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EFFECT OF THE MOLAR RATIO OF LIQUID GLASS GROUTING AGENTS ON MECHANICAL CHARACTERISTICS OF THE SOLIDIFIED SOILS

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ABSTRACT: Chemical grouting techniques are used as ground improvement methods to obtain stable ground. In this method, soft sandy soils are solidified by injecting liquid solidification agents into the soils to prevent liquefaction as well as to increase strength. In this study, liquid glass grouting agents containing liquid glass with a higher polymerization degree (No. 5 liquid glass grouting agents) are being developed and discussed while focusing on the liquid agents to be used for chemical grouting techniques. Specifically, the solidification properties of No. 5 liquid glass grouting agents and the influence of the molar ratio of liquid glass to the strength and shrinkage characteristics of sand-gel and homo-gel are experimentally studied. As a result, the sand-gel made of No. 5 liquid glass grouting agents is superior in strength and has smaller shrinkage ratios after gelation, and therefore, is superior in soil improvement.

Keywords: Chemical grouting techniques; liquid glass grouting agents; molar ratio; soil improvement

1. INTRODUCTION

Chemical grouting techniques are used as ground improvement methods to obtain stable ground. In this method, soft sandy soils are solidified by injecting liquid solidification agents (hereafter, referred to as “liquid agents”) into the soils to prevent liquefaction as well as to increase strength. The liquid agents infiltrate into the voids of soil particles and gelate the soil particles, producing an adhesive effect to increase soil strength and to reduce the water permeability of the soil.

Because chemical grouting techniques can be implemented relatively easily by using compact equipment, they have been utilized for short-term temporary work (e.g., auxiliary underground work) [1]. However, owing to recent improvements in injection technology, including the double pipe strainer method and double pipe and double packer method, as well as the development of injection materials, their utilization is expanding to many other fields [2]. For example, in addition to conventional temporary demand, the recent performance improvement of grouting agents has led to demand increase in the fields requiring permanent stability such as liquefaction prevention. In such usage, long-term durability is required for the grouting agents. Furthermore, the remarkable advancement of injection technology has also contributed to demand increase. For example, by using the technology together with the double pipe strainer method and double pipe and double packer method, ground just beneath an established structure can be excavated with arc-shaped boring, and liquid agents can be injected into the bore hole without interrupting the functions of the established structure [3]. This way, chemical grouting techniques are expected to be utilized for a broader range of purposes including liquefaction prevention, which is a major issue nowadays, deep subterranean development work and so on [5].

As described above, the application of chemical grouting techniques is expected to expand. However, the liquid glass grouting agents, which are typically used for these methods, have some problems regarding strength and durability for both usages as homo-gel (agent only) and sand-gel (sandy soil gelated by an agent). There are some liquid glass grouting agents that have been developed specifically to solve the problems of strength and durability, but their cost is high. Thus, economical and high-performance liquid agents are under development [6-7].

In this study, in order to solve the problems mentioned above, liquid glass grouting agents containing liquid glass with a higher polymerization degree (i.e., higher molar ratio defined by the molar mass ratio of SiO_2 against Na_2O) (hereafter, referred to as “No. 5 liquid glass grouting agents”) are being developed by authors and discussed while focusing on the liquid agents to be used for chemical grouting techniques. Specifically, the solidification properties of No. 5 liquid glass grouting agents and the influence of the molar ratio of liquid glass to the strength and shrinkage characteristics of sand-gel and homo-gel are experimentally studied.

2. OVERVIEW OF LIQUID AGENTS FOR CHEMICAL GROUTING TECHNIQUES

Liquid agents used for chemical grouting techniques are chemical products or fluids. They can be classified into chemical-based agents and nonchemical-based agents as shown in Fig. 1.

The nonchemical-based agents in Fig. 1 use the hydration reaction of cement and so on, for solidification and are difficult to gelate in a specified time period. Therefore, the agents may outflow before gelating in ground that has water flow and it is difficult to achieve the expected effect. On the other hand, the chemical-based agents are currently limited to liquid glass grouting agents because use of polymer-based agents is prohibited by "Interim Guideline for Execution of Construction Work by Chemical Grouting Techniques". Liquid glass grouting agents are expected to overcome the issues of nonchemical-based agents because their gelatification time (gelling time) can be flexibly controlled and varied from a few seconds to a few hours. However, it has been pointed out that liquid glass grouting agents have the following issues in terms of strength, shrinkage characteristic, durability and economy:

(1) Residue liquid glass compromises the strength and durability of solidified sandy soils (sand-gels).
(2) Because the shrinkage ratio of sandy soils (sand-gels) after solidification is large, sufficient strength increase cannot be achieved in some cases.
(3) If the molar ratio of liquid glass is small, a large amount of sulfuric acid is required to neutralize the alkaline metal of the liquid agents. Thus, it is not economical.

Most of conventional liquid glass grouting agents are called "No. 3" based on the classification by the molar ratio of liquid glass (Table 1; Japanese Industrial Standards (JIS K 1408)). In this study, through development and discussion of liquid glass grouting agents containing liquid glass with a larger molar ratio, such as No. 5 liquid glass grouting agents in pursuit of solutions to the issues (1) to (3) above.

3. BASIC CHARACTERISTICS OF NO. 5 LIQUID GLASS GROUTING AGENTS

In this study, liquid glasses with a larger molar ratio are used to develop and discuss No. 5 liquid glass grouting agents, which are mainly made of liquid glasses with a larger molar ratio and inorganic acid (sulfuric acid) and classified as liquid glass-based inorganic solution-type medium acidity agents. The basic characteristics of No. 5 liquid glass grouting agents are as follows:

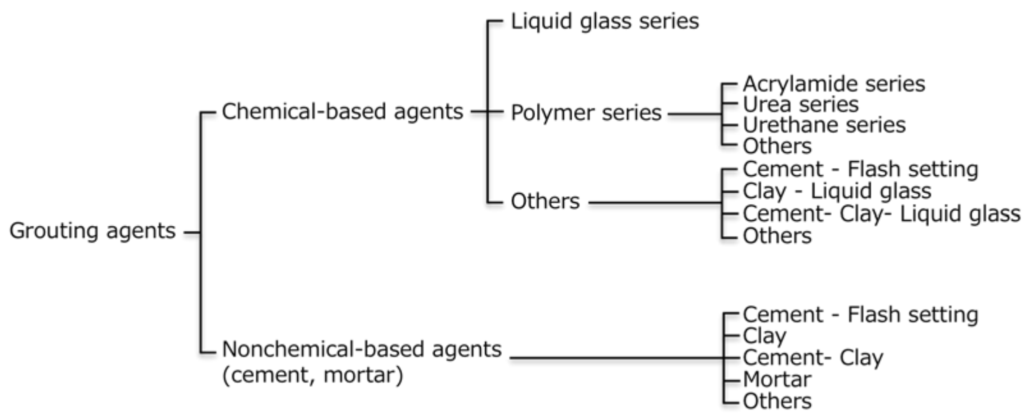


Fig. 1 Classification of grouting agents in chemical grouting techniques

Table 1 Classification by molar ratio of liquid glass grouting agents based on the JIS K 1408

	No.1	No.2	No.3	No.4	No.5
Molar ratio	2.12	2.51	3.2	3.35	3.73
Concentration of SiO_2 [%]	32.11	28.62	29.12	24.21	25.49
Concentration of Na_2O [%]	15.63	11.78	9.38	7.45	7.04
Specific gravity	1.582	1.452	1.405	1.315	1.319

3.1 Safety of No. 5 Liquid Glass Grouting Agents

No. 5 liquid glass grouting agents are acid silica fluid made by adding a hardener (sulfuric acid) to a main agent (No. 5 liquid glass). Its gelling time can be controlled by changing its pH through addition of diluted No. 5 liquid glass (Fig. 2). Because No. 5 liquid glass grouting agent using No. 5 liquid glass with less sodium (Na) suppresses the generation of sodium sulfate (Na_2SO_4) and additional diluted No. 5 liquid glass helps the agent to gelate without being affected by salt and organic matters in the soil, it is very safe regarding the surrounding environment and on-site injection conditions.

3.2 Influence of No. 5 Liquid Glass Grouting Agents on Surrounding Geo-environment

Figure 3 shows the fluctuation of the immersion water pH from the sand-gel improved by No. 5 liquid glass grouting agents of the composition of slow setting (main agent: hardener = 10: 1) and composition of flash setting (main agent: hardener = 9: 1). This way, it is clear that no alkaline metal has eluted from the sand-gel into which No. 5 liquid glass grouting agents were injected because the pH of the water used for immersion is maintained in the range of 6.5 to 7.5. In addition, because the particle diameter of the silica component contained in the main agent is relatively large, sand-gel does not shrink much and can maintain its imperviousness for a long time.

3.3 Durability of No. 5 Liquid Glass Grouting Agents

Figure 4 shows the time series variation in the unconfined compressive strength of a sand-gel specimen in which Toyoura sand and No. 5 liquid glass grouting agents with slow setting composition are mixed. The specimen complies with "Practice for making and curing chemically grouted soil specimens (Japan Geotechnical Society Criteria; JGS 0831)" and is cured under the condition of temperature 20°C. Because No. 5 liquid glass grouting agents use liquid glass with a higher polymerization degree (i.e., larger molar ratio), it contains less sodium. Thus, it is superior in reaction to hardeners to efficiently solidify silica in liquid glass. Furthermore, because alkaline metal in liquid glass, which weakens silica solidification and causes solidification deterioration, is completely neutralized by acid, the sand-gel can keep its strength over a long period of time once silica has solidified to become resistant to shrinkage and fragmentation.

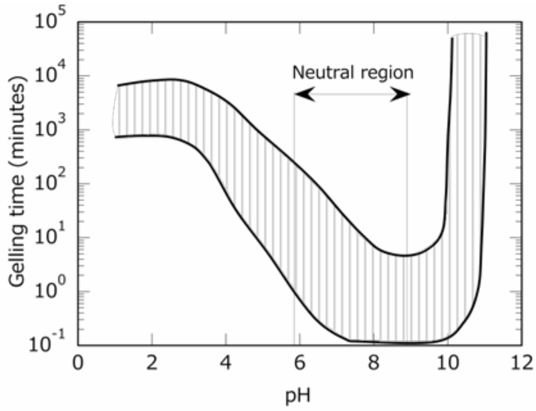


Fig. 2 Relationship between pH and gelling-time on the No. 5 liquid glass grouting agents

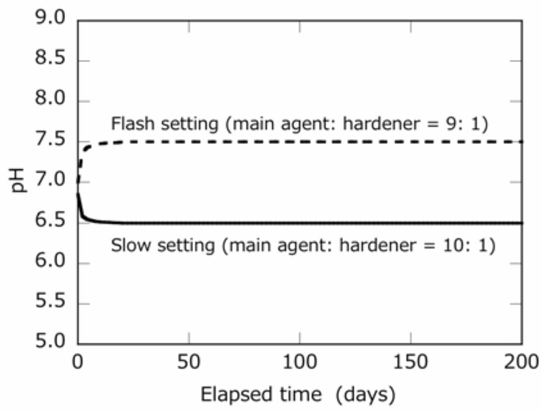


Fig. 3 Fluctuation of the immersion water pH from the sand-gel improved by the No. 5 liquid glass grouting agents

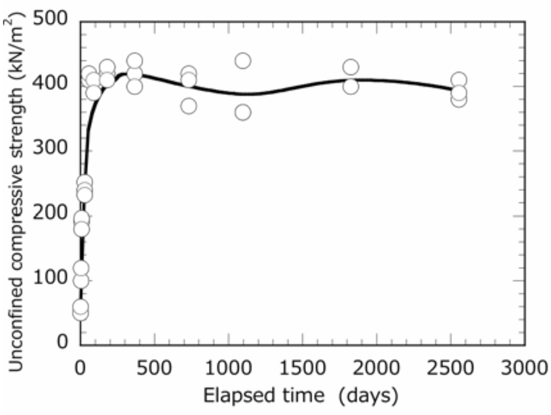


Fig. 4 Time series variation in unconfined compressive strength of the sand-gel specimen by the No. 5 liquid glass grouting agents

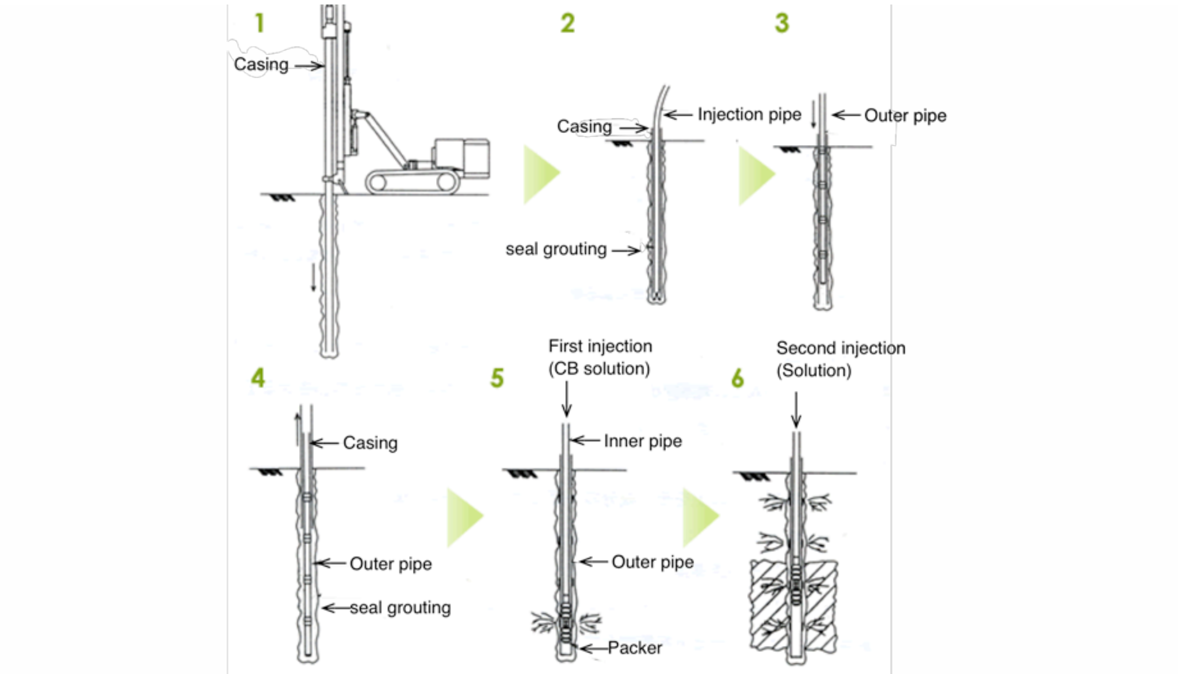


Fig. 5 Schematic view on double pipe and double packer methods

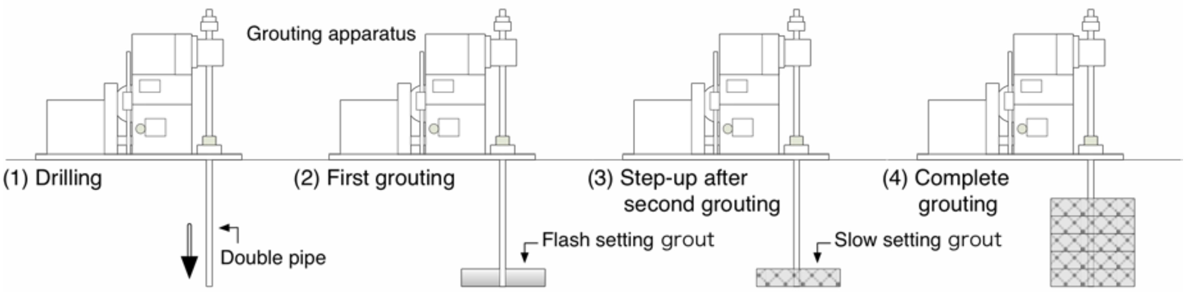


Fig. 6 Schematic view on double pipe strainer methods (diploid-phase)

3.4 Workability of No. 5 Liquid Glass Grouting Agents

When No. 5 liquid glass grouting agents are injected into the ground, the double pipe and double packer method (Fig. 5) [8] or the double pipe strainer method (Fig. 6) [8] is generally used. Because the double pipe strainer method uses casing drilling and injection separately, the injection can be more precise and uniform. Usually, injection around the filling pipe is done first and then slow setting injection is done as the secondary injection by the double packer. Because permeation grouting is used for sandy soil, the changes in structural fabric of the sandy soil are minimized and the impact on the structure above the sandy soil becomes minor.

4. MIXING AND SOLIDIFICATION REACTION OF NO. 5 LIQUID GLASS GROUTING AGENTS

4.1 Mixing of No. 5 Liquid Glass Grouting Agent and Hardener

No. 5 liquid glass grouting agents are classified as solution typed neutral and acid reagents in Fig. 7. There are typically two mixing methods of the main agent (solution typed neutral and acid liquid glass) and the hardener (sulfuric acid): “direct mixing” and “indirect mixing”. In this study, the indirect mixing method is mainly discussed.

In the direct mixing method, the main agent (liquid glass) and the hardener (sulfuric acid) are directly mixed during injection (Fig. 8). However, this method has a problem: because silica sol gelates very fast, unreacted sulfuric acid and liquid glass might remain. On the contrary, in the indirect

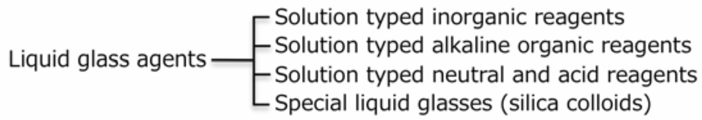
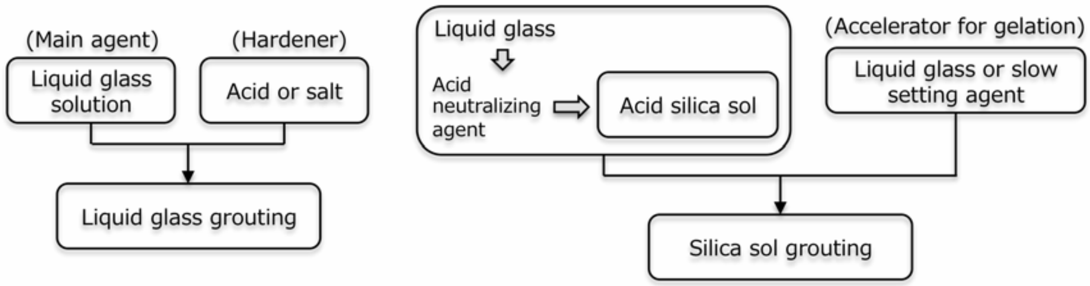


Fig. 7 Classification of liquid glass agents in chemical grouting methods



(a) Direct method (b) Indirect method

Fig. 8 Simple overview on mixing main agent and hardener during injection in direct and indirect methods

mixing method, liquid glass and sulfuric acid are mixed at the plant before injection into the soil, and there are two ways of injection: create acid silica sol beforehand and inject it into the soil, or mix liquid glass to the created acid silica sol to adjust the gelling time and inject it into the soil. In the indirect mixing method, there is no residual unreacted sulfuric acid, unlike the direct mixing method because liquid glass is mixed with sulfuric acid before injection. Generally, when liquid glass grouting agents are used, more unreacted liquid glass and sulfuric acid tend to remain because sulfuric reacts too quickly. Furthermore, because No. 5 liquid glass grouting agents have a higher polymerization degree than general liquid glass grouting agents, there is a higher possibility of leaving unreacted residues. Therefore, it is necessary to use the indirect mixing method instead of the direct mixing method to prepare the injection fluid when No. 5 liquid glass grouting agents are used.

The preparation of the injection fluid in past indirect mixing method was by manual procedures. Therefore, some errors are caused to adjusting specific gravity and fixing pH during the mixing liquid glass and the sulfuric acid. In recent years, a machine that can automate the preparation of the injection fluid has been developed as shown in Fig. 9. With this machine, the above-mentioned adjusting specific gravity and fixing pH can be carried out with sufficient accuracy.

4.2 Solidification Reaction of No. 5 Liquid Glass Grouting Agents

Acid silica sol solution ($\equiv$Si-OH$^-$) can be obtained by making silicate anion ($\equiv$Si-O$^-$ Na$^+$) in

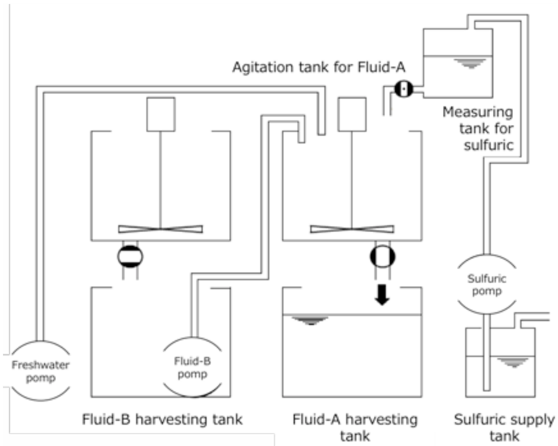


Fig. 9 Outline of the machine preparing injection fluid automatically

liquid glass ($Na_2O \bullet nSiO_2$) react to the hardener, sulfuric acid (H_2SO_4) as shown in Eqs. (1) and (2).

$$\equiv Si\text{-}O^- Na^+ + H_2SO_4 \rightarrow \equiv Si\text{-}OH + NaHSO_4 \quad (1)$$

$$\equiv Si\text{-}O^- Na^+ + NaHSO_4 \rightarrow \equiv Si\text{-}OH + Na_2SO_4 \quad (2)$$

The silicic acid ($\equiv$Si-OH) contained in the acid solution reacts due to the dehydration and condensation shown in Eq. (3) and it gelates within several 10 hours.

$$\equiv Si\text{-}OH + HO\text{-}Si\equiv \rightarrow \equiv Si\text{-}O\text{-}Si\equiv + H_2O \quad (3)$$

If a neutral region is maintained by adding liquid glass to the acid silica sol solution, the dehydration and condensation as shown in Eq. (4)

Table 2 Mixing ratio of main agent, hardener and reagent of each liquid glass grouting agent

		Fluid-A		Fluid-B		Fluid-C		Total amount of liquid glass	Total conc. of SiO_2 [wt%]	Total conc. of Na_2O [wt%]
		Sulfuric acid	Water	Liquid glass	Water	Liquid glass	Water			
No.1	[mL]	15.50	64.50	20.00	100.00	30.70	169.30	50.70	5.89	2.87
	[g]	26.50	64.50	31.64	100.00	48.56	169.30	80.20		
No.2	[mL]	13.10	66.90	20.00	100.00	42.00	158.00	62.00	5.89	2.42
	[g]	22.40	66.90	29.04	100.00	60.98	158.00	90.02		
No.3	[mL]	10.10	69.90	20.00	100.00	42.00	158.00	62.00	5.87	1.89
	[g]	17.27	69.90	28.10	100.00	59.01	158.00	87.11		
No.4	[mL]	9.50	70.50	30.00	90.00	49.00	151.00	79.00	5.83	1.79
	[g]	16.25	70.50	39.45	90.00	64.44	151.00	103.89		
No.5	[mL]	8.60	71.40	30.00	90.00	45.00	155.00	75.00	5.86	1.62
	[g]	14.71	71.40	39.57	90.00	59.36	155.00	98.83		

will be generated. Because of the reaction (Eq. (4)) is quite faster than that shown in Eq. (3), it can be promoted to the gelation.

$$\equiv Si\text{-}OH + Na^+ \, {}^-O\text{-}Si\equiv + H_2SO_4 \text{ or } NaHSO_4 \rightarrow \equiv Si\text{-}O\text{-}Si\equiv + H_2O + NaHSO_4 \text{ or } Na_2SO_4 \quad (4)$$

Because sodium ion (Na^+) hydrolyzes silica polymer, silicate anion having lower polymerization degree of liquid glass is solved in water. When sulfuric acid is mixed, it neutralizes Na^+ to produce sodium sulfate (Na_2SO_4) and sodium hydrogen sulfate ($NaHSO_4$), which means a decrease of Na^+ depolymerization. As a result, gelation is promoted. It is possible to have this reaction in the acid region and also in the neutral region by changing the amount of liquid glass to be added. Note that gelation occurs in a few seconds to up about 20 seconds in the neutral region (pH: 6.5 to 8.5) and it takes nearly 10 to 100 hours in the acid region (pH: 2 to 3).

5. MOLAR RATIO AND STRENGTH OF LIQUID GLASS

Liquid glass is classified into five categories from No. 1 to No. 5 depending on molar ratio. While No. 3 liquid glass is usually used, in this study we use No. 5 as the main agent for No. 5 liquid glass grouting agents (Table 1). Compared with others, No. 5 has a smaller amount of alkaline (Na_2O); so, it can reduce the amount of sulfuric acid necessary for neutralization, resulting in cost saving. As an additional advantage, it can also reduce sodium sulfate that is produced in the reaction of alkaline metal to sulfuric acid, which means a smaller effect on the environment. The molar ratio here is defined as the molecular weight proportion of SiO_2 against Na_2O.

5.1 Unconfined Compression Test and Shrinkage Test

In order to compare liquid glass grouting agents using liquid glass (sodium silicate) with different molar ratios as shown in Table 1, in this study a unconfined compression test for each sand-gel and a shrinkage test for each homo-gel were conducted. Table 2 shows the mixing ratio of the main agents with sulfuric acid (Fluid-A) and liquid glass (Fluid-B), also hardener with diluted liquid glass (Fluid-C) of each liquid glass grouting agent (400ml). One of the main agents is sulfuric acid with a concentration 78% and specific gravity 1.71. The composition of slow setting for No. 5 liquid glass grouting agents under development and discussion contains 75 liters of No. 5 fluid glass per 400 liters, with silica concentration 5.8% and pH about 2.5. No. 1 to 4 liquid glass grouting agents were also prepared with the same silica concentration and pH.

In the unconfined compression test of each sand-gel, the specimens are prepared by mixing liquid glass grouting agents with different molar ratios (Table 2) with Toyoura sand after adjusting its relative density to be 50%. The way of making the specimen complies with “Practice for making and curing chemically grouted soil specimens (Japan Geotechnical Society Criteria; JGS 0831)”. After calculating the weight of Toyoura sand according to the relative density, it is fed by free fall into a mold (5cm in diameter and 10cm in height) filled with a liquid glass grouting agent with a specific molar ratio. Then, the specimens are cured under the condition of temperature 20°C for 7 or 28 days to prepare for unconfined compression tests.

The test method of the homo-gel shrinkage ratio is described as follows: first, the specific gravity of liquid glass grouting agents with different molar ratios (Table 2) is measured, and 400ml of each injection agent is put in a polypropylene container (500ml) for gelation. Then, the weight of syneresis water is measured after 7 days, 14 days, 21 days and 28 days to calculate the volume shrinkage ratios. Three specimens are prepared for each type of liquid glass grouting agent for measurement.

5.2 Results and Discussion

As a result of unconfined compression tests of sand-gel of liquid glass grouting agents using liquid glass with different molar ratios and shrinkage tests of homo-gel, it was confirmed that sand-gel mixed with liquid glass grouting agents with larger molar ratios can achieve higher unconfined compressive strength (Fig. 10), and that sand-gel mixed with liquid glass grouting agents with larger molar ratios can have smaller shrinkage ratios (Fig. 11). In other words, both homo-gel and sand-gel of No. 5 liquid glass grouting agents have larger strength than those of No. 1 to No. 4 liquid glass grouting agents and shrink less due to gelation. Therefore, No. 5 liquid glass grouting agents are superior in soil reinforcement.

Figures 10 and 11 indicate a strong correlation among the molar ratio of liquid glass, shrinkage ratio and solidified soil strength. Shrinkage of homo-gel occurs when sulfuric acid (H_2SO_4) is added to liquid glass in which SiO_2 is depolymerized by Na^+ and OH^-, and Na^+ and OH^- are neutralized to make SiO_2 polymerize again and dehydrating the structure. The amount of lost moisture during the shrinkage of homo-gel influences the shrinkage ratio. In addition, the amount of lost moisture depends on the concentration of Na_2O: as the volume is smaller, the amount of lost moisture becomes smaller, then the shrinkage ratio calculated based on the amount of lost moisture becomes smaller as well. Therefore, when the molar ratio becomes larger in liquid glass, the shrinkage ratio of homo-gel becomes smaller. As shown in Fig. 12, the liquid glass grouting agents are injected to fill voids in the soft ground and have the effect to bond soil particles. At that time, if the liquid glass grouting agents shrink considerably, the homo-gel that fills

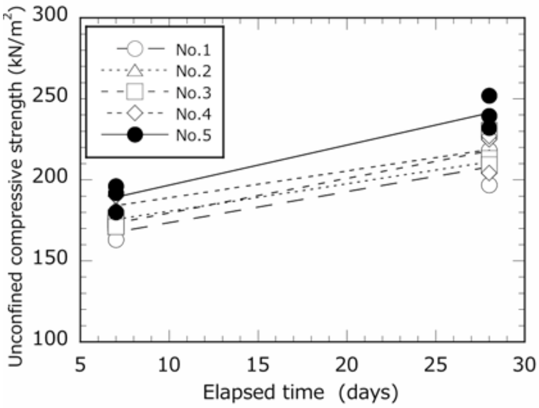


Fig. 10 Time series variation in unconfined compressive strength of the sand-gel specimens mixed with liquid glass grouting agents with each molar ratio

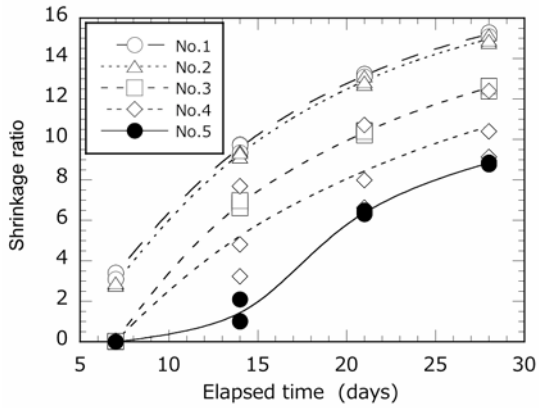


Fig. 11 Time series variation in shrinkage ratio of the homo-gel specimens mixed with liquid glass grouting agents with each molar ratio

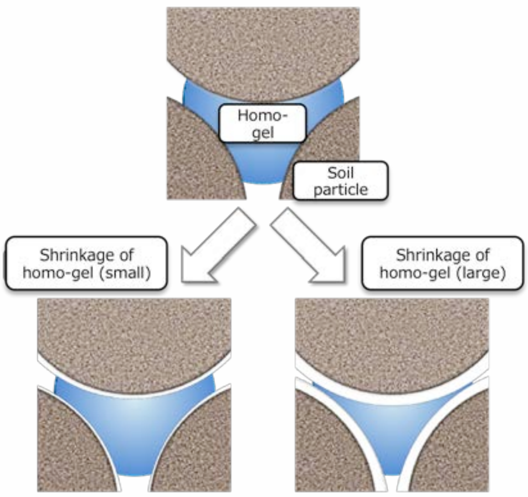


Fig. 12 Simple image on liquid glass grouting agents in voids of soil particles

voids becomes smaller and detaches from soil particles. As a result, its bonding effect decreases. Therefore, liquid glass grouting agents with smaller shrinkage ratios are required. In addition, No. 5 liquid glass grouting agents under development and discussion have a higher molar ratio of liquid glass and are superior in soil reinforcement to existing liquid glass grouting agents.

However, hand mixing of No. 5 liquid glass grouting agents may cause unevenness of composition and it affects gelling time. Therefore, it is required to install a dedicated plant in which mixing of No. 5 liquid glass grouting agents can be done by a machine. In other words, because mixing of No. 5 liquid glass grouting agents yet involves sensitive work, it is preferable that the mixing is done by a machine in a dedicated plant.

6. CONCLUSIONS

Because No. 5 liquid glass grouting agents use No. 5 liquid glass with higher molar ratios as the main agent, the concentration of alkaline (Na_2O) is smaller. Therefore, the amount of hardener (sulfuric acid) required for neutralization can be reduced; and at the same time they affect the environment less and reduce cost because generation of sodium sulfate form reaction can be suppressed. In addition, the sand-gel made of No. 5 liquid glass grouting agents is superior in strength and has smaller shrinkage ratios after gelation, and therefore, is superior in soil improvement.

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