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***Research Article***

# The Effect of Admixture Percent of Sulfonate-Based Admixtures on Mechanical and Microstructural Properties of Cement Mortars

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**Abstract**

Concrete is one of the most widely used building materials all over the world. The main purpose of the concrete industry is to produce more durable concrete. Concrete, which is the most basic need of people, especially in residences, consists of cement, aggregate, water, and chemical admixtures if necessary, according to the desired feature. Plasticizing admixtures are used to increase the workability of fresh concrete and reduce the water amount in the mixture. This study investigated the effects of sulfonatebased superplasticizers on flow rate, compressive strength, and crystalline structure. Cement, sand, water, and two different admixtures (naphthalene sulfonate, lignosulfonate+naphthalene sulfonate) were used to prepare cement mortars. The water/cement ratio was constant at 0.5 while the admixture percentages varied at different rates as 0.2, 0.5, and 1. The flow rates were determined using a flow table. The compressive strength tests were performed on each cement mortar aged 7 and 28 days. The crystalline structure of the 28-day aged samples was determined by X-ray Diffractometry (XRD) Spectrometer. The flow rates increased with the increasing admixture ratio. The highest compressive strength was obtained as 49.94 MPa and 51.33 MPa for naphthalene sulphonate and lignosulphonate+ naphthalene sulfonatebased admixtures, respectively. The intensity of the main crystalline structure increased with increasing admixture ratios up to 0.5% and decreased with increasing ratio to 1%. The highest compressive strengths were obtained with 0.5% naphthalene sulfonate and 0.2% lignosulfonate+naphthalene sulfonate admixtures for the 28 days aged cement mortars.

**Keywords:** – Naphthalene Sulfonate based admixture, Lignosulfonate+ naphthalene sulfonate based admixture, Compressive Strength, Crystalline Structure

# Sülfonat Esaslı Katkı Yüzdesinin Çimento Harçlarının Mekanik ve Mikroyapısal Özelliklerine Etkisinin Belirlenmesi

**Öz**

Beton, tüm dünyada en yaygın kullanılan yapı malzemelerinden biridir. Beton sektörünün temel amacı daha dayanıklı beton üretmektir. Özellikle insanların en temel ihtiyacı olan konutlarda beton, istenilen özelliğe göre çimento, agrega, su ve gerekirse kimyasal katkılardan oluşmaktadır. Taze betonun işlenebilirliğini artırmak ve karışımdaki su miktarını azaltmak için akışkanlaştırıcı katkılar kullanılır. Bu çalışma, sülfonat bazlı süper akışkanlaştırıcıların yayılma çapı, basınç dayanımı ve kristal yapı üzerindeki etkilerini araştırmıştır. Çimento harçlarının hazırlanmasında çimento, kum, su ve iki farklı katkı (naftalin sülfonat, lignosülfonat+naftalin sülfonat) kullanılmıştır. Su/çimento oranı 0,5'te sabit kalırken, katkı yüzdeleri 0,2, 0,5 ve 1 gibi farklı oranlarda değiştirilmiştir. Yayılma çapları bir yayılma tablası kullanılarak belirlenmiştir. 7 ve 28 günlük her bir çimento harcı üzerinde basınç dayanımı testleri yapılmıştır. 28 günlük numunelerin kristal yapısı, X-ışını Difraktometrisi (XRD) Spektrometresi ile belirlenmiştir. Artan katkı oranı ile akış hızları artmıştır. En yüksek basınç dayanımı naftalin sülfonat ve lignosülfonat+naftalin sülfonat esaslı katkılar için sırasıyla 49.94 MPa ve 51.33 MPa olarak elde edilmiştir. Ana kristal yapının yoğunluğu, katkı oranlarının %0.5'e kadar artmasıyla artmış, %1'e artmasıyla ise azalmıştır. 28 günlük çimento harçlarında en yüksek basınç dayanımları %0.5 naftalin sülfonat ve %0.2 lignosülfonat+naftalin sülfonat katkıları ile elde edilmiştir.
**Anahtar Kelimeler:** Naftalin Sülfonat esaslı katkı, Lignosülfonat+ naftalin sülfonat esaslı katkı, Basınç Dayanımı, Kristal Yapı

# 1. Introduction

Cement, which was discovered by mixing gypsum and crushed stones, was developed as a result of the efforts of scientists in the 1800s and started to be produced by mixing gypsum and clay at high temperatures. The cement used today is formed by melting or sintering aluminum oxide, calcium oxide, iron oxide, and silicon oxide [1].

The structure, which is a composite material containing cement, water, aggregate, and chemical or mineral admixtures designed according to the area is called concrete [2].

Superplasticizers (also called high-range water reducers) are a class of plasticizers that have less harmful effects and can be used to increase workability more than conventional plasticizers. Superplasticizers are also can be used to increase compressive strength. It increases the workability of concrete and reduces the need for water content by 15-30% [3].

The purpose of plasticizing admixtures is to reduce the water content in the concrete while maintaining its fluidity and workability. The strength and durability of concrete increase with decreasing water content. The disadvantages of superplasticizers when added to the concrete in large quantities are causing delay and bleeding in the hardening of the concrete and entering a high amount of air [4].

Lignosulfonates (LS) based admixtures are the first admixtures used in the group of water-reducing admixtures in concrete. Lignosulfonates are formed as a result of the delignification of lignin, which is the raw material of the paper industry, the degree of sulfonation is increased to control both the solubility and the plasticizing effect. They have a water reduction capacity of 8-10% [5].

Naphthalene sulfonates (NS) are produced by the sulfonation of naphthalene with sulfuric acid. It is widely used as a concrete admixture due to its resistance to freezing and the introduction of air into the concrete [5].

As the number of water increases, the workability of the concrete increases. Water enters between the cement particles and ensures that the cement paste is in a fluid state, but this leads to loss of strength and bleeding. For this reason, it is necessary to use appropriate chemical admixtures [6].

This study, it was aimed to determine the appropriate plasticizer amount used in cement mortars which obtain the highest fluidity and compressive strength.

# 2. Material and Method

## 2.1. Material

In this experimental study, cement, sand, water, and two different commercial sulfonate-based superplasticizers (lignosulphonate+ naphthalene sulfonate and naphthalene sulphonate) were used. The experiments were carried out with a constant water/cement ratio of 0.5 while the superplasticizer ratios varied from 0.2%, 0.5%, and 1%.

## 2.2. Method

Cement mortars were prepared according to TS EN 196-1. The superplasticizers were first mixed with water and then added to the cement and sand mixture. Flow values of each mortar were measured with a flow table. The mortars were poured into 4x4x16 cm steel molds. The molds were vibrated to prevent air bubbles. After 24 hours the mortars were removed from the mold and cured in a water bath for 7 and 28 days. Compressive strengths were performed on all samples. The crystal structures of the samples cured for 28 days were determined by X-ray Diffractometry (XRD). The composition of the cement used was examined with X-ray Fluorescence (XRF) Spectrometer and the result is given in Table 2.1.

**Table 2.1**. *Chemical composition of cement*

| Composition | % |
|---|---|
| CaO | 62.2583 |
| $SiO_2$ | 18.9675 |
| $Al_2O_3$ | 4.7248 |
| $Fe_2O_3$ | 2.9604 |
| $SO_3$ | 2.6057 |
| MgO | 1.8218 |
| $K_2O$ | 0.6058 |
| $TiO_2$ | 0.4928 |
| $Na_2O$ | 0.3893 |
| $P_2O_5$ | 0.0866 |
| $Cr_2O_3$ | 0.0613 |
| MnO | 0.0387 |

# 3. Results and Discussion

## 3.1. Flow Test & Compressive Strength Analysis

The flow rates of each cement mortar prepared by using two different plasticizers with constant water/cement ratio and varying admixture ratios changed as 0.2%, 0.5%, and 1% were given in Figure 1.

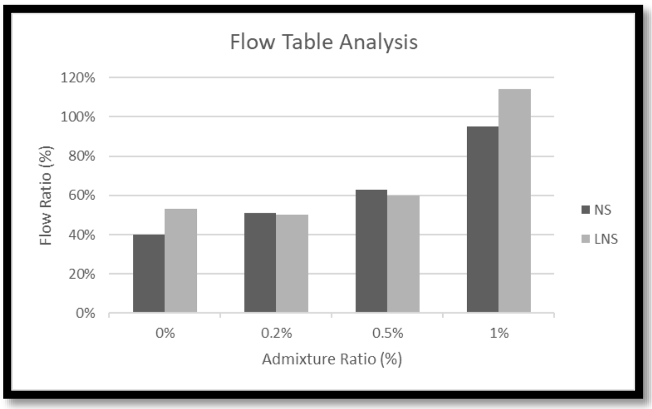


**Figure 3.1.** *Flow rates of the cement mortars with/ without admixture*

In Figure 3.1. it was observed that as the amount of admixture increased, the workability and flow diameters were increased. The increment was very significant when the admixture ratio was increased to 1%. The highest flow rate was obtained as 95% with lignosulfonate+naphthalene sulfonate (LNS) admixture. The compressive strength values of cement mortars prepared with naphthalene sulphonate-based admixture and aged for 7 and 28 days are given in Figure 3.2.

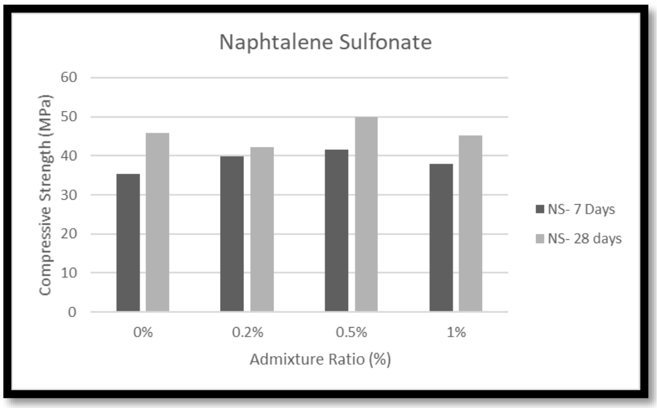


**Figure 3.2.** *The compressive strength values of cement mortars prepared with naphthalene sulphonate based admixture and aged for 7 and 28 days*

It was observed that the compressive strengths of the mortars increased by increasing the admixture ratio up to 0.5. The strengths of the mortars increased to 49.95 MPa from 45.9 MPa when the admixture ratio was increased to 0.5%. $C_3S$

The compressive strength values of cement mortars prepared with lignosulphonate + naphthalene sulfonate-based admixture and aged for 7 and 28 days are given in Figure 3.3.

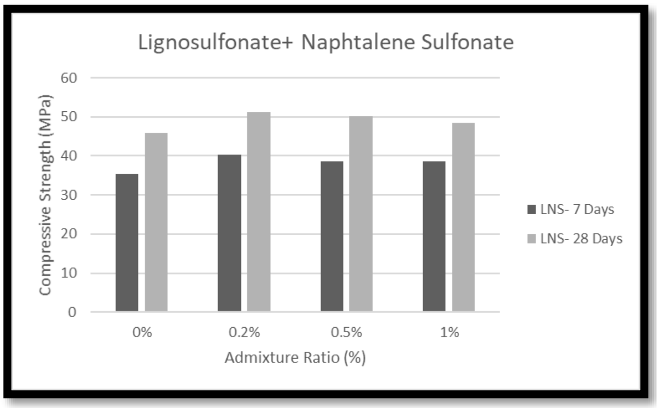


**Figure 3.3.** The compressive strength values of cement mortars prepared with lignosulphonate+ naphthalene sulfonate based admixture and aged for 7 and 28 days

The compressive strengths of the cement mortars prepared with lignosulphonate+ naphthalene sulfonate-based admixture and aged for 7 and 28 days were increased when compared to the reference sample. The strength values slightly decreased with the increasing admixture ratio. The highest compressive strength value was obtained as 51.33 MPa with a 0.2% admixture ratio when the strength of the reference sample was 45.9 MPa.

## 3.2. X-Ray Diffraction (XRD) Analysis

The crystalline structure of the cement mortars prepared with two different admixtures and aged for 28 days was determined by X-Ray diffraction (XRD) spectrometry. The XRD spectrum of the mortars prepared with naphthalene sulfonate-based admixtures is given in Figure 3.4. The peaks of ~20°, 26°, 37°, 40°, 60°, 70° in XRay diffraction spectrometer of the 28 days aged samples prepared by using 0.2%, 0.5%, and 1% admixtures are called quartz ($SiO_2$). ~35°, 45°, 50°, 55°, and 65° are called portlandite ($Ca(OH)_2$). These peaks are numbered at the top of the graph and are valid for all 4 spectrometers.

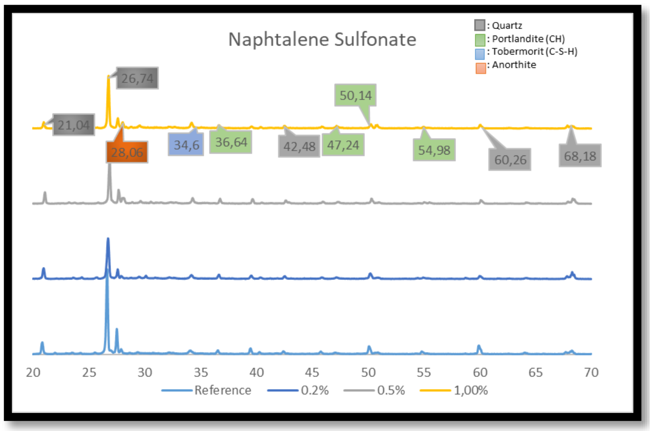


**Figure 3.4**. *28- day XRD spectrum of samples prepared by naphthalene sulfonate based admixtures*

Tobermorit is one of the C- S- H minerals. The intensity of the portlandite peak is the highest in the reference sample, but the peak intensity decreased with an increasing admixture ratio. [7-8]. The XRD spectrum of the mortars prepared with lignosulphonate+ naphthalene sulfonate-based admixtures are given in Figure 3.5.

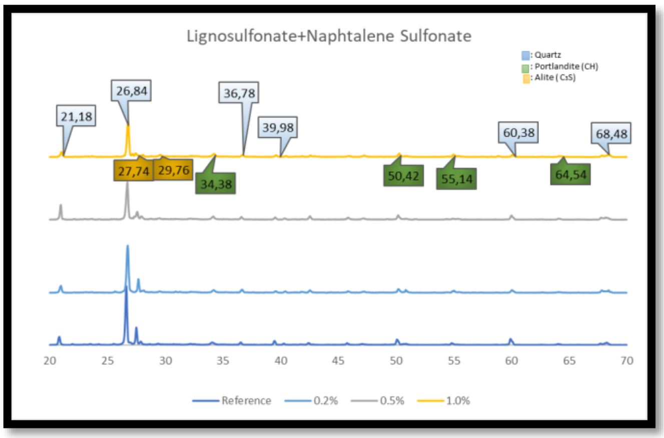


**Figure 3.5.** *28- day XRD spectrum of samples prepared by lignosulphonate+ naphthalene sulfonate based admixtures*

The presence of the alite peak indicates that not all of the cement particles come into contact with water and react [14]. The intensity of the portlandite peak is the highest in the reference sample, but the peak intensity decreased with increasing admixture ratio as seen in Figure 3.4.

## 3.3. Discussion

The flow rates and compressive strengths of the samples aged for 28 days increased as the admixture ratio increased (regardless of the admixture type) compared to the reference admixture. When the admixture ratio increased to %1, the compressive strength decreased in LNS. In the study of Torres et al., as the fluidity increased, a decrease in compressive strength was observed. In the studies of Collepardi et al. in 1998 and by Uyan et al. in 1996, it was observed that superplasticizer admixtures contribute to the processing of concrete and increase the compressive strength [9-11]. The highest compressive strengths of the mortars were obtained with the 0.5% admixture ratio for both lignosulphonate+ naphthalene sulfonate and naphthalene sulphonate admixtures as in the studies by Kandemir and Ekinci. In Figure 3.2 and Figure 3.3, it was determined that the optimum value of naphthalene sulfonate-based admixture was 0.5%, and increasing the admixture dose did not affect the compressive strength. Samples containing LNS admixtures have high compressive strength even at low admixture ratios [12-13]. The presence of non-hydrated cement components in the hardened concrete indicates insufficient reaction with cement and water. Although there are non-hydrated cement particles in all hardened concrete, it is important to increase the amount of non-hydrated cement with increasing admixture dosage. This thesis was also supported by the amount of portlandite (CH). The amount of CH decreased as the dosage increased [14].

## 4. Conclusions and Recommendations

The flow diameter of the mortars increased as the admixture ratio increased in the samples aged when compared to the reference sample as expected. The flow ratio values of the mortars prepared LNS and NS admixtures were close to each other. The highest flow rate was obtained with a 1% admixture ratio for the lignosulphonate+ naphthalene sulfonate-based admixture as 51.33 MPa. The compressive strengths of the mortars prepared with two different admixtures increased when the admixture ratios were increased to 0.5%, however, the strengths decreased for the 1% admixture ratio. The highest compressive strength was obtained as 49.94 MPa for naphthalene sulphonate-based admixture with a 0.5 % ratio and 51.33 MPa for lignosulphonate+ naphthalene sulfonate based admixture with a 0.2 % ratio at the end of the 28- day aging period. In some spectrums, an alite peak that was expected to transform into C-S-H gel was observed as a result of hydration reactions within 28 days. This shows that some cement particles remained unreacted [15]. As can be seen from XRD graphics and literature reviews, the intensity of the CH peak decreased as the amount of admixture increased.

## 5. Acknowledge

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