Sulfate Resistance of Cement Mortar Containing Metakaolin

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Abstract. Many of an extensive researches have been carried out by Libyan Industrial Research Center in natural row materials in south region of Libya [1] and the results found that there are many of material can be used as building material, one of these materials which widespread over large area is Kaolin clay.

From that point of view civil Eng. Dep. at Sebha university-Libya start thinking of using Kaolin clay as a building material and a team work has been formed to carry out a deep research study work on possibility of using the local natural pozzolana in south of Libya as a partially replacement of Portland cement. Previous laboratory study [2] which carried out by the team work proved that there is a possibility to use south Libya natural Kaolin clay as partial replacement of OPC after calcination and milling to get active calcined clay (Metakaolin).

Metakaolin being used very commonly as pozzolanic material or as supplementary cementing materials in mortar and concrete, and has exhibited considerable influence in enhancing the mechanical and durability properties of mortar and concrete as well as utilization of natural calcined clay has widely spread attention in the world to minimize the Portland cement consumption and manufacturing of which being environmentally damaging, in addition to its economic advantage.

Metakaolin when mixed with cement the silica of the pozzolana combines with the free lime released during the hydration forms additional cementitious C-S-H gel. This paper investigates the effect of metakaolin (MK) cement replacement on the resistance of mortar to sulfate attack for two different metakaolin quarries. Four MK replacement levels were considered in the study: 0%, 10%, 15%, and 20% by weight of cement for two quarries. After the specified initial moist curing period, mortar specimens were immersed in 5% sodium sulfate solution for a total period of one year. The degree of sulfate attack was evaluated by measuring expansion of mortar prisms, compressive strength reduction and observation of appearance of deterioration that usually accompanying with sulfate attack. All metakaolins examined in this study led to increases in strength and resistance to sulfate attack in comparing with controls samples or according to standard limits.

Keywords: Metakolin · Sulfate resistance · Expansion · Compressive strength · Visual inspection

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1 Experimental Program

1.1 Materials

1.1.1 Sources for Metakaolin

The clay soil (Pozzolana) used in this study was collected from two different locations near to Sebha city (Sebha and Temenhint sites) (Table 1).

Table 1. Chemical composition percentage of calcined clays

Oxides	Samples calcined clay	
	Sebha (A)	Temenhint (B)
SiO2	53.42	70.33
Al2O3	40.84	25.32
Fe2O3	0.975	1.05
Total SiO2, Al2O3, Fe2O3	95.20	96.70
SO3	0.033	0.016
MgO	0.130	0.140
CaO	0.100	0.080
Na_2O	0.220	0.350
K_2O	0.160	0.366
L.O.I	0.880	0.760

The collected stones were generally in dry condition with blocky structure and in order to obtain MK the stones were crushed and calcined at 800 °C for a period of 2.0 h. After the calcination process, the calcined clay stones were cooled then milled to pass on 150 μm sieve.

1.1.2 Cement

Locally available ordinary Portland cement (ASTM Type I).

1.1.3 Standard Sand

The standard used sand is locally siliceous sand.

1.1.4 Sodium Sulfate

Anhydrous sodium sulfate (Na_2So_4) was used.

1.2 Mixture Details

Seven mortar mixtures were prepared and used in this study as shown in Table 2.

Table 2. Mix proportions of blended cement

Sample/Mix	Symbol	Clacined clay MK (%)	Portland cement (%)
Control Sample	CS	0	100
Sebha sample (A)	AI	10	90
	AII	15	85
	AIII	20	80
Temenhint sample (B)	BI	10	90
	BII	15	85
	BIII	20	80

1.2.1 Specimens Preparation

The water-cementitious ratio (w/b) was fixed at 0.49 by mass without plasticizer. For compressive tests, 50.0 mm cubes and for expansion test and visual inspection 25 * 25 * 285 mm prisms were prepared. The cubes and prisms are cured one day in molds by covering with wet cotton. After demolding, specimens were immersed in lime water until day of testing.

1.2.2 Sulfate Solution

According to ASTM C1012 each liter of solution is contained 50.0 g of Na_2SO_4. The solution should be stored at 23.0 ± 2.0 °C. The pH of the solution must be determined before use and the solution is rejected if the pH range is outside 6.0 to 8.0.

2 Test Procedure

2.1 Cubes

The first group of cubes stored in water tank for an initial moist curing period of 3, 7, 28,180 and 365 days. The second group is cured for 28 days in water then immersed in sulfate solution for period of 6 and 12 month. The results of compressive strength are used to determine the Strength Activity Index (SAI) accordance to ASTM C618 as follows:

$$SAI = A/B * 100 \tag{1}$$

Where A is the compressive strength of test mixture, and B is compressive strength of control mixture.

Determination the Compressive Strength Reduction (CSR) for cubes immersed in sulfate solution comparing to that stored in water for the period of 6 and 12 months was done using the next formula:

$$CSR = \frac{\sigma_m - \sigma_s}{\sigma_m} * 100 \tag{2}$$

Where σm is the average compressive strength (in MPa) of three cubes cured in water and σs is the average compressive strength (in MPa) of three cubes immersed in 5% sulfate solution.

2.2 Prisms

The length change measured by using the length comparator in accordance with Specification ASTM C 490. Length change is calculated accordance to ASTM C1012 at any age as follows:

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100 \tag{3}$$

Where ΔL is the change in length at x age, %, Lx is the comparator reading of specimen at x age, Li is the initial comparator reading of specimen at the same time and Lg is the nominal gage length, or 250 mm as applicable.

3 Result and Discussion

3.1 Pozzolanic Activity

Pozzolanic activity is determined by strength activity indexes (SAI) which must be greater than or equal 75% according to ASTM 618-03 as shown on Table 3.

Table 3. Strength activity index

Samples	AI	AII	AIII	BI	BII	BIII
SAI % at 7 days	105.73	93.97	113.77	102.76	87.30	93.26
SAI % at 28 days	104.77	103.15	116.03	116.99	104.27	106.83

3.2 Compressive Strength Reduction CSR

CSR is another reliable measurement to indicate the sulfate attack as shown in Fig. 1.

Loss in strength during the exposure period can be as the result of cracking caused by expansion of ettringite and gypsum formation and/or the loss of C-S-H. Metakaolin suffered less strength loss than the control mortar as shown in Fig. 1. The compressive strength reduction ratio is varying from 2.14% to 24.23% for 6 months period and from 15.86% to 62.03% for 12 months period. The max. value of CSR at both periods was for control sample (CS). The best result is achieved by replacing 20% of cement by MK of sample BIII sample AIII respectively.

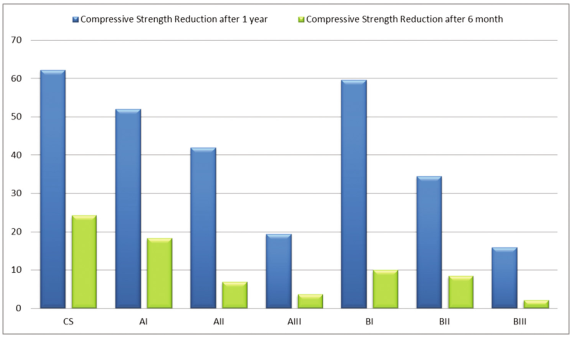

Fig. 1. Compressive Strength Reduction (CSR)

3.3 Expansion Test Results

From the results given in Fig. 2. all MK mortar series (except AI) fall below the standard limit 0.05% and 0.1 for 6 months period of ASTM C 1157 and ASTM C 595. The lowest expansion values were obtained by using 20% of MK of sample AIII and BIII which achieved an expansion of 0.033% and 0.034% respectively which lower than the control sample by 78% and 77% respectively.

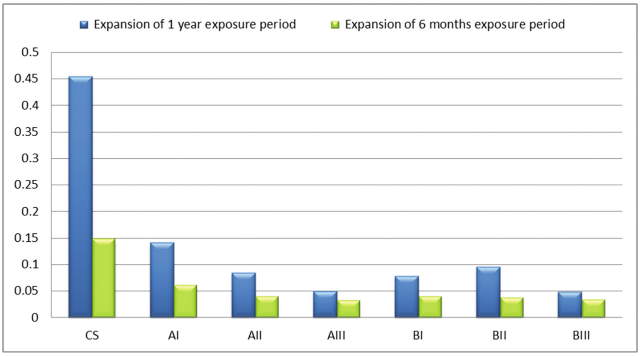

Fig. 2. Expansion of prism mortars after 6 and 12 months

After 12 months of exposure to sulfate solution only CS and AI fail to comply with requirements of ASTM C 1157 of expansion limit 0.1%. The lowest values were achieved also by using 20% of MK of samples (AIII and BIII) which gained an expansion of 0.049% of both samples which lower than the control sample by 89%. The reasons which can be given for the increase in the sulfate resistance in mortar with MK are that the pozzolanic reaction between the MK and calcium hydroxide (CH) released during cement hydration, witch consume part of (CH). Thus the quantity of expansive gypsum formed by the reaction of calcium hydroxide will be less in MK mortar than in plain mortar at specified level. Furthermore, the formation of secondary C-S-H by the pozzolanic reaction, although less dense than the primary C-S-H gel is effective in filling segmenting large capillary pores and increase the impermeability of the mortars [2–8].

3.4 Visual Inspection

A visual inspection of specimens as shown in Fig. 3 revealed the deterioration of the prismatic samples under sulfate attack.

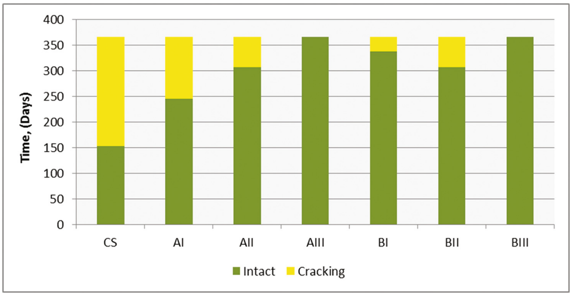

Fig. 3. Visual inspection of prismatic samples of one year period

The presence of MK is beneficial regarding appearance of cracks or any signs of deterioration associated with sulfate attack, since it could delay or avoid formation of ettringite or gypsum which lead to expansion, cracking and deterioration of mortar samples.

Under sulfate attack, using of Sebha and Tenminhint MK with ratio of 20% as partial replacement to cement significantly delayed occurring of any visible cracks and kept the mortar prisms intact up to the 12 months.

4 Conclusions

This study presents the results of the effect of MK cement replacement on the resistance of mortar to sulfate attack. Based on the results obtained from this study, the following conclusions can be drawn:

1. The chemical analysis of all samples collected from two quarries is complied with ASTM C618 and the natural pozzolan (Metakaolin) can be classified as N class accordingly.
2. Metakaolin replacement of cement (10 to 20%) was found effective in improving the resistance of mortar to sulfate attack. The use of optimum value of MK replacement level which 20% for every quarry reduced effectively the sulfate expansion at the end of 6 month by 58–77% and by 69–89% at the end of 12 month in comparing with the control mortar.

3. Most of results of MK mortar expansion results comply with expansion limits of ASTM C 1157 standard performance specification for hydraulic cement and ASTM C595 Standard Specification for blended hydraulic cement.
4. Deteriorations such which usually associated with sulfate attack to mortar sample can be delayed or prevented by using 10 to 20% of metakaolin that produced by fired kaolin clay collected from two studied locations.
5. Metakaolin suffered less strength loss than control mortar. The use of 10–20 MK of two quarries reduces the compressive strength reduction when kept in sulfate solution.
6. For more confirmation of these results, sulfate testing should continue for a period of 18 month or until mortar bars have expanded beyond the length of the comparator.

References

1. Industrrial Research Center: Raw material in SEBHA and WADI SHATTI region. Indus. Res. J. **7**, 126–138 (1998). Tripoli, Libya
2. Alamin, H.M.A.: Use of Local Poaaolanic Material for Concrete Production. Sebha University (2008)
3. American Concrete Institute Reports: ACI C201-2R-01 Guide to Durable Concrete
4. American Concrete Institute: ACI 232.1R-00 Use of Raw or Processed Natural Pozzolans in Concrete
5. Kalny, J.S., Marchand, J., Odler, I.: Sulfate Attack on Concrete. Spon Press, London (2002)
6. Alakhras, N.M.: Durability of metakaolin to sulfate attack. Cem. Concr. Res. **36**, 1727–1734 (2006)
7. Khatib, J.M., Wild, S.: Sulfate resistance of metakaolin concrete. Cem. Concr. Res. **28**(1), 83–92 (1998)
8. Yazici, S., Arel, H.S., Anuk, D.: Influence of metakaolin on the durability and mechanical properties of mortar. Arab. J. Sci. Eng. **39**, 8585–8592 (2014)

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