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Effect of gypsum type on properties of cementitious materials containing high range water reducing admixture

Ali Mardani-Aghabaglou [a, *], Onur Can Boyacı [a], Hojjat Hosseinnezhad [a], Burak Felekoğlu [b], Kambiz Ramyar [a]

[a] Department of Civil Engineering, Ege University, Bornova-Izmir, Turkey
[b] Department of Civil Engineering, Dokuz Eylul University, Buca-Izmir, Turkey

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ABSTRACT

In this study, the effect of cement gypsum type on properties of the properties of cement paste, mortar and concrete mixtures containing high range water reducing admixture (HRWR) was investigated. Two different types of cement prepared from the same clinker but containing either calcium sulfate hemihydrate or dihydrate as retarder were used. The fresh and hardened (compressive strength and drying-shrinkage) properties as well as static and dynamic rheological behavior of the mixtures were investigated. Compared to the mixtures containing dihydtate, the fresh and rheological properties of mixtures were negatively affected when cement-containing hemihydrate was used. However, hemihydrate utilization had a positive influence on the early compressive strength. The adverse effects on fresh properties were more significant in paste mixtures. These negative effects decreased in the mortar and concrete mixtures. The presence of hemihydrate in cement was found to increase the drying-shrinkage.

1. Introduction

Water reducing admixtures may show incompatibility with some cement [1–3], which may result in some side effects in concrete such as early stiffening, set retardation, loss of workability, segregation, increasing risk of bleeding and shrinkage [4–8]. These effects may be arisen from cement and water reducing admixture themselves as well as mix proportions, concrete temperature and curing conditions [4,5,9]. The negative effects resulted from cement may include alkali content of the cement; crystal structure of the major components; fineness of the cement; type and content of the gypsum. The major factors contributing to the incompatibility and arisen from the polycarboxylate-based high range water reducing admixture can be listed as chemical composition, molecular weight, length of the main chain, number and length of the side chains and type of the bond between the molecules. Besides, sequence and time of addition of the admixture into the concrete are known to affect its performance in concrete [4,5,9–16]. The results of some studies are summarized below.

The effect of utilization of natural anhydrite gypsum type instead of natural gypsum rock on the properties of cement paste prepared from CEMI and CEMII cements was investigated by Tzouvalas et al. [17]. It was shown that natural anhydrite could efficiently retard the setting of cement with no significant change in the physico-mechanical properties of the hardened paste. In another research, the effect of different types of calcium sulfate (gypsum rock, bassanite and anhydrite) on the hydration of calcium sulfoaluminate eco-cement was investigated. The dissolution rate of the calcium sulfate was found to be an important factor to control the hydration reactions. Bassanite dissolved very fast and hence the initial setting time of the pastes and mortars was too short to produce homogeneous samples. Anhydrite dissolved slowly so after 1 day the amount of ettringite formed was lower than that in pastes containing gypsum rock, resulting in mortars with lower compressive strengths. After 3 days, pastes containing anhydrite showed slightly larger ettringite contents and hence, mortars with slightly higher compressive strengths. Ettringite content was reported to be the chief parameter to explain the strength development in these cements [18]. In another study a blast furnace slag portland cement was added to β-hemihydrate to observe the changes in mechanical properties and microstructure of the mixture. A plaster made from this mixture and exposed to the external environment for three years showed no volume change, indicating that no ettringite was formed. SEM analysis

* Corresponding author.
E-mail address: ali.mardani16@gmail.com (A. Mardani-Aghabaglou).

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showed a more compact structure resulting in a water resistant composite [19]. The use of calcium sulfate-bearing retarder materials such as natural gypsum, anhydrite and flue-gas desulfurization (FGD) gypsum in cement industry was investigated by Papageorgiou et al. [20]. The addition of FGD gypsum increased the setting time without affecting the compressive strength. The degree of dehydration of the hydrated calcium sulfate was found to regulate the setting and strength performance of the cement.

Determining the rheological properties of cement-based composites such as yield stress and viscosity are important. As known, the yield stress is divided into two as dynamic and static ones. According to Roussel [21], division of the yield stress into two types is a requirement of thixotropic behavior of cement-based composites. Static yield stress is defined as the shear stress that occurs right after the preparation of the mixture without any pretreatment. This value is mostly related with the time from the beginning of the mixing to the placement of the mixture into a rheometer. Static yield stress is the highest shear stress value obtained from a shear stress-time diagram in an undisturbed mixture. However, dynamic yield stress is defined as the point where the curve crosses shear stress axis in a shear stress-shear rate diagram. According to Roussel [21] the dynamic yield stress is related with the mix properties and it is not affected by the factors such as shear history because a balanced flow curve is already occurred upon the given effect. The dynamic yield stress depends on the degree of hydration and time. However; at very early stages of hydration where the degree of hydration is negligible, the dynamic yield stress may be accepted to be a constant value [22].

In the literature, no study about the effect of gypsum type in cement on the rheological properties of the binder system was found. In this study, rheological tests on the paste and mortar mixtures were performed and test results modeled with Herschel-Bulkley approach. In addition, there are many studies about the cement-water reducing admixture compatibility [23–27]. However, there is not an exact conclusion about the effect of gypsum type used in cement on properties of the cementitious materials containing HRWR admixture because of the huge number of factors involved.

Due to seasonal temperature variations, cement plants may change the type of gypsum used as retarder in their cements. The results of the present study showed the great variations in the fresh and hardened properties of the cementitious systems upon changing the type of gypsum in cement. Thus, this paper may provide a good guide for cement and concrete producers to have an idea about the possible changes in the behavior of cements containing different gypsum types. In this study, the effect of gypsum type in cement on properties of the cementitious materials containing high range water reducing admixture (HRWR) was investigated in cement paste, mortar and concrete mixtures. For this purpose, two different types of commercial cements prepared from the same raw materials but containing either hemihydrate or dihydrate as retarder were studied. The cement paste, mortar and concrete mixtures having different water/cement (W/C) ratios were prepared. Marsh-funnel flow time, mini-slump value, dynamic and static yield stresses and final viscosity of the cement pastes were investigated. Moreover, XRD analysis of hardened paste mixture was carried out to determine ettringite and monosulfate intensities. Admixture requirement, flow value, loss of flow, V-funnel flow time, loss of V-funnel flow time, rheological properties, compressive strength and drying shrinkage of the mortar mixtures were investigated. In the concrete mixtures, unit weight, air content, slump, loss of slump, slump flow, compressive strength and drying shrinkage tests were performed.

2. Materials, mix preparation and test procedures

2.1. Materials

In this study, two commercial CEMI 42.5 R type cements conforming to EN 197-1 [28] and containing different gypsum types were used. The clinkers of both of the cements were same, but the type of gypsum added to the clinker during the grinding was different. Depending on gypsum type, the cements and the mixtures prepared from these cements were designated as HG (hemihydrate gypsum-bearing) and DG (dihydrate gypsum-bearing). During the production of cement HG, calcium sulfate hemihydrate containing ½ mole of water ($CaSO_4 \cdot 1/2H_2O$), and in cement DG, calcium sulfate dihydrate containing 2 mol of water ($CaSO_4 \cdot 2H_2O$) were used. The chemical composition, mechanical and physical properties of the cement and gypsums given by the producer are shown in Tables 1 and 2, respectively.

The experimental study was performed in three stages on cement paste, mortar and concrete mixtures. Standard sand conforming to EN 196-1 [29] was used in the mortar mixtures. In the concrete mixtures, four crushed limestone aggregates with different size fraction as 0–3, 0–5, 5–15 and 15–25 mm were used. The proportions of these size fractions were adjusted to obtain a combined aggregate having a gradation conforming to TS 802 [30] requirements, as shown in Fig. 1. Specific gravity and water absorption capacity of the aggregates were determined in accordance with EN 1097-6 [31] standard. Physical properties of the aggregates are shown in Table 3. A polycarboxylate ether-based high range water-reducing admixture (HRWR) was used in the mixtures. The properties of the admixture given by its manufacturer are shown in Table 4.

2.2. Mixture preparation

2.2.1. Preparation of cement paste mixtures

The Marsh-funnel flow time and mini-slump test producers are similar to those applied by Aïtcin [32] and Kantro [33]. Regarding the previous studies, the water-cement (W/C) ratio of the paste mixtures was chosen as 0.35 for the Marsh-funnel and mini-slump tests. For each cement type, in addition to the control mixture without any chemical admixture, six different mixtures containing HRWR admixture ranging from 0.5% to 1.75 wt% of cement were prepared. However; during the investigation of rheological parameters, for each cement-admixture couple, 6 different cement pastes were prepared with two different W/C ratios (0.35 and 0.32) and three different admixture dosages (0, 0.1 and 0.2 wt% of cement). When the W/C ratio of the mixture was more than 0.35, the admixture requirement was negligible and segregation was observed in cement paste. When the W/C ratio was less than 0.32, the mixtures were too stiff to be tested by the available rheometer. On the other hand, preliminary study conducted by the authors of the paper showed that the available rheometer is not suitable for the paste mixtures having W/C ratio lower than 0.32. Thus, the mixtures prepared in this study had a W/C ratio between 0.32 and 0.35. The pastes were mixed by using a high-speed mixer to obtain a homogenous mixture.

2.2.2. Preparation of mortar mixtures

Mortar mixtures were prepared in compliance with the ASTMC109 [34]. In the entire mixtures W/C ratio, sand/cement (S/C) ratio and flow value were kept constant as 0.485, 2.75 and 270 ± 10 mm, respectively. In order to obtain the required flow value, the same polycarboxylate ether-based superplasticizer was used in all of the mixtures.