



Sodyum Silikat ile Düşük Oranda Çimento İçeren Karışımların Reolojik Özellikleri

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Öz

Bu deneysel çalışmada, farklı oranlarda hazırlanmış çimento-sodyum silikat (C-SS) karışımlarının reolojik özellikleri araştırılmıştır. Deneyler, 25 °C sıcaklıkta, toplam hacim üzerinden kütlece %3 ile %6 oranında çimento ilave edilerek, hacimce %30, %50 ve %70 sodyum silikat/çözelti (SS/S) oranlarına sahip karışımlar için gerçekleştirilmiştir. Reolojik özelliklerin belirlenmesine yönelik hazırlanan numuneler sabit hızda (100 rpm) karıştırılmıştır. Jelleşme deneylerinde reaktör olarak iki farklı çimento türü, Normal Portland Çimentosu (OPC) ve Ultra İnce Taneli Çimento (UFC) kullanılmıştır. Deneylerde kullanılan ve su camı olarak da bilinen sodyum silikat sıvı formdadır ve modülü 3'tür. OPC' nin Blaine değeri 350 m²/kg iken, UFC' nin Blaine değeri 900 m²/kg'dır. Daha yüksek Blaine değerine sahip olması nedeniyle, UFC-sodyum silikat karışımları, OPC-sodyum silikat karışımlarına kıyasla daha kısa jelleşme süresine sahiptir. Her iki çimento türü için de çimento oranı arttıkça jelleşme süresi azalmış; sodyum silikat oranı arttıkça ise jelleşme süresi artmıştır. Sinerez yüzdeleri, çimento oranı arttıkça azalmış; sodyum silikat oranının %50'ye kadar artmasıyla sinerez artmıştır ancak %50'den sonra azalma göstermiştir. Viskozite değerleri hem sodyum silikat oranının hem de çimento miktarının artmasıyla birlikte artmıştır. Bu deneysel çalışmada, literatüre kıyasla daha düşük çimento oranları kullanılarak sodyum silikat-çimento reaksiyonları sonucunda jelleşmenin gerçekleşebileceği gözlemlenmiştir. Çalışmanın bir diğer özgün yönü, silikat esaslı enjeksiyon karışımlarında reaktör olarak çimentonun da kullanılabilmesidir. Alternatif enjeksiyon malzemesi olarak kullanılacak bu karışımların reolojik özellikleri belirlenmiştir.

Anahtar kelimeler: Ultra ince taneli çimento, Enjeksiyon, Jelleşme süresi, Viskozite, Sinerez

*Yazışılan yazar



Rheological Properties of Lightly Cemented Sodium Silicate Grouts

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Abstract

In this experimental study, the rheological properties of cement-sodium silicate (C-SS) mixtures prepared at different ratios were investigated. The experiments were carried out by adding 3% to 6% cement by mass of the total volume to 30%, 50%, and 70% sodium silicate/solution (SS/S) mixing at 25 °C. The samples prepared for the rheological property determination experiments were mixed at a constant speed of 100 rpm. In the gelation experiments, two different cements, ordinary Portland cement (OPC) and ultra-fine cement (UFC) were used as reactants. Sodium silicate, also known as water glass, was used in the experiments in liquid form, and its modulus was 3. The Blaine value of OPC was 350 m²/kg, whereas that of UFC was 900 m²/kg. UFC-sodium silicate mixtures have faster gelation times than OPC-sodium silicate mixtures due to their high Blaine values. For both cements, the gelation time was shortened as the cement ratio increased, and the gelation time was extended as the sodium silicate ratio increased. The syneresis percentages decreased as the cement ratio increased, whereas the sodium silicate ratio increased up to 50% and decreased after 50%. Viscosity values increased with increasing sodium silicate ratio and cement amount. In this experimental study, gelation reactions could be achieved as a result of sodium silicate-cement reactions using lower cement ratios than those reported in the literature. Another unique aspect of the study is that cement can also be used as a reactant to prepare silicate grouts. The rheological properties of these mixtures, which can be used as alternative grout materials were determined.

Keywords: Ultrafine cement, Grouting, Gelation time, Viscosity, Syneresis

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1. Introduction

Soil improvement is one of the most important geotechnical engineering applications. Recent academic studies have widely preferred chemical injections because of their ease of application and effectiveness in fine-grained and low-permeability soils. Low-viscosity mixtures are injected into the ground, reducing its permeability and increasing its strength and carrying capacity [1].

Sodium silicate is frequently preferred as a chemical injection material because of its low viscosity, easy gelation control and economy [2]. The use of sodium silicate alone can cause negative effects, such as insufficient strength and early gelation [3]. For this reason, various reactants have been used in the literature to control the gelation of sodium silicate grouts. Cement derivatives are frequently used as reactants. Kazemian et al. investigated effect of the amount of cement on the viscosity and gelation time in cement-sodium silicate mixtures [3, 4]. They observed rapid gelation at high cement ratios. Avci et al. examined the permeability and strength properties of cement-sodium silicate grouts, emphasized that the cement ratio is an important parameter for gel stability [5].

The use of high-rate cement can lead to a rapid increase in the viscosity and failure to control the gelation time in field applications [6, 7]. Therefore, the study aimed to delay gelation time and control injection time by using low cement rates between 3% and 6% in sodium silicate grouts. The Blaine value of the ultrafine cement (UFC) was 900 m²/kg. Thus, even when used at a low rate, it can initiate a reaction because of the high pH effect [8, 9]. In addition, the effect of specific surface factors on the rheological properties were investigated using OPC in addition to UFC.

Not all cement particles are water-soluble. One of the most significant problems encountered in cement injection is the difficulty encountered in injecting into low-permeability soils such as fine sand and silt due to the large particle sizes. In this context, the most significant advantage of fine-grained cement over conventional Portland cement is its approximately ten-fold smaller particle size.

In this experimental study, sodium silicate/water ratios were determined as 30%, 50%, and 70% by volume. The cement was added by mass at a rate of 3% to 6% of the solution mixture volume. The rheological properties such as viscosity, gelation time, and syneresis of the grout systems were determined and evaluated. As a result of experimental studies, as an alternative to grout materials with high cement ratios in the literature; sodium silicate-UFC and sodium silicate-OPC mixtures provide a unique contribution in terms of having sufficient and controllable application time, low viscosity, and more economical and environmentally sustainable properties. In addition, in most studies in the literature, sodium silicate has been added to cement-water mixtures, and its performance has been evaluated. The use of cement as a reactant material is another unique aspect of this study.

2. Literature Review

Some prominent studies in the literature evaluating the engineering and gelation control of sodium silicate mixtures with cement and different reactant materials can be listed as follows:

Kazemian et al. conducted viscometer tests on mixtures prepared with different cement, sodium silicate, kaolinite, and water ratios. It was found that cement and sodium silicate were the most important determinants of viscosity. It has also been reported that high cement ratios cause rapid gelation [4]. Kazemian et al. investigated the effect of sodium silicate on cement-based mortar systems. In the study, viscosity and strength tests were performed on mixtures with different ratios. As the sodium silicate ratio increased, gel time decreased and viscosity increased [3]. Avci determined the rheological properties of sodium silicate-formamide mixtures such as gelation time, viscosity, and syneresis. The gelation time decreased with increasing the amount of reactant [9]. Zhang et al. tested the injection performance of cement/sodium silicate mortars at 1:1 and 1:2 ratios on rock cracks. They emphasized that the injection performance depends on the viscosity and crack aperture [2]. Zhu et al. studied the time-dependent viscosity behavior of cement-sodium silicate mixtures at different temperatures. Gelation times are shortened at high temperatures and viscosity

control becomes difficult at high temperatures [10]. Avci et al. performed injection applications on sandy soils using sodium silicate and fine-grained cement with different proportions. The strength and permeability decreased with the addition of sodium silicate to cement mixtures. Viscosity values increased with the increase in the sodium silicate additive ratio [5].

As a result, the cement ratio was generally kept between 10% and 55%, but this situation caused high viscosity and rapid gelation problems in field applications [2-5, 8, 11-15]. In this experimental study, mixtures with low viscosity and controllable gelation rates that can be used as soil injection materials were obtained using low cement content.

Studies in the literature generally initiate the C-S-H reaction using cement as the main binder injectable. In this context, sodium silicate was added to the mixtures, and its effects on strength and rheological properties were primarily investigated on samples. However, high viscosity and the inability to control gelation time pose significant obstacles to the effective use of such mixtures as injection materials.

In the experimental study, sodium silicate was selected as the main injection material, and cement types were used as reactants to initiate the chemical reactions. This approach constitutes one of the most important and unique aspects that distinguishes our study from existing studies. Furthermore, the low viscosity and controllable gelation time of the mixtures with determined rheological properties demonstrates their potential as injection materials compared to other studies.

3. Materials and Methods

3.1. Sodium silicate

Sodium silicate, also known as water glass, is a chemical material widely used in the construction industry and underground applications. The product coded EGENAT 3203, produced by Ege Kimya A.Ş. in Turkey, was used in the experimental study. The physical and chemical properties of sodium silicate are summarized in Table 1.

Table 1. Physical and chemical properties of sodium silicate (data from manufacturer)

Properties	Values
General Formula	Na_2SiO_3
Appearance	Clear liquid, colorless
Weight Ratio, $\text{SiO}_2/\text{Na}_2\text{O}$	3-3.3
Molar Ratio, $\text{SiO}_2/\text{Na}_2\text{O}$	3.1-3.4
Bome (at 20°C)	39-41
Density (at 20°C, gr/cm^3)	1.37-1.39
Na_2O (%)	8.5-9.5
SiO_2 (%)	26-28
Viscosity (at 20°C, cp)	75-300
pH (at 20°C)	11.0-12.5

3.2. Ordinary portland cement and ultrafine cement

In the experiments, CEM-I 42.5 R normal Portland cement (hereinafter referred to as OPC) produced by Bursa Cement Inc. (TR) and MasterRoc MP900 fine-grained cement (hereinafter referred to as UFC) supplied by BASF were used. The physical and chemical properties of the cement are summarized in Table 2.

Table 2. Physical and chemical properties of cements (data from manufacturer)

Properties		Values
SO ₃ (%)	Ordinary Portland Cement (CEM-I 42.5R)	<4
MgO (%)		<4
CaO (%)		<3
SiO ₂ (%)		<1
Cl ⁻ (%)		<0.1
Density (g/cm ³)		3.10-3.15
Blaine (m ² /kg)		350-370
Insoluble Residue (%)		<5
LOI (%)		<5
Appearance		Ultrafine Cement (MasterRoc)
Colour	Gray	
CaO (%)	<3	
CaCO ₃ (%)	<3	

Table 2. (Continue) Physical and chemical properties of cements (data from manufacturer)

Properties		Values
MgO (%)	Ultrafine Cement (MasterRoc)	<3
CaSO ₄ (%)		<5
pH (at 20 °C)		12-13
Density (g/cm ³)		3.05
Blaine (m ² /kg)		>900

3.3. Rheological properties

3.3.1. Gelation time

Gel time is used to express the time until the injection mixtures transition from liquid to gel (semi-solid) state. Parameters such as reactant ratio, sodium silicate content, and ambient temperature affect gel time.

Meng et al. reported that high water/cement ratios prolonged gel times in their study on gel times with different water/cement ratios [15]. Similarly, Lee and Jeon stated that gel times were shortened with increasing cement amount in their study on sodium silicate mixtures with recycled material additives [16]. Govindan showed that gel times were sensitive to the test method and initial temperature of the mixture [17].

3.3.2. Syneresis

Syneresis is defined as the volume contraction that occurs when a mixture in gel form expels water as a result of reactions over time. Shrinkage and water accumulation on the surface occur with volume contraction. It is an important parameter in terms of gel stability and long-term durability.

Syneresis is the main indicator of the physical stability of the mixture. As the sodium silicate ratio increases, the tendency for syneresis increases and decreases as the cement ratio increases [7].

3.3.3. Viscosity

The viscosity is a measure of the resistance of a liquid to flow. The value is expressed in Pa.s or cp (centipoise) units in different unit systems. Viscosity is an important parameter in terms of ease of injection, spreading and gel time control in injection applications. Although low viscosity provides an advantage in filling ground voids, high viscosity has a spreading-reducing effect. In these aspects, viscosity is one of the basic rheological parameters affecting the success of injection [2, 4].

3.4. The chemical reaction

As a result of mixing cement with water, a hydration reaction occurs and calcium hydroxide is produced (Eq. 1). Due to the small amount of cement, there is limited C-S-H production. A small amount of calcium hydroxide is formed.

The sodium silicate ions are dissociated into sodium silicate ions when sodium silicate is mixed with water. (Eq. 2). By adding cement to sodium silicate-water solutions that are basic and stable, the pH of the environment increases and excess silicate ions undergo a polycondensation reaction within themselves and gelation occurs. This reaction produces an amorphous silica gel is formed (Eq. 3). Some water is also released by the reaction [18]. The low cement ratio ensured a slow increase in the pH and controlled gelation.



4. Results and Discussions

In the experimental studies, 3 different sodium silicate-water solution ratios were used. The solution mixtures were mixed according to the percentages specified by volume in Table 3. The rheological properties of the ratios formed by mixing 3%, 4%, and 5% Ultrafine Cement (UFC) and 4%, 5%, and 6% OPC for the total solution volume were determined. The test procedure was as follows:

The cement was mixed in the specified proportions at 100 rpm for 3 minutes. The relevant amount of sodium silicate was added to the solutions and the mixture was mixed at a constant speed until gelation began. The gelation time is defined as the interval between the moment the mixture turns from liquid to semi-solid (gel). At this moment, the viscosity of the mixture begins to increase and it resists flow. Completed gelation samples were kept in closed containers to determine the percentage of syneresis. In the viscosity tests, the initial viscosities of the samples mixed in the specified ratios were measured using a rheometer device during the gelation period. The measurements were completed before gelation.

Table 3. Rheological properties of different sodium silicate-cement mixtures

Water (%)	Sodium Silicate (%)	Cement Type	Cement content (%)	Gelation Time (sec)	Syneresis (%)	Initial Viscosity (cp)	
70	30	Ultrafine Cement (MasterRoc MP900)	3	352	11.06	1.87	
50	50			942	14.19	4.45	
30	70			4120	12.72	6.65	
70	30		Ultrafine Cement (MasterRoc MP900)	4	200	8.50	2.01
50	50				451	10.74	4.73
30	70				1037	9.95	6.83
70	30			5	146	6.81	2.44
50	50				176	8.77	4.83
30	70				715	8.33	7.09
70	30	Ordinary Portland Cement (CEM-I)	4	644	6.39	1.96	
50	50			1005	9.01	4.44	
30	70			3532	7.63	6.7	
70	30		5	476	5.21	2.21	
50	50			620	6.91	4.69	
30	70			1666	6.60	6.89	
70	30		6	365	3.63	2.55	
50	50			530	5.24	4.89	
30	70			1213	5.28	7.05	

4.1. Gelation time

Gelation times were determined by continuously mixing with a mechanical mixer at 100 rpm to prevent cement particles from settling into the solution. The experiments were performed on 100 ml mixtures. In preliminary experiments, it was observed that if continuous mixing was not performed, the cement particles would settle over time and the bottom of the container would gel earlier. In laboratory and field applications, injection applications should not exceed 100 rpm. Otherwise, early gelation problems and strength losses are anticipated.

In the experiments, two types of reactants (cement) were used: OPC and UFC. The most important difference between these two types of cement is the specific surface. UFC has a higher specific surface area compared to OPC. This allows the gelation time to be more controllable. In addition, it is possible to start the same chemical reaction and gelation process using less material.

The gelation times of the mixtures prepared with UFC varied between 146 seconds and 4120 seconds, and those prepared with OPC varied between 365 and 3532 seconds (Figures 1 and 2). As the amount of cement increases, the gelation time decreases as an exponential function. With increasing sodium silicate percentage at the same UFC and OPC concentrations, the gelation time increases as an exponential function. The R^2 values of the trend lines in Figure 1 and Figure 2 are greater than 0.93. The gelation times of the UFC mixtures at the same ratios are shorter compared to OPC.

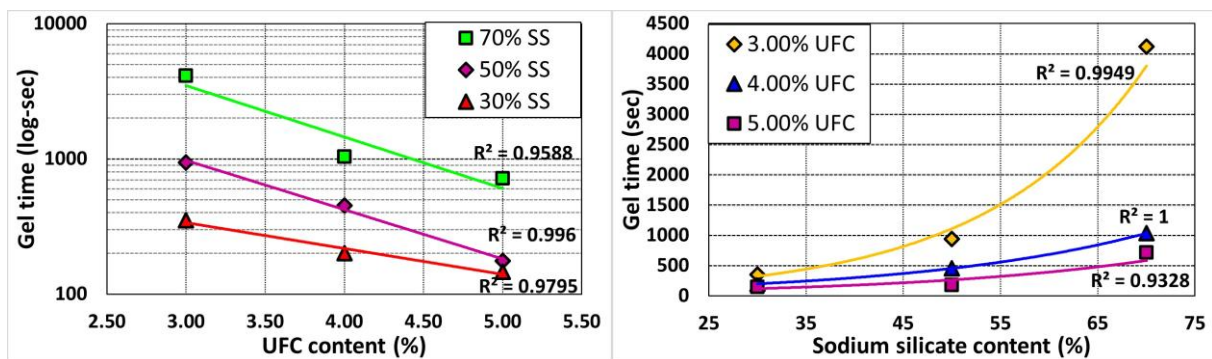


Figure 1. Variation in gel time at different cement (UFC) and sodium silicate ratios

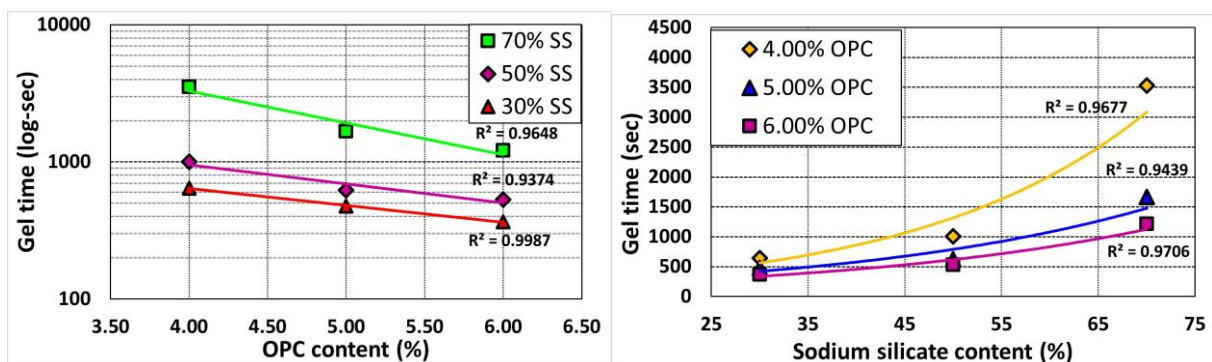


Figure 2. Variation in gel time at different cement (OPC) and sodium silicate ratios

4.2. Syneresis

The mixtures that completed the gelation period were kept in closed containers for 180 days and syneresis measurements were performed. The final measurement results for the different mixtures are shown in Figure 3 and Figure 4, respectively. The percentage of syneresis was calculated by dividing the volume of water released by the shrinkage of the samples by the initial volume of the sample.

The syneresis percentages of mixtures containing UFC varied between 6.81% and 14.19%, whereas that of mixtures containing OPC varied between 3.63% and 9.01%. The syneresis percentages decrease exponentially with the increase in the amount of UFC and OPC content. The syneresis percentages of both the UFC and OPC mixtures used at the same rate increased up to 50% sodium silicate and then decrease. The R^2 values of these trend lines expressed by polynomial equations are equal to 1 for all rates in both graphs.

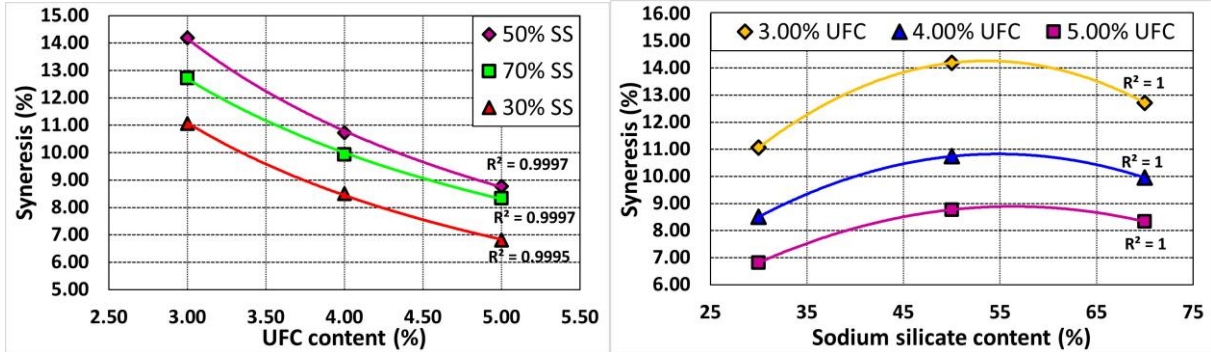


Figure 3. Syneresis variations at different cement (UFC) and sodium silicate ratios

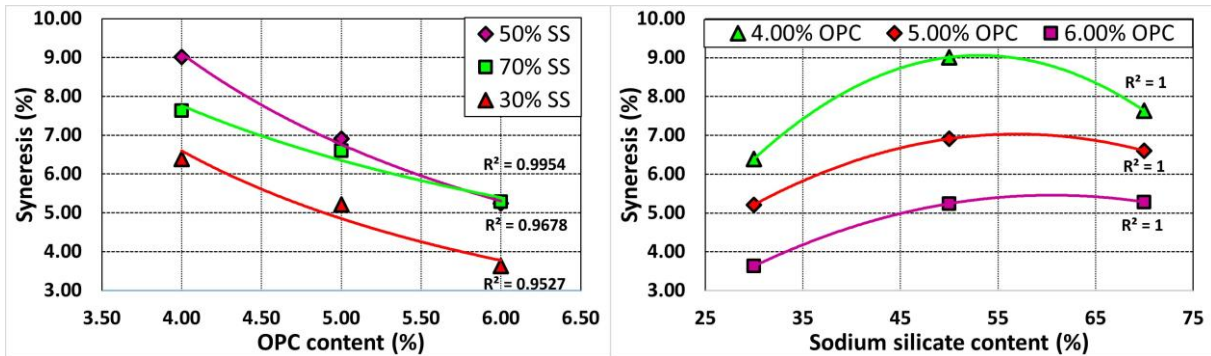


Figure 4. Syneresis variations at different cement (OPC) and sodium silicate ratios

4.3. Viscosity

The initial viscosity values of the mixtures in the experimental studies were determined with a Brookfield DV3T model rheometer device at 100 rpm rotation speed and 25 ° C. The duration of each measurement has been specified as 1 minute. The relationship between the sodium silicate content, reactant (cement) ratio and the viscosity values is shown in Figures 5 and 6.

In mixtures containing UFC, the viscosity values ranged from 1.87 to 7.09 cp, and in mixtures containing OPC, the viscosity values ranged from 1.96 to 7.05 cp. Viscosity values increased linearly with increasing sodium silicate percentage. In mixtures containing both UFC and OPC, viscosity values increased linearly with increasing cement ratio.

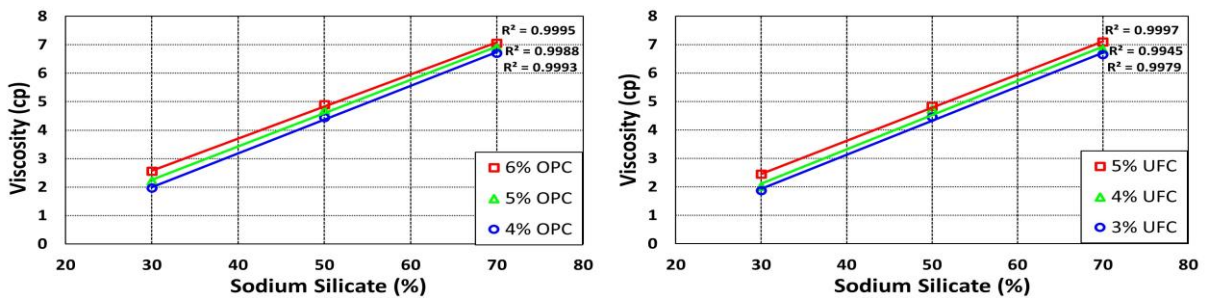


Figure 5. Variation in the viscosity at different sodium silicate ratios

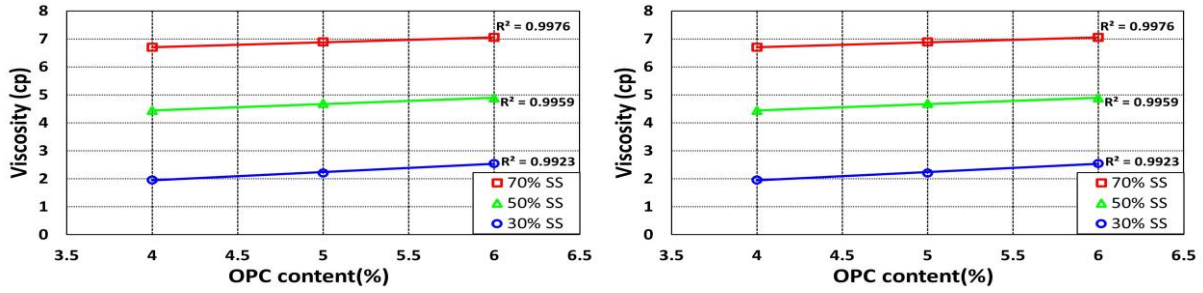


Figure 6. Variation in the viscosity at different OPC and UFC contents

The parameters, equations and R^2 values of the trend lines for all figures are presented in Table 4.

Table 4. E quations of trendlines

Y-axis	X-axis (unit)	Parameter of the Trendline	Equation	R^2
Gel time (sec)	UFC content (%)	70% SS	$y=48177e^{-0.876x}$	0.9588
		50% SS	$y=12069e^{-0.839x}$	0.9960
		30% SS	$y=1263.8e^{-0.44x}$	0.9795
	Sodium silicate (%)	3% UFC	$y=51.253e^{0.0615x}$	0.9949
		4% UFC	$y=58.018e^{0.0411x}$	1.0000
		5% UFC	$y=36.22e^{0.0397x}$	0.9328
OPC content (%)	70% SS	$y=27856e^{-0.534x}$	0.9648	
	50% SS	$y=3422.4e^{-0.32x}$	0.9374	
	30% SS	$y=19926e^{-0.284x}$	0.9987	
Sodium silicate (%)	4% OPC	$y=156.95e^{0.0425x}$	0.9677	
	5% OPC	$y=164.87e^{0.0313x}$	0.9439	
	6% OPC	$y=137.46e^{0.03x}$	0.9706	
Syneresis (%)	UFC content (%)	50% SS	$y=39.909x^{-0.943}$	0.9997
		70% SS	$y=31.588x^{-0.83}$	0.9997
		30% SS	$y=31.417x^{-0.948}$	0.9995
	Sodium silicate (%)	3% UFC	$y=-0.057x^2+0.6165x-2.26$	1.0000
		4% UFC	$y=-0.038x^2+0.415x-0.5412$	1.0000
		5% UFC	$y=-0.03x^2+0.338x-0.63$	1.0000
	OPC content (%)	50% SS	$y=57.616x^{-1.331}$	0.9954
		70% SS	$y=26.974x^{-0.898}$	0.9678
		30% SS	$y=44.509x^{-1.377}$	0.9527
Sodium silicate (%)	4% OPC	$y=-0.005x^2+0.531x-5.04$	1.0000	
	5% OPC	$y=-0.0025x^2+0.286x-1.1087$	1.0000	
	6% OPC	$y=-0.002x^2+0.2375x-1.7288$	1.0000	
Viscosity (cp)	Sodium silicate (%)	6% OPC	$y=0.1125x-0.795$	0.9995
		5% OPC	$y=0.117x-1.2533$	0.9988
		4% OPC	$y=0.1185x-1.5583$	0.9993
	OPC content (%)	5% UFC	$y=0.1163x-1.0258$	0.9997
		4% UFC	$y=0.1205x-1.5017$	0.9945
		3% UFC	$y=0.1195x-1.6517$	0.9979
UFC content (%)	70% SS	$y=0.175x+6.005$	0.9976	
	50% SS	$y=0.225x+3.5483$	0.9959	
	30% SS	$y=0.295x+0.765$	0.9923	
UFC content (%)	70% SS	$y=0.22x+5.9767$	0.9891	
	50% SS	$y=0.19x+3.91$	0.9304	
	30% SS	$y=0.285x+0.9667$	0.9206	

5. Conclusion

In this experimental study, which was conducted to determine the rheological properties of the mixtures made by adding reactants (UFC and OPC) to sodium silicate-water mixtures at low rates compared to the literature, the following basic findings were obtained:

- Constant and low speed mixing is necessary to ensure gelation, syneresis, and viscosity control and to accurately determine the rheological properties.
- Gelation times were shortened with increasing cement amount. Gelation times were extended with increasing sodium silicate amount in mixtures containing the same cement amount (both UFC and OPC). The increasing and decreasing functions are expressed as exponential trend lines.
- The gelation time of UFC in the same amount is shorter than that of OPC. This is because UFC has a higher specific surface area than OPC.
- The syneresis percentages of the mixtures containing UFC and OPC decreased with the increase in the amount of cement. This decrease is expressed as an exponential trend line. In the mixtures made with the same amount of cement, the sodium silicate content increased up to 50% and then decreased polynomially.
- Viscosity values increased with increasing sodium silicate amount and cement ratio. These increases are expressed as linear functions. Increasing cement ratio increased viscosity values less compared to increasing sodium silicate amount.

In this experimental study, gelation reactions could be achieved as a result of sodium silicate-cement reactions using lower cement ratios than those reported in the literature. Another unique aspect of the study is that cement can also be used as a reactant for silicate injection. As a recommendation, laboratory tests should be conducted on these mixtures, the rheological properties of which have been determined, to investigate their suitability for injection into soils. Future studies should start with coarse sand and gradually working down to fine sand sizes.

6. Author contributions

Author contributions to the study are as follows:

Author 1: design, literature review, experimental study and writing; Author 2: idea and literature review; Author 3: writing and experimental study.

7. Ethics committee approval and conflict of interest

Ethics committee permission is not required for the prepared article. There is no conflict of interest with any person/institution in the prepared article.

8. Ethical Statement Regarding the Use of Artificial Intelligence

During the preparation of this manuscript, the writing assistance tool ‘Trinka’ was used solely for limited linguistic editing; all scientific content, analyses, and conclusions remain entirely the responsibility of the authors.

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