Temperature Curing

The figures show the compressive strength development of the concrete mixes containing Metakaolin relative to that of the M1. It is clear that the one day strength of the Metakaolin concretes is about 20-23 % higher than the M1, with the M3 showing the highest strength. This is due to the filler effect and acceleration in OPC hydration discussed earlier. Between the age of 1 day and 3 days, there is a clear trend that the M3 and M4 concretes showing significant increase in strength in comparison to the M1, while the M2 only showing marginal increase in strength. This conspicuous trend could be attributed to the pozzolanic reaction of MK with $Ca(OH)_2$ liberated from OPC hydration reaction. The pozzolanic reaction results in refinement in the pore structure of the binder matrix of the concrete to become much denser, and hence increases the concrete strength. For the M2 concrete, probably there was insufficient MK available to induce significant pozzolanic reaction, and thus the strength was only marginally increased. Between 3 days and 180 days, there seems to be a reduction in relative strength of the MK concretes, but the actual long-term strength is still marginally higher than that of the OPC. The reduction in relative strength could be due to the depletion of MK as a result of the pozzolanic reaction. Previously, Wild and Khatib [2] found that for cement paste containing 15 % MK, there is still a substantial amount of $Ca(OH)_2$ available even after one year, indicating that higher replacement level is needed to fully consume the $Ca(OH)_2$ produced. At the same replacement levels of 5, 10 and 15 %, and using commercially available MK, Wild *et al.* [10] observed a maximum development in relative strength between the age of 7 and 14 days. This seems to suggest that the potential pozzolanic reactivity of the MK produced in this investigation is greater than that of the commercially available MK. The results as shown in Figures 4 and 5 also indicate that the optimum MK replacement level to provide maximum short-term and long-term strength is 10 %. Similar finding was obtained by Megat Johari *et al.* [9] in their study on high-strength concrete utilizing commercially available MK.

Figure 5 and 6 shows the inclusion effect of MK7003 as cement replacement minerals under different curing conditions. For specimens that have been cured under air (figure 5) and high temperature conditions (figure 6), the strength development of all the mixes have been reduced. Even the relative strength development of concrete for all mixes was effected by the curing conditions but the performance of concrete containing Metakaolin is still better than a mix using 100% OPC.

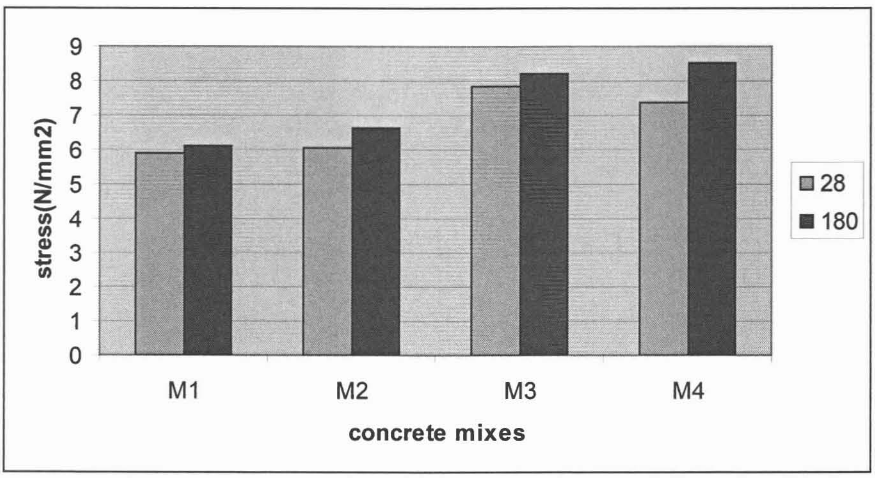

Figure 7: Effect of Metakaolin on Flexural Strength of Concrete Under Normal Curing

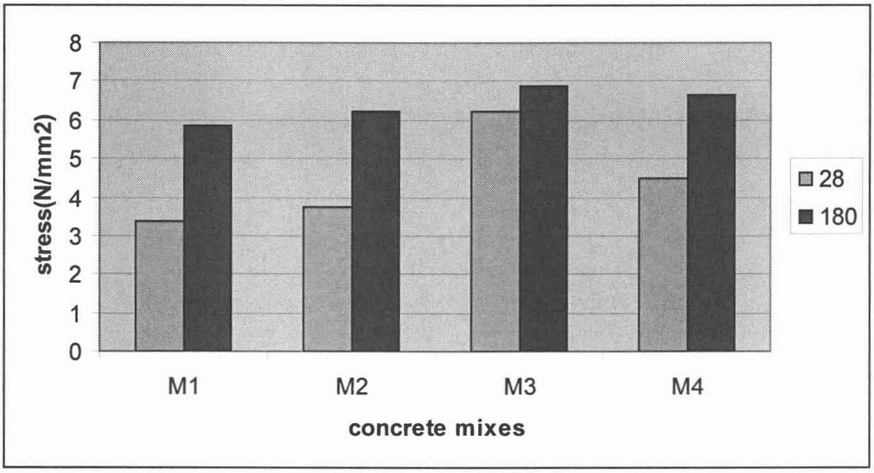

Figure 8: Effect of Metakaolin on Flexural Strength of Concrete Under Air Curing

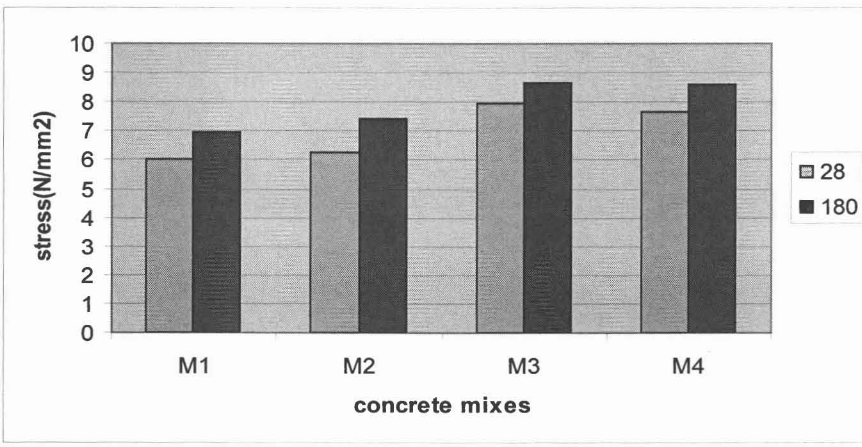

Figure 9: Effect of Metakaolin on Flexural Strength of Concrete Under High Temperature Curing

Effects of Metakaolin on Flexural Strength

From the results as shown in Figure 7, 8 and 9, it is obvious that the inclusion of Metakaolin enhanced the flexural strength of concrete as in the case of compressive strength even in different curing conditions. The flexural strength test was performed after 28 and 180 days of curing. Similar to compressive strength, the improvement in flexural strength as a result of

Metakaolin inclusion could be attributed to the filler effect, the accelerating effect on OPC hydration as well as the pozzolanic reaction. The M3 which contained 10% of Metakaolin recorded the maximum flexural strength at the age of 28 and 180 days, respectively. For the effect of curing condition on the concrete performance containing Metakaolin compare to OPC mix, the flexural strength of concrete containing Metakaolin is still batter than concrete using OPC in all curing conditions. The similar findings were obtained from Poon *et al* [3].

CONCLUSIONS

From the results presented in this paper, the following conclusions are offered:

1. The inclusion of MK increases the water demand of binder pastes to achieve standard consistency with greater effect at higher replacement levels.

2. The use of MK significantly reduces the workability of concrete in particular at higher replacement levels. Hence, superplasticiser must be used to obtain adequate workability.

3. The use of MK retards the setting times of binder pastes with the initial set being significantly delayed. The final set is only marginally retarded, hence the use of MK should not cause any delay in construction process.

4. The soundness test indicates that MK tends to reduce the potential expansion of binder pastes.

5. Both the compressive and flexural strength of concrete are enhanced with the use of MK. For compressive strength, the improvement is noticeable even from very early age. The optimum replacement level to give maximum short-term and long-term strength is 10 %.

6. The performance of concrete containing MK7003 is better than OPC mix even under air curing and high temperature curing.

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