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Effect of high dosage lignosulphonate and naphthalene sulphonate based plasticizer usage on micro concrete properties

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HIGHLIGHTS

- Overdose usage of lignosulphonate (LS) and naphthalene sulphonate (NS) based admixtures were observed.
- Optimum dosage of LS and NS improved mortars' physical properties.
- Overdose usage of LS and NS has negative effect on mortar.
- SEM analysis proved porous structure of overdosed LS and NS mixtures.
- Pore volume decreased with optimum dosage, while it is increasing with over dosage.

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ABSTRACT

In concrete production, because of plasticizers are used for a small percentage by weight of cement (in the range from 0.3% to 1.5%), there is a possible risk adding more admixture in concrete than calculated from personnel or equipments sensitivity errors. In this situation concrete's strength and durability performances are diminishing. In this work, it is investigated the effect of high dosage lignosulphonate based water-reducing admixture and naphthalene sulphonate based high-range water-reducing admixture usage on mortar properties. It was carried out unit weight, flowability, setting time, air content, compressive strength, flexural strength, ultrasonic pulse velocity tests and microstrucural inspections on specimens which were produced with 5 different dosages for each admixture. As a result of experiments, in case of using admixtures with overdose, there would be loss of quality of physical and mechanical properties of concrete, for this reason it is concluded that, there must be some legal regulations using chemical admixtures sensitively.

1. Introduction

With the improvement of concrete technology, there are same parallel improvements in the sub-sectors that support concrete industry. Today, with the use of ready-mixed concrete, there is an increase in the use of chemical admixtures. In 90's, chemical admixture usage was not higher than 20% [1] in concrete industry in the world, but today, chemical admixtures become an essential component of ready-mixed concrete production [2]. Among long list of admixtures, normal plasticizers and superplasticizers are the major type of concrete admixtures in terms volume consumed [3].

Plasticizers (water-reducers) and superplasticizers (high range water reducers) are admixtures which reduce the water content of mixed concrete without detriment to its consistency or enhance its slump with or without change to the water content or cause both effects simultaneously. Plasticizers and superplasticizer essentially contain lignosulphonate, naphthalene sulphonate, melamine sulphonate or polycarboxylate/polycarboxylic [4–8].

Lignosulphonate (LS) is an inexpensive and water-soluble product which is separated from brown liquor by the pulp and paper industries [9]. After alcohol extraction, evaporation, sulphonation and spray drying, the resulting admixture which is in powder form, has about 60% lignosulphonates [10]. LS has been broadly used in concrete production as regular water-reducing admixture for many years due to its relatively low price [11]. Naphthalene sulphonate (NS) is made of naphthalene, sulforic acid, formalin and

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alkali, through sulphuration, hydrolyzation, condensation and neutralization reaction. Both type of admixtures act by electrostatic repulsion effect [12,13]. The admixture gives cement particles a negative potential owing to the effect of sulfonic group separation [14,15].

Because of being very big research area, plenty of work conducted about plasticizers, but some of them are summarized in this section of the work. Nagrockiene et al. [16] researched the effect of cement type and plasticizer addition on concrete properties and found that plasticizer type and dosage play a key role in successful cement application in concrete. Ouyang et al. [17] studied adsorption amounts on the surface of cement particles, zeta potential of cement particles containing lignosulphonate and found that when the dosage of lignosulphonate is less than 0.5 wt.% of the cement, the lignosulphonate fraction with a molecular weight of 10,000–30,000 gives a maximum fluidity in the cement paste. Burgos-Montes et al. [18] investigated the compatibility between CEM II cement and four type superplasticizers, and concluded that naphthalene (NS) is the most effective traditional admixture in all the type II cements studied.

In concrete production, because of chemical admixtures are used for a small percentage by weight of cement (for example, plasticizers are used about 0.3–1.5%), there is a possible risk adding more admixture in concrete than calculated, because of personnel fault or equipment sensitivity errors. In this situation, unwanted secondary effects may occur. In some cases, these secondary effects end up a rapid and irreversible slump loss, in other cases result in a strong retardation, in other cases come out a segregation or extreme bleeding [19]. Some chemical admixtures don't affect the concrete stability, but they extend the setting time extremely that cause unable to de-mold concrete [1].

There are plenty of works about plasticizers but there are limited works about high dosage usage of plasticizers which are generally focused on strength properties. This work is different from others with taking the subject in wide perspective consisting fresh and hardened state properties and microstructure analyses. The aim of this study is to determine the effect of overdose usage of lignosulphonate based plasticizer and naphthalene sulphonate based superplasticizer on concrete properties. For this purpose, it was produced mortars with the use of five different admixture dosages. All components (sand, water, cement) except admixture were treated equally. To determine the fresh state properties of mortar; unit weight, flowability, setting time and air content tests were conducted. To determine the properties of hardened state of mortars; compressive strength, flexural strength and ultrasonic pulse velocity were observed. Finally micro structure analyses were conducted.

2. Experimental study

2.1. Materials

Locally available CEM I 42.5 R Portland cement, which satisfies EN 197-1, was used. The chemical and physical properties of cement are given in Table 1. As mixing water, Eskişehir tap water was used. The chemical analysis of the drinkable water is given in Table 2. In order to produce mortar, CEN standard sand that satisfies EN 196-1 was used. In order to investigate the effects of lignosulphonate and naphthalene sulphonate based water-reducing admixtures, two different commercially available admixtures, supplied from Grace Company, were used and their characteristics are given in Table 3.

Table 1
Chemical and physical properties of cement.

SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	SO_3	LOI
19.42	63.80	4.47	2.70	1.21	0.28	0.59	2.89	4.18

Spec. gravity	Blaine, cm^2/g	Compressive strength, MPa		
3.06	3455	25.2 (2-day)	44.9 (7-day)	59.8 (28-day)

Table 2
Chemical analysis of mixing water.

pH (20 °C)	Cl- mg/l	SO_4 mg/l	Mg mg/l	Ca mg/l	Zn mg/l	Cu mg/l	Fe mg/l	NO_3 mg/l	ClO_2 mg/l
7.49	6.53	91.5	41.5	63.8	0.375	0.092	0.074	4.35	<0.09

Table 3
Properties of admixtures.

Properties	LS	NS
Ingredient	Sodium lignosulphonate	Naphthalene sulphonate
Color	Dark brown	Dark brown
State	Liquid	Liquid
Density g/ml (20 °C)	1.136	1.188
pH (20 °C)	10.3	10.1
Total chloride %	<0.10	<0.10
Total solid (%)	19.9	35.2

2.2. Method

In order to observe the differences in concrete by the dosage of admixture, cement and sand quantities were set to equal in each mixture. Mixing water was reduced as much as admixture amount. The water-cement ratio is selected as 0.50 and sand-cement ratio is selected as 3 for the mortar production. In principle, 5 different norm dosages including control were prepared for each admixture. But in order to reach definitive results for some tests, like compressive strength test, additional dosages in the range of 0–5% were applied. Dosages were assigned with preliminary trials. It was observed with preliminary trials that specimens lost their stability and properties of being concrete after the dosage of 5%. For this reason, maximum dosage was chosen as 5%. The designation and norm dosages of admixtures are given in Table 4. Lignosulphonate and Naphthalene sulphonate based admixtures prepared in this study are denoted as LS and NS, respectively.

Prepared mortar was cast into 4 × 4 × 16 cm dimensioned formworks made of steel directly after mixing. The samples were separated into groups, each cured at a constant temperature of 20 °C in the curing pool.

2.3. Tests

With the produced mixtures, fresh state tests; such as unit weight, flowability, setting time and air content tests were conducted. Also, in order to observe hardened state properties, ultrasonic pulse velocity, flexural and compressive strength tests were conducted. At the last part of the work micro structure inspections, X-ray diffraction (XRD), Scanning electron microscope (SEM) and nitrogen absorption/desorption analysis were conducted.

2.3.1. Fresh state tests

Fresh unit weight was obtained by following EN 12350-6 and using Eq. (1).

$$D = \frac{m_2 - m_1}{V} \quad (1)$$

where D is unit weight (kg/dm^3), m_1 empty weight (kg) of cast, m_2 weight (kg) of cast with mixture and V is the volume (dm^3) of cast.

To evaluate the flowability of mixtures, which is one of the key tests of plasticizers, the flow table tests were carried out following EN 1015-3 [20]. As a test procedure; after lifting the slump cone, two diameters perpendicular to each other are measured and their mean is noted as relative slump.

Table 4
The designation and norm dosages of admixtures.

Admixture	Optimum dosage[a]	Dosages and designations				
LS	0.3–1.0%	0% Control	1.0%	3.0%	4.0%	5.0%
		C-0	LS-1	LS-3	LS-4	LS-5
NS	0.8–1.5%		1.0%	3.0%	4.0%	5.0%
			NS-1	NS-3	NS-4	NS-5

[a] Optimum dosage refers to dosage range that suggested by producer. It refers to a range, because optimum dosage must be assessed after preliminary trials depending upon the actual mix constituents and specifications required.