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The effect of lignosulfonates on concretes produced with cements of variable fineness and calcium aluminate content



Faculty of Architecture, İzmir University, Gürsel Aksel Bulvarı, No: 14, 35350, Üçkuyular, İzmir, Turkey

HIGHLIGHTS

• The highest viscosities were measured for A-, B-, A2-, and B2-type cements containing 0.8% LSm.

• The highest air content of 4.65% was observed in the B2-LSm mixture.

• The determined water reduction values for LSc were higher than those of other LSs.

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1. Introduction

1.1. Preliminary remarks

Lignosulfonates (LSs), or sulfonated lignins, are water-soluble anionic polyelectrolyte polymers that are generated as byproducts of wood-pulp processing using sulfite pulping [1]. Usually, delignification in sulfite pulping involves the acidic cleavage of ether bonds, which connect several constituents of lignin. The electrophilic carbocations produced during ether cleavage react with bisulfite ions (HSO₃) to produce sulfonates [2] in the following reactions:

$$\mathbf{R} - \mathbf{O} - \mathbf{R}' + \mathbf{H}^+ \to \mathbf{R}^+ + \mathbf{R}'\mathbf{O}\mathbf{H} \tag{1}$$

$$\mathbf{R}^{+} + \mathbf{HSO}_{3}^{-} \to \mathbf{R} - \mathbf{SO}_{3}\mathbf{H}$$
(2)

ABSTRACT

Lignosulfonates are water-soluble anionic polyelectrolyte polymers produced during industrial paper manufacturing. This study investigated concrete produced using lignosulfonates with various chemical contents at different concentrations. Therefore, concrete mixtures were produced using Ca-lignosulfonate (LSc), Na-lignosulfonate (LSn), Mg-lignosulfonate (LSm), and K-lignosulfonate (LSk) at concentrations of 0.4% and 0.8% by weight in the cement. Samples containing cements with different C₃A, C₃A + C₄AF, and Na₂O + K₂O contents and variable fineness were tested to determine a compatible combination of cement and lignosulfonates. Results revealed that LSc was suitable for use in cements with greater C₃A content and fineness and 0.8% was an appropriate usage concentration.

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A total of 50 million tons of lignin (LS and alkali lignin) produced worldwide annually in paper factories [3]. Fig. 1 demonstrates the process of sulfonation, which occurs on side chains rather than on aromatic rings, as in p-toluenesulfonic acid [4].

Various researchers have reported that LSs have high molecular weights and that their ability to be adsorbed increases with molecular weight [5,6]. Several positive counter-ion $(Ca^{2+}, Mg^{2+}, N^*, K^*, Zn^{2+}, etc.)$ complexes are formed with LS [7]. Differences in the LS chemical structure (e.g., free sugar content and counter-ion type), which depend on the production process, cause different interactions with the chemical components in cement. This also results in different behavior upon hydration among carboxyl groups adsorbed to Ca^{2+} ions in the chemical structure of cement and oxygen ions with hydroxyl groups [8]. LSs have a retarding effect on hydration (although it varies depending on the sugar content); however, the presence of large amounts of C_3A in the environment mitigates this effect [9].

Aluminates (C₃A and C₄AF) constitute approximately 20% of cement. The aluminate content is calculated using the formula $C_3A = 2.65A - 1.692F$. C_3A significantly contributes to the





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E-mail address: sahanarel@gmail.com

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Fig. 1. Generalized structure of lignosulfonates (LS) [4].

chemical durability of cement. It generates a large amount of heat during hydration and an excess volume of salt (ettringite or Candlot salt) by reacting with calcium sulfate, i.e., gypsum [10]. Aluminates also occur in the form $C_4AF = 3.043F$. The effects of C₄AF are moderate compared with those of C₃A [11]. Ettringite is the first stable hydration product that is formed by the reaction of C_3A , calcium sulfate (gypsum), and water [12,13]. When all gypsum is consumed and the concentration of sulfate ions becomes low, ettringite becomes unstable. The reaction of C_3A with sulfate ions is controlled in the presence of superplasticizers [14]. The needle-shaped particles of ettringite adsorb water in the environment and decrease the workability of the cement paste. Superplasticizers adhere to the newly formed ettringite and delay their formation into needle-like structures [12]. The incorporation of alkali materials (Na⁺, K⁺), $C_3A + C_4AF$, and SO_3^- into the chemical structure of cement, C₃A/CaSO₄ molar ratio, and fineness directly affects hydration and therefore the performance of superplasticizers [15]. A common solution for improving the mechanical performance of concrete is to increase the hydration rate and degree of hydration in cement [16]. Thus, temperature and cement fineness are additional factors that affect the hydration kinetics [17]. Cement fineness, especially at an early stage, improves the compressive strength of concrete in later years [18]. This improvement is considered to result from the high specific surface area of cement particles produced by the grinding process [19].

1.2. State of the art

The use of calcium lignosulfonate (LSc) at a ratio of 0.25 wt% in cement increases the 28-day compressive strength by 2%, whereas a ratio of less than 0.5% results in the maximum viscosity of the cement paste [4]. In cement paste admixture samples containing 4% LSc and 4% sodium lignosulfonate (LSn), very few needle-like ettringite particles were observed. The ettringite particles exhibited ovoid shapes [20]. Danner et al. [20] reported that LSs use increased compressive strength and determined that the highest concrete sample strength was achieved with a cement-LS content of 0.2 wt% in samples cured at various temperatures (50, 60, 70-80 °C) [21]. The setting duration for a cement paste containing 0.4% LS admixture and low C₃A content was determined to be 36% longer than that of cement paste with high C₃A content [22]. LSs are made from renewable materials, and every kilogram of LS used in concrete represents a reduction of 14 kg of CO₂. High-quality LS is easy to handle and does not exhibit formation of crystals, sludge, or insoluble material with changes in temperature. LS forms robust admixtures, and variations in the composition and quality of cement has minor effects on the performance of LS-based water-reducing admixtures [23]. Addition of LS increases the compressive strength of concrete, with maximum values achieved at LS concentrations of up to 0.2 wt% in cement at various temperatures. The addition of LS

in higher amounts results in a decrease in the compressive strength of the cement [21]. Different types of LS admixtures have functions ranging between set-retarding, water-reducing, and high-range water-reducing admixtures. LSs are suitable for concrete admixture formulations, particularly in areas with high ambient temperatures, where set retardation is needed [24]. The presence of LSn during cement hydration stabilizes and extends the duration of both the pre-induction stage and dormant stage and also stabilizes Ca(OH)₂ into period IV efficiently [25]. Peroxyacetic acid (PAA) oxidation and sulfomethylation are efficient methods for improving LSn performance. Improved waterreducing capabilities of modified LSn are attributed to the increases in molecular weight and sulfo-group content after modification [26]. In general, the principal explanation for rapid setting is that the use of LSc accelerates the reaction rates between C₃A and anhydrite at the initial stage of hydration. This generates abundant large ettringite crystals that do not hinder the subsequent hydration of cement [27]. The mechanical strength of mortar modified with ACL (an azo-LSc synthesized by the coupling of a diazonium salt made from p-aminobenzene sulfonic acid and LSc) is substantially higher than that used with LSc, particularly at concentrations higher than 0.5%. The air content of ACL-modified mortars is significantly lower than that of LSc-modified mortars, and the dispersion of ACL is greater than that of LSc at concentrations higher than 0.5% [28].

1.3. Significance of the research

This study is crucial for understanding how the reactivity and compatibility of LS changes with variations in chemical compositions according to the type of wood, the process used to obtain it, and the combination of these factors with variations in the fineness and chemical structure of cement. The chemical and physical structures of cement affect the form, process, and speed of hydration and are also crucially involved in the mode of operation and effects of chemical admixtures. Therefore, cements of different chemical and physical structures were used in this study. Variations in the chemical compositions of LSs also prevent the use of multiple admixtures in the same concrete. Therefore, this study investigated whether LS simultaneously acts as a set-retarder, a plasticizer, and an air-entrainment agent, which will help prevent the adverse effects caused by the use of admixtures of various chemical compositions to ensure the desired properties of a concrete. In addition, the properties that result from the use of different types of LS in concrete were investigated. From an environmental perspective, even a moderate increase in the use of LS produced as a by-product will contribute to the production of green concrete. Moreover, it will have a positive impact on the concrete production costs compared to the environmental damage and production costs associated with synthetic products used as concrete admixtures.

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