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Comparison of fly ash, silica fume and metakaolin from mechanical properties and durability performance of mortar mixtures view point

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HIGHLIGHTS

- The contribution of silica fume and metakaolin to the strength was started as early as 3 days.
- For the fly ash this duration was 180 days.
- Mineral admixtures improved transport properties greater than mechanical properties.
- The presence of the mineral admixture and its type changed the ettringite morphology.

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ABSTRACT

In this study the effect of cement replacement with fly ash, silica fume and metakaolin on the compressive strength, dynamic elastic modulus, chloride-ion penetration, water absorption, water sorptivity, and freeze–thaw and sulfate resistance of the mortar mixtures were comparatively investigated. In addition, micro-structural investigation was performed on some selected mortar mixtures, and regression analysis was applied on the sulfate resistance test results. It was observed that, the presence of the mineral admixture and its type changed the ettringite morphology. Besides, only ball-ettringite and a special type of ettringite were observed in the silica fume- and metakaolin-bearing mixtures, respectively. The needle-like and ball-ettringite formation were found in the fly ash mixtures. In the control mixture the needle-like, ball-ettringite and massive ettringite were detected. Overall test results revealed that the performance of the mixtures was arranged in descending order as silica fume-, metakaolin-, fly ash-bearing mixtures and the control one.

1. Introduction

Some of the mineral admixtures used nowadays are industrial by-products [1,2]. Silica fume is a by-product of the manufacture of silicon metal and ferrosilicon alloy and it contains more than 80–85% SiO_2 in amorphous form. Thus, it has highly pozzolanic properties and is suitable to use in the cement and concrete industries [3–6]. Metakaolin is produced by calcinations of pure or refined kaolinitic clay at a temperature range between 650 and 850 °C. It is also a processed amorphous silica material [7]. Fly ash is obtained as a waste product upon the combustion of pulverized coal in thermal power plants [8–10].

Mineral admixtures are used as cement replacement material in mortar mixtures and several special types of concrete such as self-compacting, reactive-powder, roller compacted and lightweight concrete. Mineral admixtures are used in order to improve mechanical properties of the mixture because of its pozzolanic and/or self cementitious nature. In addition, resistance of the mixture to freeze–thaw cycles [11], sulfate attack [12,13], acidic attack [14], alkali–aggregate reaction [15] and reinforcement corrosion [16] as well as transport properties of the mixture [17,18] were reported to be improved with the addition of mineral admixture. However, the improvement of the above mentioned properties of the cementitious systems were found to be higher when ultrafine mineral admixtures such as silica fume and metakaolin were incorporated into the mixture [19,20]. Besides, mineral admixtures cause to decrease cost of the mixture upon enhancement of workability of fresh concrete. Moreover, fresh concrete mixtures containing mineral admixtures are less prone to bleeding [1].

The effect of mineral admixtures on the properties of mortar or concrete mixtures was investigated by many researchers. However, there is relatively little data on the microstructure of the binders exposed to various deleterious effects. The aim of this study was to investigate comparatively the effect of fly ash, silica fume and metakaolin on the strength, ultrasonic pulse velocity,

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dynamic elastic modulus, chloride ion penetration, water absorption, water sorptivity, sulfate resistance and freeze–thaw resistance of cement mortars. For this purpose, 10 w% of the cement was replaced with fly ash, silica fume and metakaolin. The 7, 28, 90, 180 and 300-day compressive strength, as well as 300-day transport properties and freeze–thaw resistance of the mortar mixtures were determined. Besides, the behavior of the mixtures exposed to sodium and magnesium sulfate solutions for 300 days was studied. In addition micro-structural investigation and regression analysis was performed on mortar mixtures exposed to sulfate attack.

2. Experimental study

2.1. Materials

In this study, a CEM I 42.5 R type cement conforming to EN 197-1 standard [21] was used. A high-lime (class C according to ASTM C 618-03 [22]) fly ash conforming to EN 450-1 standard [23], a silica fume and a metakaolin were used as the cementitious material. Pozzolanic activity indices of the mineral admixtures were determined according to ASTM C311 [24]. The chemical composition, as well as some mechanical and physical properties of the cement, fly ash, silica fume and metakaolin obtained from their manufacturers, is presented in Table 1. The standard sand conforming to EN 196-1 standard [25] with saturated surface dry bulk specific gravity of 2.72 and absorption capacity of 0.70% was used.

In all of the mixtures, water/binder (w/b) ratio and sand/binder (s/b) ratio were kept constant as 0.485 and 2.75 (by weight), respectively. In addition to the control mixture containing no mineral admixture; in the test mixtures, 10 wt% of portland cement was replaced with fly ash, silica fume and metakaolin. Flow values of the fresh mortar were determined in accordance with ASTM C1437 [26]. The recorded flow values were in the range of 170 ± 20 mm. The lowest flow values were obtained in the mixtures containing either silica fume or metakaolin. This was due to the extremely high fineness values of these admixtures.

2.2. Test procedures

50 mm cube specimens were prepared for compressive strength and ultrasonic pulse velocity tests. 7, 28, 90, 180 and 300-day compressive strengths and ultrasonic pulse velocities of the mortar mixtures were determined according to ASTM C109 [27] and ASTM C597 [28] standards, respectively. Dynamic elastic moduli of the mixtures were calculated regarding their ultrasonic pulse velocity and density values using Eq. (1) [2,29]:

$$E_{dn} = \rho c^2 \frac{(1+v)(1-2v)}{(1-v)} \quad (1)$$

where, E_{dn} = dynamic elastic modulus of the mortar (MPa), ρ = hardened density (kg/m^3), c = ultrasonic pulse velocity (km/s) and v = Poisson's ratio. Poisson's ratio was assumed as 0.2 for all of the mixtures.

The 100 × 50 mm cylinder specimens were prepared for chloride-ion penetration, water absorption and freeze–thaw resistance tests. In addition, 40 × 40 × 160 mm prism specimens were prepared for water sorptivity test. The chloride-ion penetration, water absorption, water sorptivity and freeze–thaw resistance of the mortar mixtures cured for 300 days in water were determined in accordance with ASTM C1202 [30], ASTM C642-97 [31], ASTM C1585 [32] and ASTM C 666 [33] standards, respectively.

For chloride penetration test, the amount of electrical current passed through the cylinder specimens was measured for 6 h. At the end of 6 h the total charge passed, in coulombs, was measured. For water absorption test, saturated surface dry specimens were weighed and then kept in an oven at 105 ± 5 °C until attaining a constant mass. For freeze–thaw resistance test, the mortar specimens were frozen in air from 5 ± 2 °C to −18 ± 2 °C within 3 h and were thawed in 5 ± 2 °C water within 1 h in a single cycle. The changes in weight of each specimen were calculated at every 30 freeze–thaw cycles until 300 cycles in accordance with TSE CEN/TS 12390-9 [34] standard. For water sorptivity test, the specimens were dried at 105 °C until a constant weight, and then the side surfaces of the specimens were sealed with an acrylic copolymer-based sealing material. Supporting rods were placed at the bottom of a pan and the pan was filled with the tap water to provide 1–3 mm water level on the top of the supporting rods. At 0, 5, 10, 20, 30, 60, 120, 180, 240, 300, 360 min time intervals the specimens were weighed. The sorptivity, I, was obtained from Eq. (2):

$$I = \frac{m_t}{A \cdot d} \quad (2)$$

where, I = sorptivity (mm), m_t = change in weight at the time t, A = exposed area of the specimen (mm^2), and d = density of the water (g/mm^3).

25 × 25 × 285 mm prism specimens were prepared for sulfate resistance tests. Sulfate resistance tests were performed according to ASTM C1012 standard [35]. The specimens were separately immersed in 5% sodium sulfate and 4.2% magnesium sulfate solution. The length changes were measured every 30 days until 300 days. Then micro-structural investigations were performed on selected specimens. After exposure to sulfate solutions, the specimens were cut and dried at 35 °C and 50% relative humidity for 2 h. The micro-structure of the specimens was studied by using JEOL JSM 6060 electron microscope. Scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) analyses were attempted to identify the composition of these materials and their morphology. EDS results are the average of three measurement.

3. Test results and discussion

3.1. Compressive strength

Compressive strength values of the mortar mixtures are shown in Fig. 1. The silica fume-bearing mortars showed the highest compressive strength values compared to those of the other mortar mixtures. However, the fly ash mortars presented the lowest compressive strength values up to 180 days. Beyond this age, compressive strength values of the fly ash-bearing mixtures were higher than that of the control mixture. The compressive strength of the mortar mixtures containing metakaolin and silica fume were higher than that of the plain mixtures at all ages. As it is well known, the pozzolanic materials form additional calcium silicate hydrate (C–S–H) upon the reaction of reactive silica of pozzolan and calcium hydroxide (CH) produced by the cement hydration. This provides additional strength particularly at later ages. Since,

Table 1
Some physical, chemical and mechanical properties of using materials.

Chemical Composition (%)					Physical Properties of Cement		
	Cement	FA*	SF*	MK*	Initial setting time (min)		110
SiO_2	23.84	32.80	87.29	63.53	Final setting time (min)		166
Al_2O_3	4.2	13.77	0.47	32.36	Volume expansion (mm)		1
Fe_2O_3	3.4	4.78	0.63	0.54	Specific gravity	Cement	3.11
CaO	61.0	39.69	0.81	0.29		Fly ash	2.29
MgO	1.8	2.05	4.47	0.18		Silica fume	2.10
Na_2O	0.20	0.40	1.25	0.33		Metakaolin	2.20
K_2O	0.46	1.18	1.28	1.08	Specific surface area (cm^2/g)		
SO_3	2.93	4.22	0.22	0.01	Cement (Blaine method)		3360
Cl^-	0.0064	–	–	–	Fly ash (Blaine method)		4040
LOI	1.74	1.34	2.70	1.00	Silica fume (BET method)		18000
IR	0.30	–	–	–	Metakaolin (BET method)		11768
Compressive Strength of Cement (MPa)					Pozzolanic activity index (%)	7-Day	28-Day
3-day				25	Fly ash	69	79
7-day				37.2	Silica fume	101	132
28-day				44.6	Metakaolin	102	110

* FA = Fly ash, SF = Silica fume and MK = Metakaolin.