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# Gelation and Consolidation Characteristics of Cement-Sodium Silicate Grout within Water

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**Abstract:** Cement-sodium silicate grout (CSG) is now widely adopted in water plugging engineering to prevent water inrush disasters, and the gelation and consolidation characteristics of CSG in water environment significantly affect the grouting effect. To obtain an in-depth understanding of the performance of CSG in water, the CSGs with different water-cement ratios and volume ratios were tested, and the gelation properties, the deposition characteristics and the diffusion process of CSG within water were examined. The compressive strength and microstructure of CSG consolidation formed in air and water were also analyzed comparatively. The test results indicate that the CSG consolidation exhibits obvious stratification and segregation phenomena in water, which can be divided into three layers with different substance compositions. The compressive strength of CSG consolidation formed in water is much lower than that of the grout consolidation formed in air, indicating that the water environment has a significant weakening effect on the consolidation properties of CSG. The hydration products and microstructure of CSG consolidation in water change clearly due to the dilution effect of water.

**Keywords:** cement-sodium silicate grout; gelation property; grout consolidation; water environment; compressive strength; microstructure

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## 1. Introduction

During the construction of tunnel and underground engineering in water-rich regions, water inrush disasters occur frequently due to the exposure of different water-bearing and water-conducting structures, such as faults and karst caves [1–3]. At present, grouting is one of the most common technologies to deal with water inrush disasters [4–6], and the effect of grouting greatly depends on the performances of grouting materials. Generally, cement-sodium silicate grout (CSG), as a mixture of cement grout and sodium silicate solution in certain proportions, has the typical characteristics of fast gelling and relatively high strength [7–11]. Thus, it is widely used to plug water inrush channels and to reinforce water-rich weak strata in engineering practices [12–14].

To date, extensive studies on the CSG have been carried out, mainly focusing on the influences of the modulus and dosage of sodium silicate solution, the water-cement ratio of cement grout, and the category and dosage of admixtures on the physical and mechanical properties of CSG and its consolidation [15–25]. The properties of the CSG consolidation are crucial for evaluating the applicability and effectiveness of the CSG in engineering applications. Most previous studies have taken the CSG consolidation formed in air as the research object. However, in actual grouting engineering, the CSG injected into strata usually solidifies in the water-rich environment. In addition, the present double-fluid grouting technology cannot ensure the effective mixing and immediate consolidation of CSG after the two fluid components flow out of the grouting pipes, Thereby, the grout can easily be scoured and diluted by the groundwater, leading to low retention rates and weak consolidation strengths, which reduces the water plugging effect of grouting to a great extent [26]. Moreover, there is no effective adjustment method to guide the mixture ratio of CSG when entering water. Therefore, it is necessary to perform in-depth research on the gelation and consolidation characteristics of CSG in the water environment to provide more effective references for the application of CSG in engineering.

At present, there are few studies on the gelation and consolidation characteristics of CSG in the water environment, and no standard test method has been found through previous studies. However, the underwater consolidation molding test method of underwater non-dispersed concrete has formed the China industry standard GB/T 37990 (2019) [27], which provides a new experimental idea for this study. The diffusion process and compressive strength of underwater non-dispersible concrete grout can be obtained by this method. Therefore, this study performed the test through a self-processed underwater testing apparatus on the basis of referring to the test method stipulated in the above standard.

In addition, the CSG is a kind of Bingham fluid with viscosity time-varying [28–30], and the volume ratio between cement grout and sodium silicate solution and the residence time in the grouting pipe will affect whether the grout solidifies when entering water. Therefore, the CSGs with different water-cement ratios of cement grout, different volume ratios between the cement grout and sodium silicate solution and different mixing time were prepared to investigate the gelation properties, the diffusion process and the consolidation strength characteristics of CSGs in water. The hydration products and microstructure of CSG consolidation were also analyzed using an X-ray diffractometer (XRD) and scanning electron microscope (SEM). The main findings of this study are expected to provide guidance for the selection and preparation of CSGs in water plugging engineering.

## 2. Experimental Material and Scheme

### 2.1. Experimental Material

In this study, the ordinary Portland cement (OPC) with a strength grade of #42.5 and a density of 3.15 g/cm$^3$, which was produced by the China Resources Cement (Guigang) Company Limited, was used to prepare cement grout, and its chemical composition provided by the manufacturer is listed in Table 1. To prepare the CSGs, the commercial sodium silicate solution with a density of 1.32 g/cm$^3$, a Baume degree of 35, a modulus of 3.3 and a solid content of 30% was adopted.

**Table 1.** Chemical compositions of cement.

| $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | Loss on Ignition |
|---|---|---|---|---|---|---|
| 22.62 | 7.13 | 3.45 | 58.73 | 4.16 | 2.8 | 1.11 |

### 2.2. Experimental Scheme

The CSG was mixed by cement grout and sodium silicate solution, and the performance of CSG was controlled primarily by the water-cement ratio of cement grout (denoted by *W/C*) and the volume ratio between the cement grout and sodium silicate solution (denoted by *VR*). In this study, the values of *W/C* were set as 0.8 and 1.0, respectively, and the values of *VR* were set as 1, 3, and 5, respectively, which corresponds to the actual situation of engineering. For the convenience of assessment and comparison, CSG with a uniform volume of 4000 mL was prepared for each preparation condition, and the related mixing proportions are listed in Table 2.

In the table, the symbol $m_{cs}$ denotes the mass of cement in the cement grout; $m_{ss}$ denotes the content of solids in the sodium silicate solution; $v_c$ and vs. represent the volumes of cement grout and sodium silicate solution, respectively; $m_{wcs}$ and $m_{scs}$ indicate the masses of water and solids in the CSG, respectively; $m_{ss}/m_{cs}$ is the mass ratio of solid contents in the sodium silicate solution and the cement grout, which ranges from 8.9 to

52.5%; and $W_{cs}/C_{cs}$ denotes the actual water-cement ratio in the CSG, which is changed due to the mixing of cement grout and sodium silicate solution.

**Table 2.** Mixing proportions of CSG in different preparation conditions.

| **No.** | ***VR*** | **Cement Grout** | | | **Sodium Silicate Solution** | | **CSG** | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | $W/C$ (-) | $m_{cs}$ (g) | $v_c$ (mL) | $m_{ss}$ (g) | $v_s$ (mL) | $m_{wcs}$ (g) | $m_{scs}$ (g) | $m_{ss}/m_{cs}$ (%) | $W_{cs}/C_{cs}$ (-) |
| 1-1 | 1 | 1.0 | 1508 | 2000 | 792 | 2000 | 3356 | 2300 | 52.5 | 2.23 |
| 1-2 | | 0.8 | 1789 | 2000 | 792 | 2000 | 3279 | 2581 | 44.3 | 1.83 |
| 2-1 | 3 | 1.0 | 2262 | 3000 | 396 | 1000 | 3186 | 2658 | 17.5 | 1.41 |
| 2-2 | | 0.8 | 2683 | 3000 | 396 | 1000 | 3071 | 3079 | 14.8 | 1.14 |
| 3-1 | 5 | 1.0 | 2511 | 3333 | 265 | 667 | 3130 | 2776 | 10.6 | 1.25 |
| 3-2 | | 0.8 | 2979 | 3333 | 265 | 667 | 3002 | 3244 | 8.9 | 1.01 |

### *2.3. Experimental Method*

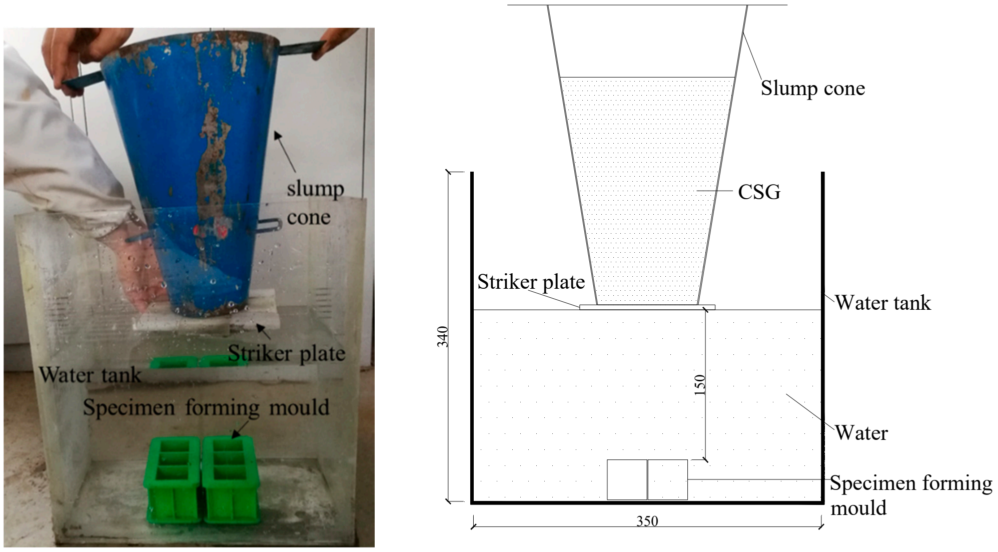
In this study, laboratory tests were conducted to investigate the gelation time of CSG in the air, the diffusion process of CSG in the water and the strength properties and microstructure characteristics of grout consolidation formed in the water. According to the China building materials industry standard of JC/T 2536 (2019) [31], the gelation time of CSG was measured through the pouring method, in which CSG with a volume of 200 mL was poured from one cup to another repeatedly until the grout lost its fluidity. The duration of this process represented the gelation time of grout.

The observation of CSG diffusion and the formation of CSG consolidation in the water were performed using a self-processed underwater testing apparatus in reference to the standard of GB/T 37990 [27], as shown in Figure 1. This apparatus was mainly composed of a slump cone, a water tank, a striker plate and a certain number of specimen molds. The slump cone served as the container of the prepared CSG, with an upper diameter of 200 mm, lower diameter of 100 mm and height of 300 mm. The water tank was used to simulate the underwater environment, and its dimensions were 350 × 245 × 340 mm (length × width × height). The cubic plastic molds, with a uniform internal side length of 40 mm, were placed at the bottom of the tank and used for specimen formation in the water.

During the tests, the specimen molds were first fixed on the bottom surface of the water tank. Then, water was added into the tank until the surface was 150 mm away from the top of the specimen molds. Second, the inverted slump cone was moved to the central position of the water tank, and the bottom of the slump cone was closed tightly with the striker plate. At the same time, the CSG was prepared according to the mixing proportions listed in Table 2, and the mixed cement grout and sodium silicate solution were stirred by a high-speed blender until the designed time (the initial mixing time) was reached. In this study, the stirring rate was approximately 600 r/min to ensure the mixing uniformity of two components, and the initial mixing time was set as 5 s for most test conditions. Finally, the stirred CSG was poured into the slump cone, and the striker plate was removed quickly to let the CSG flow into the water.

The diffusion process of CSG in water was recorded by a high-definition camera, and the deposition and stratification status of grout after half an hour were observed and measured using a camera and a steel ruler. Then, the specimen molds filled with deposited grout were taken out from the water tank, scraped flat with a scraper and placed into water at a temperature of (20 ± 1) °C for 48 h for demolding. After that, the demolded consolidation specimens were again placed into water at the same temperature for curing until the specified ages were reached. The consolidation specimens formed in water with the side length of 40 mm were cured for 3, 7, 28 and 140 days, and then the uniaxial compression tests were carried out according to the standard of JC/T 2536 (2019) [31] and GB/T 17671 (1999) [32] to investigate the compressive strength of CSG consolidation.

The compressive strength was determined by the maximum load corresponding to the time of specimen failure using an automatic compression machine at the loading rate of 2.4 KN/s. Then, XRD and SEM tests were performed to examine its hydration products and microstructures. For comparison, the consolidation specimens formed in air with the same mixing proportions, stirring process and curing method were also tested in this study.



(**a**) (**b**)

**Figure 1.** Testing apparatus of CSG in water. (**a**) Photo of test apparatus; (**b**) schematic diagram of apparatus.

## 3. Test Results and Analyses

### *3.1. Gelation Time of CSG*

The gelation time of CSG refers to the duration from the mixing of cement grout and sodium silicate solution to the grout's loss of fluidity. The gelation time, considering different mixing proportions of CSG in the air, is shown in Figure 2. The results indicate that the gelation time of CSG in the air shortened with the decrease of the mass ratio between the solid contents in the sodium silicate solution and the cement grout ($m_{ss}/m_{cs}$). For the CSG with the same mixing volume ratio between cement grout and sodium silicate solution ($VR$), the gelation time decreased with the decrease of the water-cement ratio of cement grout ($W/C$). The rapid gelation of CSG was mainly caused by the reaction between the sodium silicate and the calcium hydroxide generated from the hydration reaction of tricalcium silicate in cement. During this process, a certain amount of hydrated calcium silicate gel was generated. With the continuous generation of hydrated calcium silicate and other gelling substances and the ongoing cross-linking effect, the CSG was eventually cemented as a whole. A larger value of $m_{ss}/m_{cs}$ indicates that there were fewer cement particles per unit volume, the small amount of calcium hydroxide generated by its hydration was not conducive to the gelation rate of CSG.

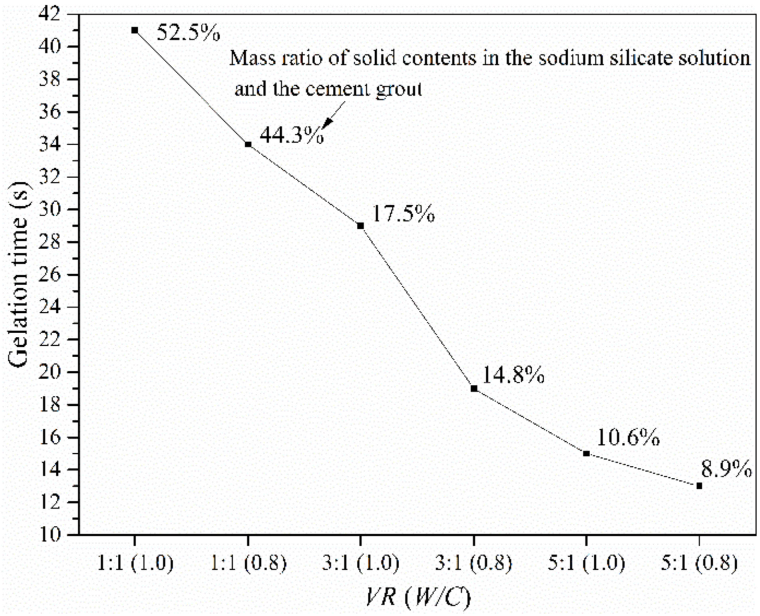


**Figure 2.** Gelation time of CSG with different mixing proportions in the air.

### 3.2. *Diffusion and Gelation Characteristics of CSG in Water*

The CSG rapidly diffused after it was poured into the water environment, and its diffusion process was greatly affected by the mixing proportions of grout and the initial mixing time. As mentioned above, the initial mixing time was set as 5 s, since the CSGs with different mixing proportions in this study are all in a flowing state at this time (see Figure 2).

Figure 3 shows the diffusion process of CSG in water (taking the case of *VR* = 1 and *W*/*C* = 1 as an example). The grout diffused throughout the water tank within a few seconds, indicating that the CSG was incapable of consolidating quickly in water for resisting the erosion and dilution effects of water. In addition, it was found that the change of mixing proportions of CSG had no clear influence on the diffusion process.

**Figure 3.** Diffusion process of CSG in water.

The CSG started to gel after 12~15 min in the water, and the gelling time was shortened with the increase of the *VR*, indicating that the quick-setting characteristic of CSG was clearly weakened in the water-rich environment. The stratification and segregation phenomena were observed after the CSG gelled in the water, as shown in Figure 4. After gelation and stratification, the CSG could be divided into three layers, including the suspension layer, the water-retaining soft gel layer and the sedimentary consolidation layer. The suspension layer was a mixture containing a number of solid particles in the solution. The water-retaining soft gel layer was mainly composed of calcium silicate hydrate gel that contained a large amount of water and a small number of cement particles. With the increase of the *VR* of the CSG, the content of the calcium silicate hydrate in the water-retaining soft gel decreased, while the water content increased. Consequently, the calcium silicate hydrate gel became soft. The sedimentary consolidation layer was mainly composed of hydrated calcium silicate gel with a large number of cement particles, and the content of cement particles increased gradually with the sedimentation depth. Consequently, the sedimentary consolidation layer became progressively denser. Under the action of gravity, the cement particles sedimented to the bottom, and the content of cement particles increased gradually from the top to the bottom. During the sedimentation process, the newly generated hydrated calcium silicate gel filled the space between cement particles and, to some extent, prevented the settling of cement particles.

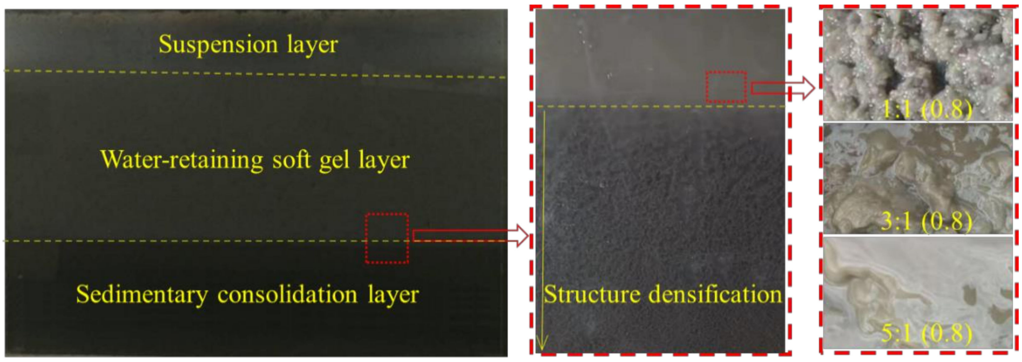


**Figure 4.** Deposition and stratification characteristics of CSG in water.

The deposition of CSG exhibited an obvious stratification phenomenon, consequently affecting the physical and mechanical properties of grout consolidation. To further study the stratification characteristics after grout deposition and gelation, the thicknesses of all layers were measured and analyzed, considering the influences of different grout mixing ratios, and the test results are shown in Figure 5. It can be found that the thickness of the sedimentary consolidation layer increased gradually with the *VR*, which was mainly caused by the large mass difference of cement in the CSG. The thickness of water-retaining soft gel layer reached its maximum value when the *VR* was 1:1, and it was approximately twice of those in the cases of *VR* = 3:1 and 5:1. This is mainly due to the fact that the sodium silicate solution is easily soluble in the water. The concentration of sodium silicate in the water is largest when the *VR* is 1:1, and it is helpful in promoting the reaction between the silicate ions and the calcium ions to form calcium silicate hydrate.

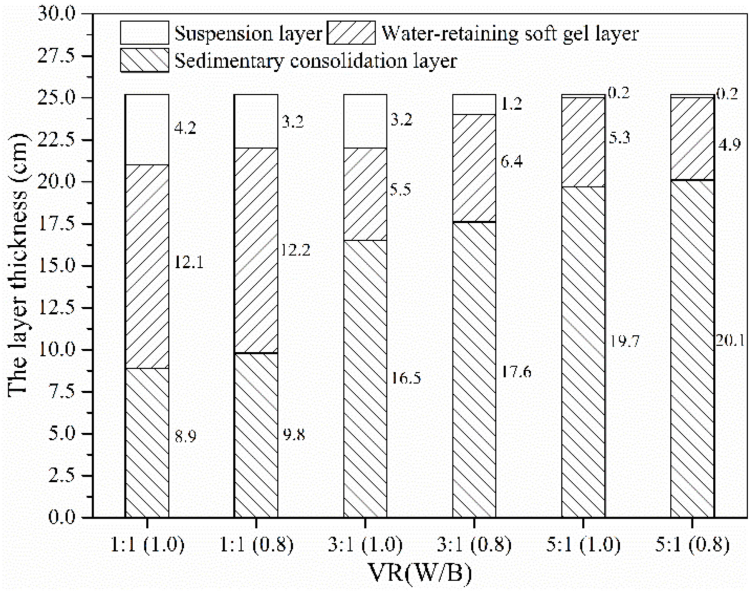


**Figure 5.** Deposition thicknesses of different layers with varying mixing proportions of CSG.

CSG is a generalized Bingham fluid with time-dependent viscosity [27–29], and the change of grout viscosity with the initial mixing time (the initial stirring time) will affect the gelation and consolidation performances of grout in the water. On the consideration of the gelation time, the CSG with a $W/C$ of 1 and a $VR$ value of 1:1 was selected to study the influence of initial mixing time, due to that the gelation time corresponding to this mixing proportion is longest. The deposition thicknesses of CSG were analyzed by controlling the initial mixing time as 5 s 15 s and 25 s, respectively, with the test results shown in Figure 6. The extension of the initial mixing time led to the decrease of the thicknesses of the suspension layer and sedimentary consolidation layer, and resulted in the increase of the thickness of the water-retaining soft gel layer. It is speculated that the extension of initial mixing time increases the reaction speed between the calcium ions generated from cement hydration and the silicate ions, thereby improving the overall viscosity of grout within the water, which consequently promotes the sedimentation of CSG in the water to a certain extent. The macroscopic representation of this is that the thickness of the sedimentary consolidation layer, containing a large amount of cement, becomes thinner with the extension of initial mixing time. The test phenomenon also indicates that the initial mixing time of 25 s has no significant effect on the diffusion of grout in the water.

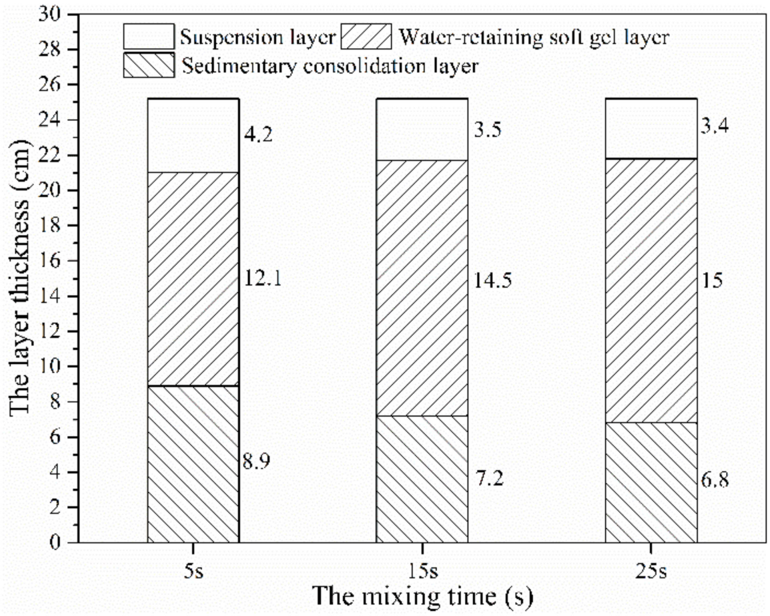


**Figure 6.** Deposition thicknesses of CSG in water with different initial mixing times.

### 3.3. Strength Characteristics of CSG Consolidation in Water

The previous studies and related standards have mainly focused on the strength of CSG consolidation formed in air. However, the CSG injected into the water-rich stratum is usually consolidated after it is washed and diluted by water in practical engineering, and the strength characteristic of grout consolidation formed in air is unapplicable for evaluating the grout performance. In this study, uniaxial compression tests on CSG consolidation specimens formed in water and air at different curing ages (the initial mixing time is 5 s) were carried out to investigate the strength characteristics of grout consolidation and the influences of water environment, with the results shown in Figure 7. Note that the black and white legends in the figure represent the strength of CSG consolidation formed in air, the colored legends denote the strength of CSG consolidation formed in water, and the values on the column charts are strength ratios between the compressive strengths of consolidation formed in water and air.

According to the test results, the compressive strength of air-formed specimens increased gradually with the curing age, and the increased rate of strength before the age of 28 days was significantly higher than that after 28 d. This phenomenon indicates that the hydration of cement, as well as the reaction between the hydrated calcium hydroxide and the sodium silicate in the consolidation, increased with age before 28 d, while the hydration reaction from 28 days to 140 days became slower. When the $W/C$ of cement grout remained constant (0.8 or 1.0), the compressive strength of CSG consolidation first increased and then decreased with the increase of $VR$ from 1:1 to 5:1, and the strength reached its maximum when $VR$ was 3:1. Although the real water-cement ratio of CSG ($W_{cs}/C_{cs}$) decreased with the increase of $VR$ (see Table 2), the compressive strength did not show a continuously increasing tendency. These results indicate that the compressive strength of grout consolidation was not only controlled by the real water-cement ratio, but depended more on the content of calcium silicate gel generated by the reaction between calcium hydroxide and sodium silicate. When the $VR$ was 1:1, the cement content was relatively small and the content of sodium silicate was sufficient, the calcium hydroxide generated by the hydration of cement was not enough to react completely with the sodium silicate. Consequently, the production amount of the gel of calcium silicate hydrated was small and the hydration products were not closely contacted. When the $VR$ increased to 5:1, the amount of calcium hydroxide generated by cement hydration was sufficient, and the amount of sodium silicate was insufficient. However, the cement content in the grout consolidation was relatively high, and its hydration was conducive to improve the compressive strength of grout consolidation. When the $VR$ was 3, the reaction between the sodium silicate and the calcium hydroxide generated by cement hydration proceeded sufficiently, and the structure of grout consolidation was more compact with a higher compressive strength.

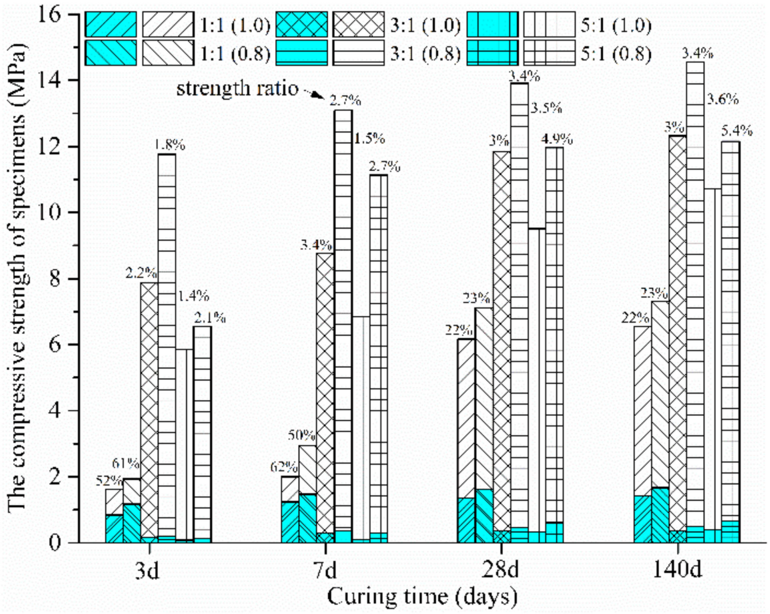


**Figure 7.** Compressive strength characteristics of CSG consolidation formed in water and air.

It can also be found from Figure 7 that the compressive strength of CSG consolidation formed in water was much lower than that formed in air, and the compressive strength in water condition at the age of 140 days was only 3~23% of that in the air condition. The $VR$ is an important factor affecting the consolidation strength. For the grout consolidation formed in water, the compressive strength reached its maximum when the value of $VR$ was 1:1, which is different from the case of air environment. When the values of $VR$ were 3:1 and 5:1, the compressive strength of consolidation at the curing age of 140 days only reached 26~40% of that at the $VR$ of 1:1. According to the analysis of thickness change of the sedimentary consolidation layer, it is known that the cement particles are easily to precipitate in the case of $VR$ = 1:1 due to the relatively long gelation time. In addition, the grout consolidation contains a large amount of sodium silicate, ensuring the continuity of hydration reaction.

However, when *VR* is 3:1 (or 5:1), the gelation time of grout is relatively short, and the deposition of cement particles under the action of gravity is restricted by the calcium silicate gel. In this condition, the grout consolidation contains fewer contents of cement and sodium silicate compared to the case of *VR* = 1. This is not conducive for the formation of calcium silicate gel, which helps improve the compactness of the grout consolidation. On the other aspect, although the compressive strengths of grout consolidation formed in water with the *VR* of 1:1 corresponding to the ages of 3 days and 7 days are 50% or more than that of the consolidation formed in air, the strength increment from 7 days to 140 days is less than 0.2 MPa, and the compressive strength at 140 days is less than 1.7 MPa. The compressive strengths at every age in the conditions of *VR* = 3:1 and 5:1 are only about 5.5% of those of the grout consolidation formed in air, and the maximum strength at 140 days is only 0.67 MPa. The results indicate that the strength of CSG consolidation was significantly weakened by the water environment, and the improvement of strength was not clear with the increase of age, which directly affected the effectiveness and durability of CSG in the water plugging and reinforcement engineering.

In grouting engineering, the transmission and mixing time of CSG in the grouting pipes directly influences the performance of grout consolidation in the water-rich environment. The long initial mixing time of CSG usually leads to the blockage of the grouting channel, while grout with a short mixing time has poor gelation and consolidation properties in the water. In this study, the effect of initial mixing time on grout strength in the water environment was studied to determine the occasion for grout entry into the water. According to the test results of gelation time and consolidation strength, CSG with a $W/C$ of 1 and a *VR* of 1:1 was examined, and the initial mixing time was controlled as 5 s, 15 s and 25 s, respectively. The test results are shown in Figure 8, in which the black and white legends and the colored legends represent the strengths of grout consolidation formed in air and water, respectively, and the data above the column charts denote the strength ratios between the grout consolidation formed in water and air. According to the test results, the extension of initial mixing time improved the compressive strength of grout consolidation at almost all curing ages, especially for the specimens formed in water. For the specimens formed in air, the 3 days strength at the initial mixing time of 15 s and 25 s increased by 0.6% and 5.6%, respectively, and the 140 days strength increased by 4.0% and 4.1%, compared to the condition with the initial time of 5 s. The strength enhancement was mainly due to the fact that the extension of the initial mixing time improved the distribution uniformity of cement particles in the grout consolidation, and consequently improved the overall compactness of consolidation. For the grout consolidation formed in water, the specimen strength corresponding to the initial mixing time of 15 s and 25 s at a curing age of 3 days increased by 32.1% and 54.8%, respectively, compared to the condition of initial mixing time of 5 s. The specimen strength increased by 21.8~31.5% after 3 d, and the strength at the initial mixing time of 25 s showed the most significant increase. The lengthening of the initial mixing time promoted the overall stability of CSG when entering water, and then improved the spatial distribution characteristics of cement particles that play the role of skeleton in the grout consolidation, thus enhancing the compressive strength of underwater forming specimens. According to the analyses, it is necessary to prolong the entering time of CSG appropriately in actual engineering on the premise of ensuring the injection capacity of grout, which will help improve the grouting effect.

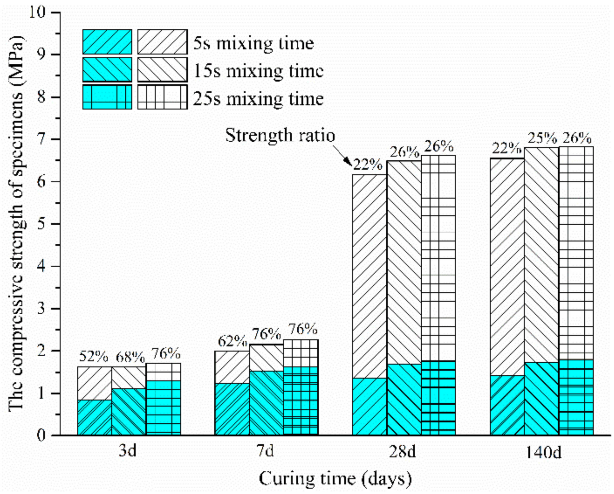


**Figure 8.** Compressive strengths of CSG consolidation formed in water and air with different initial mixing time.

### 3.4. XRD Analysis of Grout Consolidation

In order to analyze the dilution effect of water on the hydration products of CSG consolidation, XRD comparative tests were carried out on the CSG consolidation formed in water and air after curing for 140 days. In addition, the CSG with the *VR* of 1:1 and 5:1 and the *W*/*C* of cement grout of 1 was selected for analysis.

The XRD test results of the CSG consolidation with a *W*/*C* of 1 and a *VR* of 1:1 are shown in Figure 9. The photo in the upper left corner of the figure is the apparent image in the depth direction of the water-formed specimen, and the red dotted line denotes the demarcation line dividing the upper part and the lower part of the specimen with the most obvious color difference. It can be clearly seen that the color of the lower part is darker than that of the upper part, indicating that the content of cement in the lower part is significantly higher than the upper part, and the hydration products in these two parts are also different. Therefore, the upper and lower parts of the underwater formed CSG consolidation were analyzed by XRD.

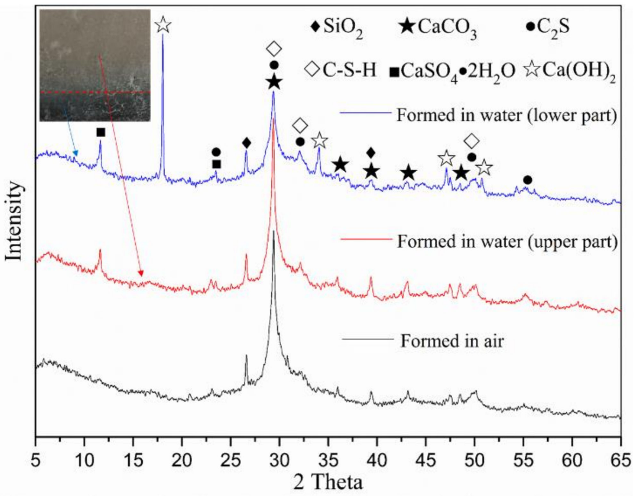


**Figure 9.** XRD patterns of CSG specimens formed in water and air with the volume ratio of 1:1.

As can be found from Figure 9, the characteristic diffraction peak of calcium hydroxide in the lower part of the underwater-formed specimen was significantly higher than that observed in the upper part of underwater-formed specimen and that observed in the specimen formed in air. Due to the dilution effect of water, the content of sodium silicate in the grout consolidation decreased, the content of cement in the lower part of specimen formed in water was relatively higher and the calcium hydroxide generated by hydration could not completely react with the sodium silicate. In addition, the characteristic diffraction peak of dihydrate gypsum in the CSG consolidation formed in water was significantly higher than that in the specimen formed in air, indicating that the dilution effect of water enlarges the spacing between cement particles, which limits the participation of dihydrate gypsum in the cement hydration. There were no obvious diffraction peaks of dihydrate gypsum and calcium hydroxide in the CSG consolidation formed in air, since the cement hydration was relatively complete, and the reaction between the calcium hydroxide generated by cement hydration and the sodium silicate was adequate.

The XRD test results of CSG consolidation with a $W/C$ of 1 and a $VR$ of 5:1 are shown in Figure 10. The image color of the underwater-formed specimen in the upper left corner of the figure shows no difference in the depth direction. Therefore stratified sampling was not performed in this study. According to the results, the diffraction peak of calcium hydroxide formed in water environment was much lower than that of the specimen formed in air, since the sodium silicate in the air condition was not enough to consume the calcium hydroxide generated by the cement hydration. In the water environment, the distance between cement particles in the CSG consolidation was relatively large, and the calcium silicate hydrate gel generated by the reaction between calcium ions and silicate ions filled the gap between cement particles, thus reducing the contents of cement and calcium hydroxide generated from cement hydration.

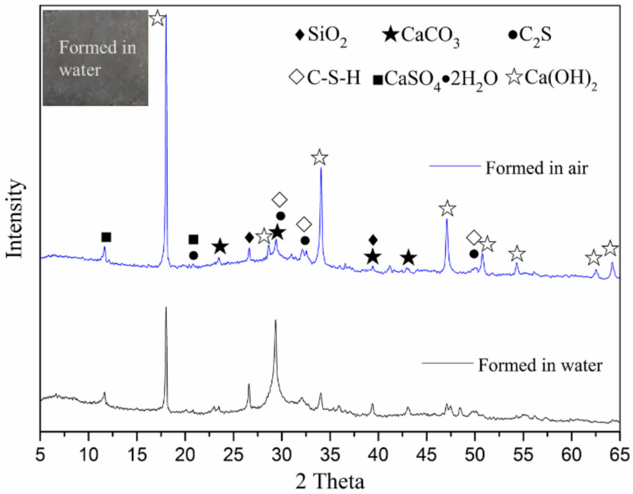
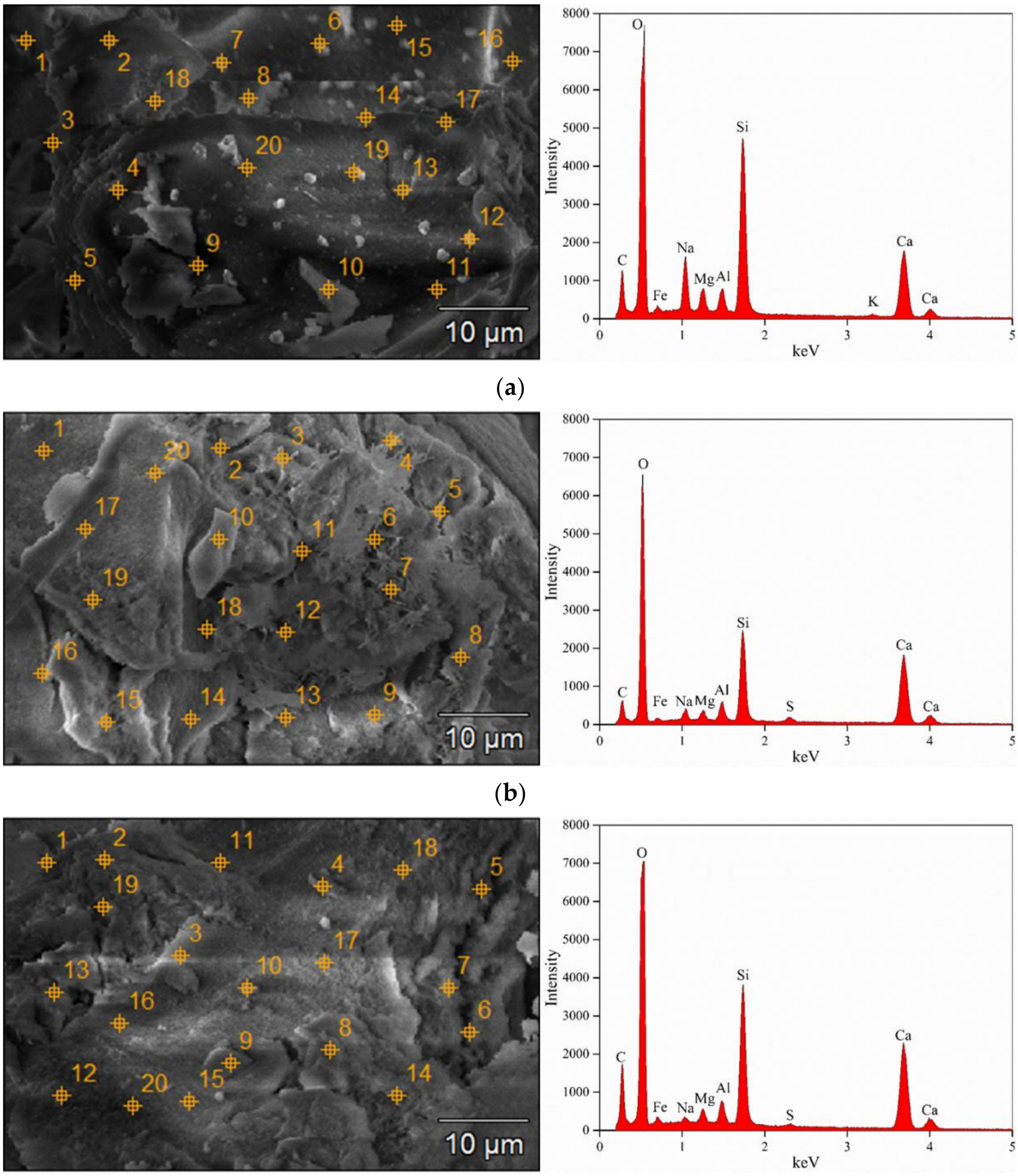


**Figure 10.** XRD patterns of specimens formed in water and air with a volume ratio of 5:1.

### 3.5. SEM Analysis of Grout Consolidation

The SEM images and energy spectra of CSG consolidation with the $VR$ of 1:1 and 5:1 formed in water and air are shown in Figures 11 and 12. In comparison to the SEM images of CSG consolidation formed in air, the surface of specimen formed in water was porous and the connection between hydration products was loose, further confirming that the dilution of water reduces significantly the compactness of grout consolidation. Moreover, the surface of grout consolidation with the $VR$ of 5:1 was looser than that in the condition of $VR$ = 1:1, which helps explain its lower strength characteristic. In addition, the surface of the upper layer of the underwater-formed specimen with the $VR$ of 1:1 contained more pores than the lower layer, which indicates that the dilution of water caused the uneven settlement of cement particles and then intensified the heterogeneity of grout consolidation.



(**a**)

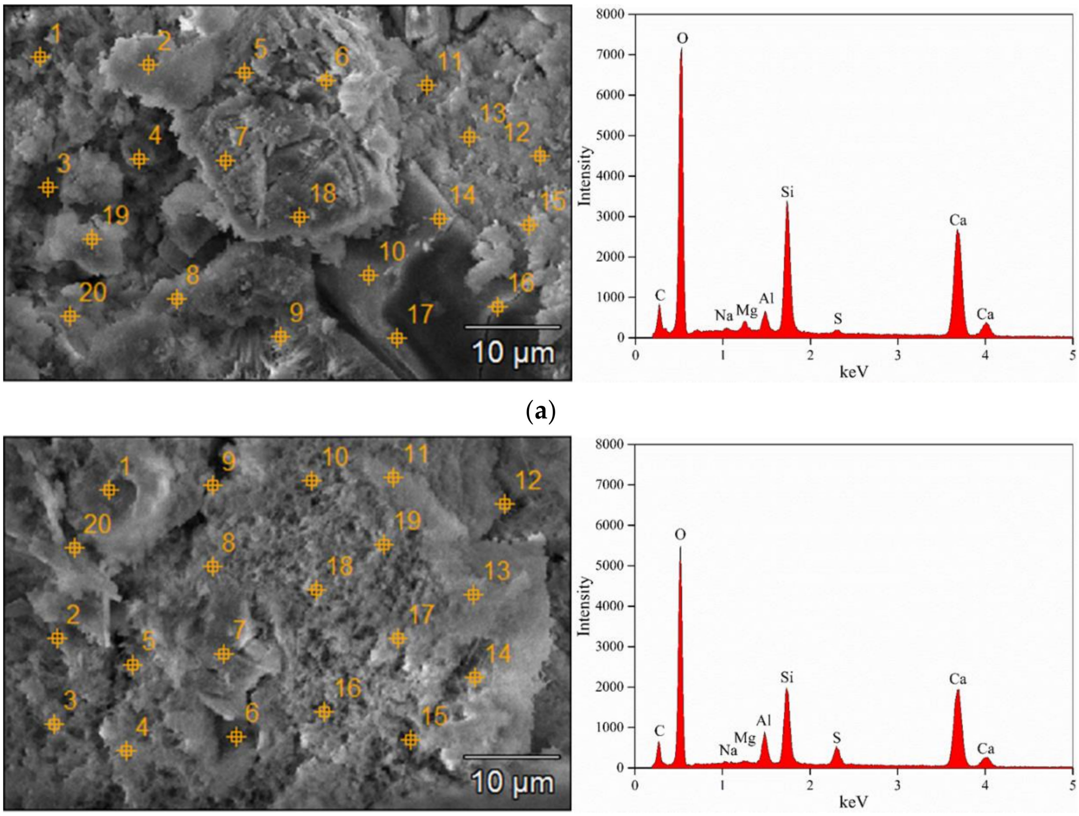
(**b**)

(**c**)

**Figure 11.** SEM images of CSG consolidation formed in water and air with a volume ratio of 1:1 (×3500). (**a**) CSG consolidation specimen formed in air; (**b**) upper part of consolidation specimen formed in water; (**c**) lower part of consolidation specimen formed in water.

Since the strength of the CSG consolidation formed in water was too low to be polished, 20 points were selected from the SEM images for energy dispersive spectrometer (EDS) analysis, as shown in Figures 11 and 12. The analysis results, after calculating the average value of chemical elements at those 20 points, are listed in Table 3. The atomic percentage of chemical elements in the CSG consolidation formed in water and air was clearly different. The atomic percentages of sodium and silicon of consolidation formed in air were significantly higher than those of consolidation formed in water when the values of *VR* were 1:1 and 5:1, mainly because the dilution of water significantly reduced the content of sodium silicate in the consolidation. However, the atomic percentage of calcium of consolidation formed in the air was lower than that formed in water when the values of

*VR* were 1:1 and 5:1, which further shows that the dilution of water causes the increase of cement particle content in the sedimentary consolidation layer.



(**a**)

(**b**)

**Figure 12.** SEM images of CSG consolidation formed in water and air with a volume ratio of 5:1 (×3500). (**a**) CSG consolidation specimen formed in air; (**b**) CSG consolidation specimen formed in water.

**Table 3.** The atomic percentage of chemical elements in the CSG consolidation obtained by EDS analysis.

| **Element** | ***VR* = 1:1** | | | ***VR* = 5:1** | |
|---|---|---|---|---|---|
| | **Formed in Air (%)** | **Formed in Water (%)** | | **Formed in Air (%)** | **Formed in Water (%)** |
| | | **Upper Part** | **Lower Part** | | |
| O | 65.10 | 65.64 | 66.53 | 64.69 | 62.40 |
| Si | 11.06 | 8.86 | 8.63 | 9.01 | 7.60 |
| Al | 1.27 | 1.52 | 1.23 | 1.14 | 2.52 |
| Ca | 10.56 | 13.15 | 13.90 | 18.86 | 20.11 |
| Na | 3.56 | 1.19 | 0.38 | 0.22 | 0.13 |
| Mg | 1.38 | 0.91 | 0.79 | 0.68 | 0.20 |

## 4. Conclusions

This study investigated the gelation and consolidation characteristics of CSG in the water environment and obtained the influence of the dilution of water on the diffusion and gelation characteristics, compressive strength and microstructure of CSG with different volume ratios and mixing times. The main conclusions and accomplishments are summarized as follows:

(1) The CSG diffused throughout the whole water body within a few seconds after entering the water, and its diffusion pattern was rarely affected by the *VR* and initial stirring time.

(2) The CSG started to gel after 12~15 min in the water, which indicates that the quick-setting characteristic of CSG in air for tens of seconds is clearly weakened in the water-rich environment.

(3) The CSG exhibited stratification and segregation phenomena after a period of time in water, and it could be divided into the suspension layer, the water-retaining soft gel layer and the sedimentary consolidation layer. The sedimentary consolidation layer gradually thickened with the increase of *VR*, and the thickness of the water-retaining soft gel layer reached its maximum at a *VR* of 1:1, which was almost twice the thickness at the volume ratios of 3:1 and 5:1. The extension of initial stirring time decreased the thickness of sedimentary consolidation layer and increased the thickness of water-retaining soft gel layer.

(4) The 140 days compressive strength of the CSG consolidation formed in water was only 3~23% of that formed in air. The compressive strength of the CSG consolidation formed in water corresponding to the *VR* of 1:1 was significantly higher than that corresponding to the volume ratios of 3:1 and 5:1, which was different from the maximum compressive strength of the CSG consolidation formed in air at a *VR* of 3:1. The extension of initial mixing time is helpful to improve the compressive strength of grout consolidation formed in water.

(5) The hydration products of CSG consolidation formed in water change significantly compared with that formed in air. The consolidation microstructure becomes loose, and the connection of hydration products is weak because of the dilution effect of water.

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