Development of Colloidal Silica Grout Using Calcium Hydroxide Reactant

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ABSTRACT

This study aims to enhance soil stability and mechanical properties using colloidal silica-based grout. Colloidal silica (CS) is a durable, silicon-based chemical grout widely used in permeation grouting. The research focuses on developing optimal grout formulations by investigating various physical, rheological, and strength parameters in combination with calcium hydroxide Ca $(OH)_2$. Laboratory tests were conducted to measure gel time, pH value, viscosity, and unconfined compressive strength (UCS) with different water to colloidal silica ratios (w/cs=0.5, 1, 2) varying different concentrations of Ca $(OH)_2$. The results revealed several key trends: as the water-to-colloidal silica ratio decreased, UCS strength increased, gel time decreased, pH value decreased, and viscosity increased with the decreased w:cs ratio. Among the tested formulations, the grout with a 0.4% concentration of reactant with w:cs=0.5 gave better strength compared to other w:cs ratios and improved the strength of the grout. The study concludes that using a Ca $(OH)_2$ concentration of 0.4% with a w:cs ratio of 0.5 is optimal for achieving improved grout strength and soil stabilization.

INTRODUCTION

Colloidal silica (CS) is a silicon-based chemical grout known for its remarkable durability and inert properties. It poses no health hazards and is unaffected by filtration, making it both chemically and biologically stable. These characteristics make colloidal silica an excellent choice for various soil treatment applications, including tunnels, dam construction, and site stabilization. One of the key benefits of colloidal silica treatment is its ability to enhance the mechanical properties of soil. It increases soil strength, reduces hydraulic conductivity, and enhances resistance to liquefaction. These improvements are particularly valuable in construction projects where soil stability is crucial for safety and structural integrity. Colloidal silica is produced through an innovative process that involves extracting alkali from sodium silicate using ion-exchange resins in a controlled factory environment. This process results in colloidal silica particles that have an electrical double layer around their surfaces. When this double layer is disrupted by adding inorganic salts, the colloidal particles bond through siloxane bonds, forming a gel network. The size of colloidal silica particles typically ranges from 10 to 100 manometers in diameter. The formation of the colloidal silica grout network occurs through the condensation and polymerization of silanol radicals on the surface of the colloidal particles. This process creates a robust and interconnected structure, which can be visualized as a network of spherical particles bonded together. The resulting gel network significantly enhances the soil's properties, providing a stable and durable foundation for various construction projects. in practical applications, colloidal silica grout is injected into the soil, where it permeates the soil matrix and sets into a stable, cohesive gel. This gel stabilizes the soil, making it less prone to erosion and shifting. It also reduces the soil's permeability, which is crucial in preventing water seepage and maintaining the integrity of underground structures like tunnels and dams. Colloidal silica's versatility and effectiveness have made it a popular choice in geotechnical engineering. Its ability to improve soil properties without posing health or environmental risks makes it an attractive alternative to traditional grouting materials. Further-more, the innovative production process and the resulting high-performance characteristics of colloidal silica grout continue to drive its adoption in various construction and stabilization projects. Whether used in large-scale infrastructure projects or smaller site stabilization efforts, colloidal silica grout offers a reliable and effective solution for improving soil performance.

This study explores the impact of varying concentrations of calcium hydroxide Ca $(OH)_2$ with colloidal silica grout, including gel time, syneresis, pH, timeviscosity, and unconfined compressive strength (UCS). The objective is to enhance the formulation of colloidal silica grout by integrating calcium hydroxide with water-to-colloidal silica ratios of 0.5, 1, and 2.

MATERIALS OF INVESTIGATION

The basic materials used in this study included colloidal silica, water, sand, and calcium hydroxide $Ca(OH)_2$. For the grouted sand samples, naturalBhadarpur sand of Orsang river near Sankheda of Vadodara District was utilized, reactant used were commercially sourced. The properties of the colloidal silica, sand, and calcium hydroxide are detailed and shown in tables 1 to 3.

Table 1. Properties of colloidal silica

Test grade	Cilicol30Ak
Concentration of SiO_2	30-31 w t %
Concentration of Na_2O	0.3-0.5wt%
pH	9.5-10.5
Particle size	10-20nm
Viscosity (cps)at 25E	<5
Specific gravity at 20	1.20-1.22
Appearance	Clear to opalescent
Stability	Semi- permanent
Manufacturing company	STERLING CHEMICALS

Table 2. Properties of sand

Properties of sand	Fine sand
d_{10}	0.33mm
d_{30}	0.43mm
d_{60}	0.51mm
specific gravity(G)	2.22
minimum dry density (gm/cc)	1.3847
maximum dry density (gm/cc)	1.6578
coefficient of curvature (C_c)	1.075
coefficient of uniformity (C_u)	1.54
I.S soil classification	SP

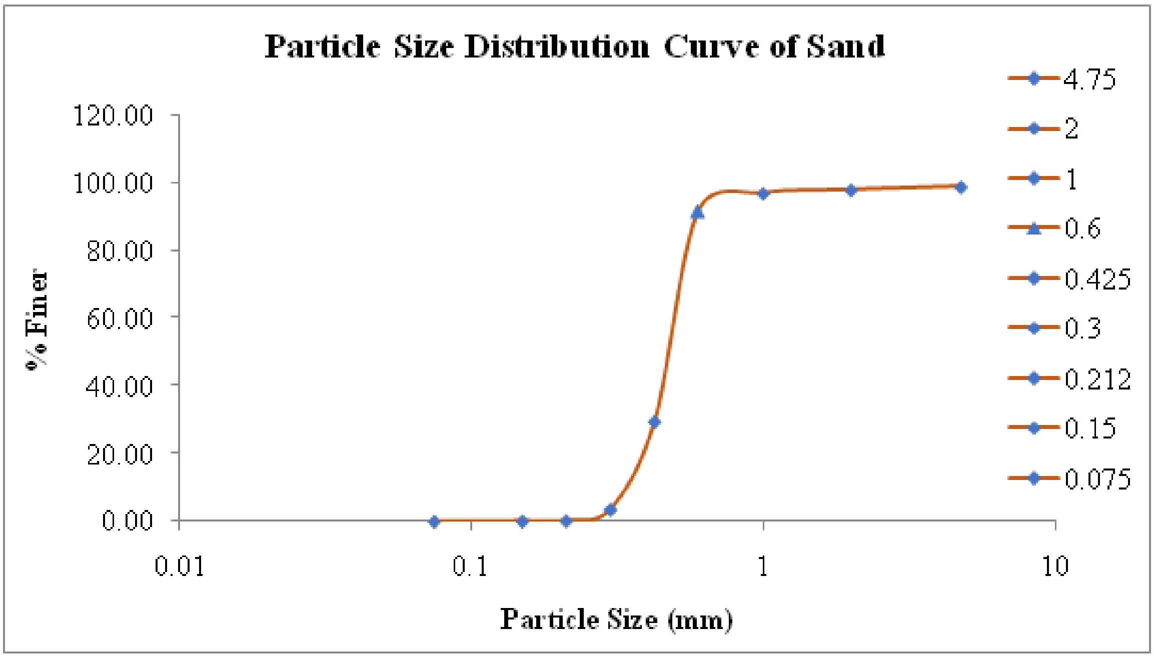

Fig.1 particle size distribution curve

Table 3. Properties of calcium hydroxide

Calcium hydroxide	**Ca $(OH)_2$**
Molecular weight	74.093 g/mol
Appearance	White powder
Odor	Odor less
Density	2.21 g/cm³, solid
Melting point	580°
Acidity	12.63 first 11.57 second
Solubility in water	1.89 g/l at 0° 1.73 g/l at 20° 0.66 g/l at 100°

Preparation of colloidal silica grout

In this research, water and colloidal silica were initially diluted and mixed in a grout mixer having stirrer rotating at @ 6000 RPM for 30 seconds. Calcium hydroxide (Ca $(OH)_2$) were added in specified proportions and mixed for an additional 30 seconds to get the homogeneous grout.

EXPERIMENTAL STUDY

The research encompasses a thorough assessment of diverse physical, rheological, and strength properties inherent in colloidal silica-based grout. Physical properties including gelification time, specific gravity, true permeation test, pH, and syneresis testswere performed. Rheological properties mainly viscosity was also meticulously analysed. Furthermore, strength properties including the needle penetration resistance test, unconfined compression strength test, and Adherent washout strength testswere conducted.

Gelation time:Gelation Time is that time at which the grout has acquired sufficient physio-chemical bond so that it reveals no obvious deformation on subject to disturbance.following the Barbadette (1955) standard method, is assessed by conducting a deformation test on a 100 ml grout sample in a 50 mm diameter beaker placed in three Position (horizontal, inclined and inverted) to access the time for partial and full gelation. (see fig. 1)

specific gravity: The specific gravity of grout is determined using the mud balance method(see fig.2)across water to colloidal silica (W/CS) ratios of 0.5, 1, and 2. Grout samples are placed in a 140cc cup attached to a calibrated metal beam, balanced by adjusting a sliding weight, and specific gravity is recorded upon achieving equilibrium.

pH: Measurements were conducted using a Hanna Instruments pH meter calibrated with a standard 7 pH buffer solution for accuracy. A pH-sensitive glass electrode and reference electrode immersed in the grout provided realtime pH readings, ensuring precise monitoring throughout the study.

Figure 1. Gel time

Figure 2.Mudbalance

Syneresis: The internal shrinkage in chemically gelled grout resulting from water expulsion, affects bonding and pore stability. Measured by volume change from gel to set time, syneresis in grout was monitored using a water displacement method over intervals: 1, 3, 28 & 90 days, providing insights into stability and performance (see fig. 3)

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Brookefieldviscometer test: Viscosity of the grout was determined using a brookefield RVT Viscometer (see fig. 4), which assesses the torque needed to rotate an immersed spindle within the fluid. This instrument employs a spindle driven by a synchronous motor through a calibrated spring, with the deflection of the spring reflected by a pointer and dial. By utilizing speeds of 2.5, 5, 10, and 20 rpm, along with interchangeable spindles, viscosity measurements can be obtained across a diverse range of viscosity levels using Michka method (Shroff and Shah).

Figure 3: Syneresis **Figure 4. Brookefield RVT viscometer**

The needle penetration resistance test: To assess the gel's resistance by penetrating needle under a specific weight in grams. This weight is exerted on a vicat wooden needle with a cross-sectional area of 1.18 cm², which is inserted to a depth of 2 cm into 100 cc of gel placed after the gelation period. (see fig.5)

Figure 5. Needle penetration resistance test **Figure 6.UCS Test**

Unconfined compressive strength (UCS), Testing involved cylindrical specimens prepared in PVC pipes, cured for durations ranging from 3,7,28 and 90 days. Tests were conducted using a strain-controlled triaxial testing machine under unconfined compression conditions, (see fig. 6) providing insights into stress-strain behaviour and specimen resilience under compressive stress.

Adherent washout strength: Tests were carried out on poorly graded sand specimens in mould, measuring 10 cm in diameter and 30 cm in height, after grouting with the optimal dosage. Following a 7-day curing period, a washout strength test was conducted, applying pressure incrementally at intervals of 0.5 kg/cm² up to 8 kg/cm², while recording permeability measurements. The aim was to evaluate the resistance of the grouted sand specimens to washout and analyse their permeability characteristics under varying pressure conditions. (see fig.7)

Figure 7. Adherent washout strength

RESULT ANALYSIS & DISCUSSION

Physical characteristics

Gel Time

Fig. 1 shows effect of different concentrations of reactant on gel time of colloidal silica grout & Table 4. Shows gel time. It can be seen that the reactant gel time decreasesas the percentage concentration of reactant increases.

Table 4. Gel time of colloidal silica grout

W/CS Ratio	concentration% of the reactant Ca(Oh)2	GEL TIME(min)
W/CS=0.5:1	0.4%	19
	0.6%	15
	0.8%	IG
W/CS=1:1	0.4%	49
	0.6%	31
	0.8%	IG
W/CS=2:1	0.4%	120
	0.6%	48
	0.8%	18

IG= Immediate gel

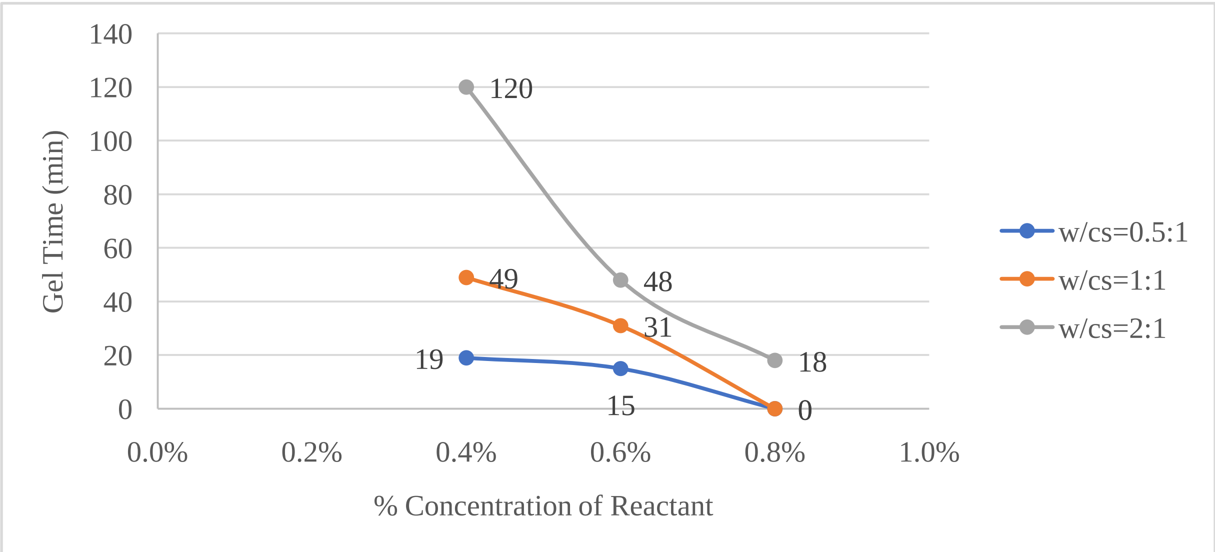

Fig.8 effects of different concentration of reactant on gel time for raw colloidal silica grout (w/cs=0.5, 1, 2)

The gel time decreases with percentage concentration of reactant increases in (W/CS = 0.5:1, 1:1, 2:1). The gel time decreases from about 120 min to 0 min for different concentrations of reactant.

The different percentage concentrations (0.4, 0.6, 0.8) of calcium hydroxide used in the colloidal silica grout, immediate gellification was observed for (W/CS=0.5:1& 1:1) with 0.8%concentration.

pH value

Fig. 9 shows the effect of different concentrations of reactant on the pH value of raw colloidal silica gel. The pH value increases from 9.6 to 9.79 as the percentage of concentration of reactant increases from 0.4% to 0.8% with different (W/CS = 0.5:1, 1:1, 2:1). The pH is maximum at (w/cs=2:1).

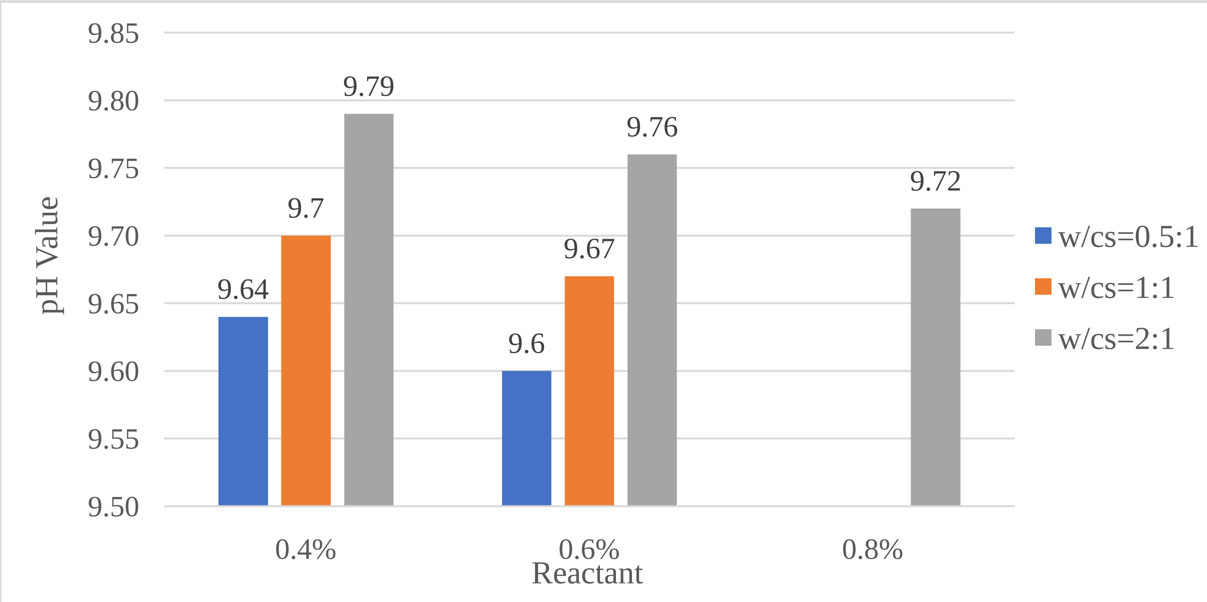

Fig.9. Effect of concentration of reactant on pH(w/cs=0.5, 1, 2)

It can be seen that pH value increases with increases in w:cs ratio. The pH value decreases with concentration of reactant increases at different w:cs ratios. In different percentage concentrations (0.4, 0.6, 0.8) of (W/CS=0.5:1& 1:1) concentration, 0.8% found immediately gel.

Specific Gravity

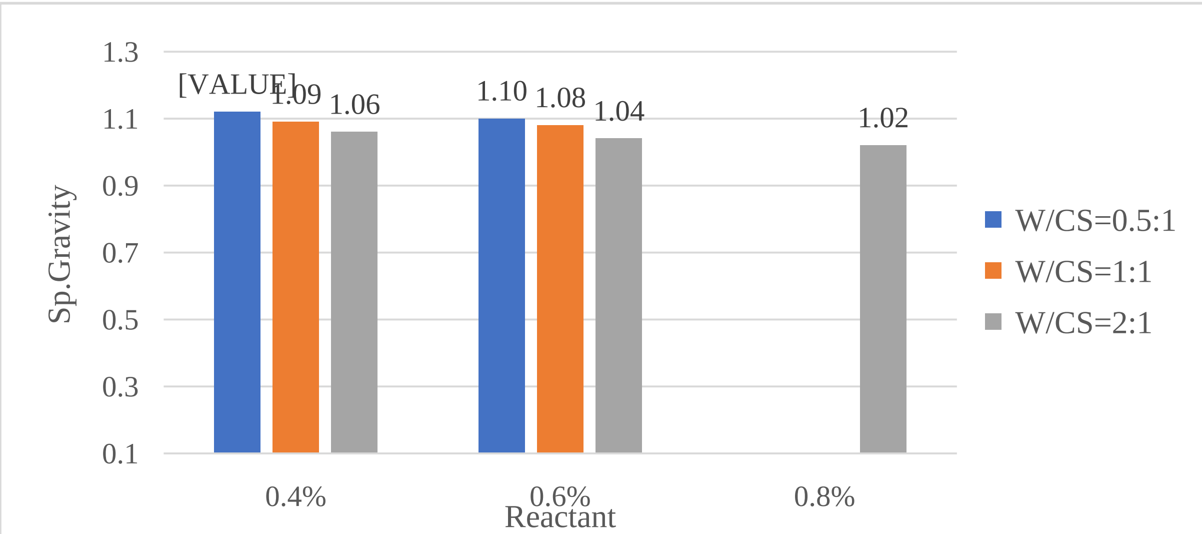

Fig. 10 shows the relation of specific gravity of colloidal silica grout with different w:cs ratios of 0.5:1, 1:1, and 2:1 with different concentrations of reactant Ca $(OH)_2$.

Fig. 10 Sp. Gravity Vs Different Concentration of Reactant of Raw Colloidal Silica Grout with Different (W:CS) ratio
It is observed that the specific gravity of colloidal silica grout is increasing with decreased w:cs ratio of grout. At a lower w:cs ratio, specific gravity is greater, and at a higher w:cs ratio, it can decrease. It can be concluded that increasing a percentage concentration of reactant, the specific gravity of grout decreases.

True permeation Test
Fig. 11. shows depth of penetration (cm) of grout Vs percentage concentration of reactant with different w:cs ratio. In true permeation, it can be observed that depth of penetration increases with an increased w:cs ratio. At lower w:cs ratio penetration at lower depth in the formation & at higher w:cs ratio depth of penetration is more as compared to lower w:cs ratio. figure shows that at w:cs ratio 0.5 penetration depth is lower as compared to w:cs ratio 2.

In different percentage concentrations (0.4, 0.6, 0.8) of (W/CS=0.5:1& 1:1) concentration, 0.8% found immediately gel.

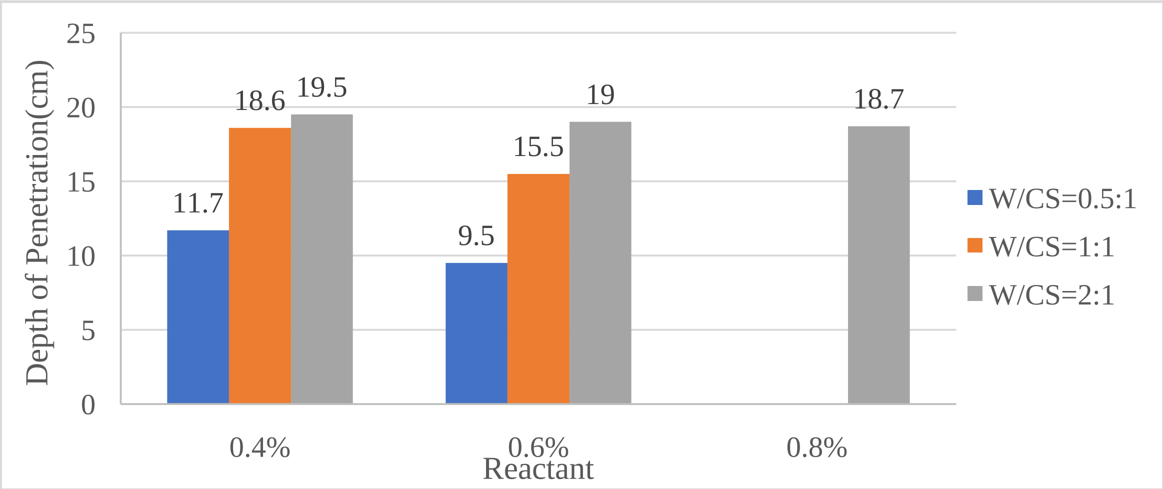

Fig. 11. Depth of Penetration V/s Reactant for Raw Colloidal Silica Grout for Fine Sand with Different W:CS Ratio.

Syneresis
Fig.5 shows the effect of time on percentage syneresis of raw colloidal silica grout in an air-dried cured curing condition. The percentage syneresis at 1days, 3days, 28days and 90days for different w:cs ratios of0.5:1, 1:1, and 2:1 with (0.4%) concentration of reactant. It can be concluded from the graph that percentage syneresis increases with time. The percentage syneresis at 1days is 11% to 23% for concentration of reactant. The percentage syneresis at 3days is 30% to 40% for concentration of reactant. The percentage syneresis at 28days is 49% to 57% for concentration of reactant. The percentage syneresis at 90days is 63% to 72% for a 0.4% concentration of reactant.

The percentage syneresis of raw colloidal silica grout increase as water to colloidal silica ratio increases.

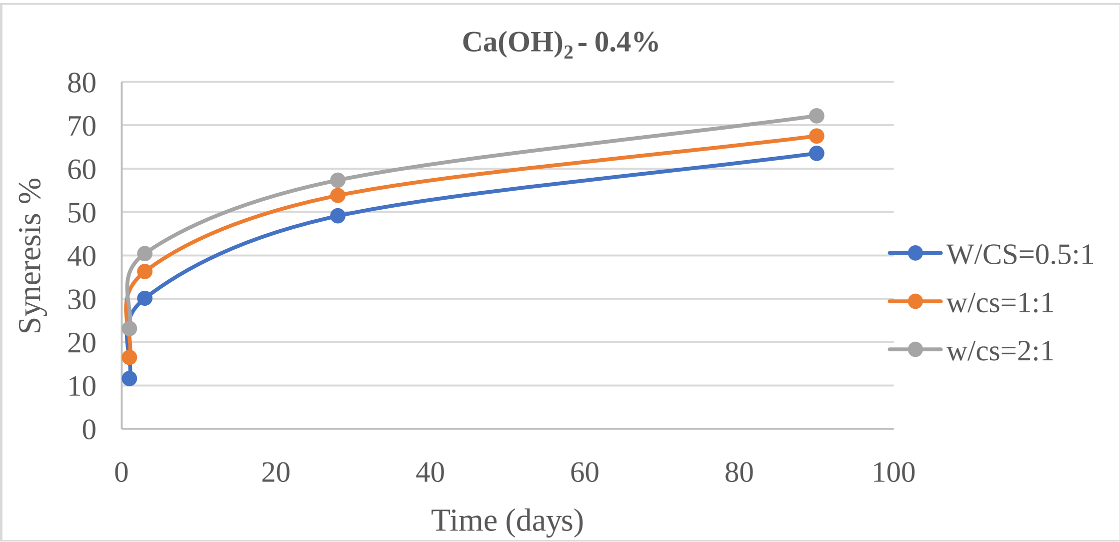

Fig. 12: syneresis of raw colloidal silica grout at various time interval (w:cs-0.5, 1, 2)

Rheological Characteristics

Flow curves at Different Time intervals

Figs.13 and 7show the plotting of shear rate v/s shear stress (flow curves) for calcium hydroxide reactant with colloidal silica grout with water to a colloidal silica ratio of 0.5:1, 1:1.

All the grout is prepared in single batches, from which it is poured into different beakers to take readings at different time intervals. Looking at all the curves, it is observed that initially Newtonian fluid changes to grout, which behaves as pseudo-plastic.with progress of time, grout bends towards the shear stress axis. At gel time, grout behaves as Binghamian fluid. In the plotting of shear rate v/s shear stress, it is observed that, as time increases, the curve deflects more towards the shear stress axis at different time intervals.

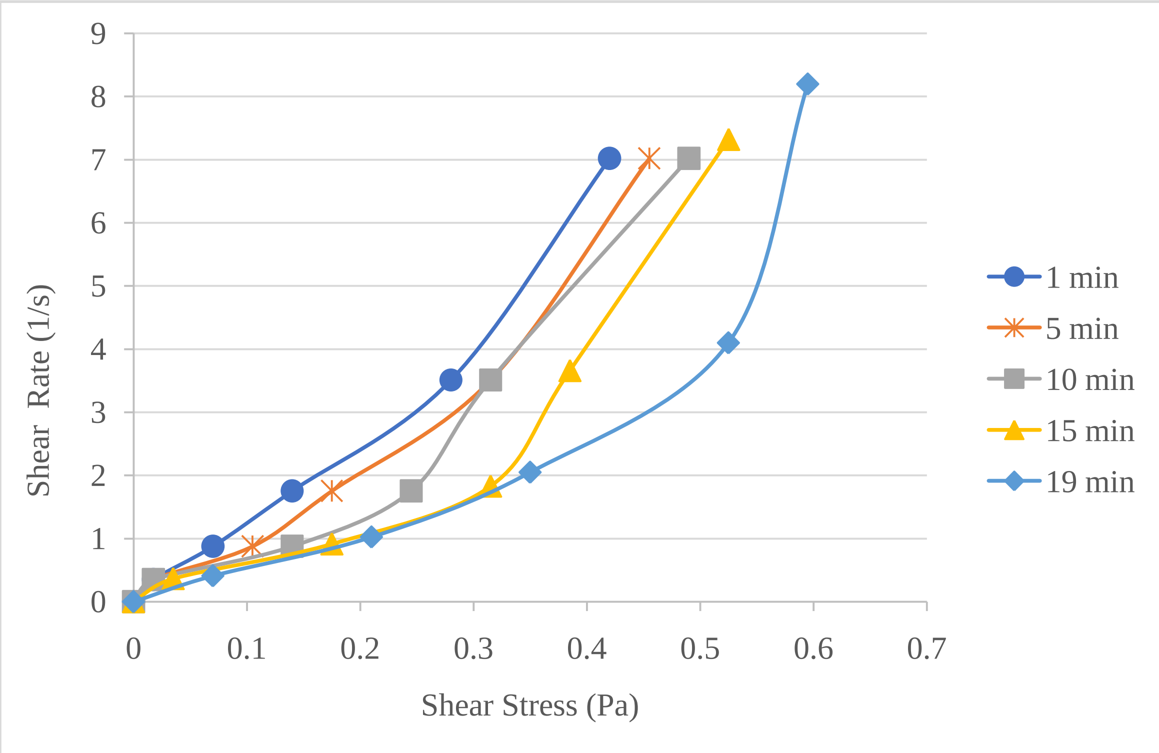

Fig.13: Flow curves of colloidal silica grout (Ca (OH)$_2$ =0.4%, W/CS=0.5:1)

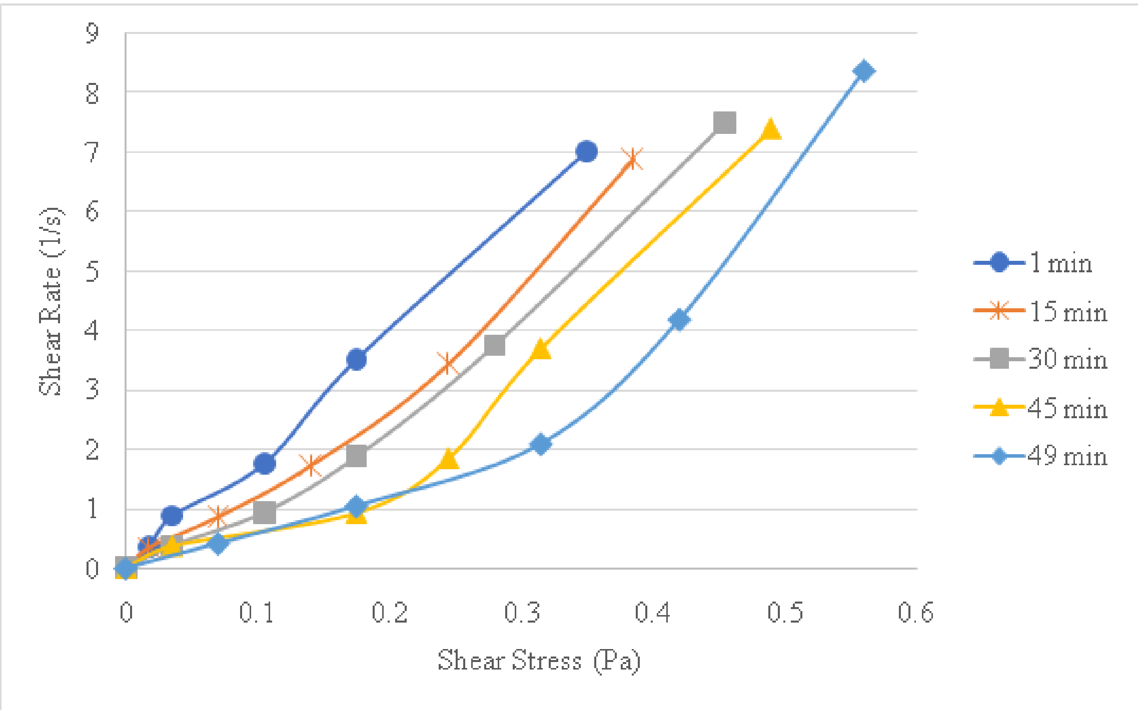

Fig.14: Flow curves of colloidal silica grout (Ca (OH)$_2$ =0.4%, W/CS=1:1)

Time-Apparent Viscosity Characteristics

Fig.15 shows time v/s apparent viscosity curve for reactant with colloidal silica grout at w/cs=0.5:1, 1:1.The viscosity increases with increase in time and decrease in water to colloidal silica ratio. At 0.4% concentration of reactant (W:CS=0.5:1) gives the highest initial viscosity at time t=1 min.

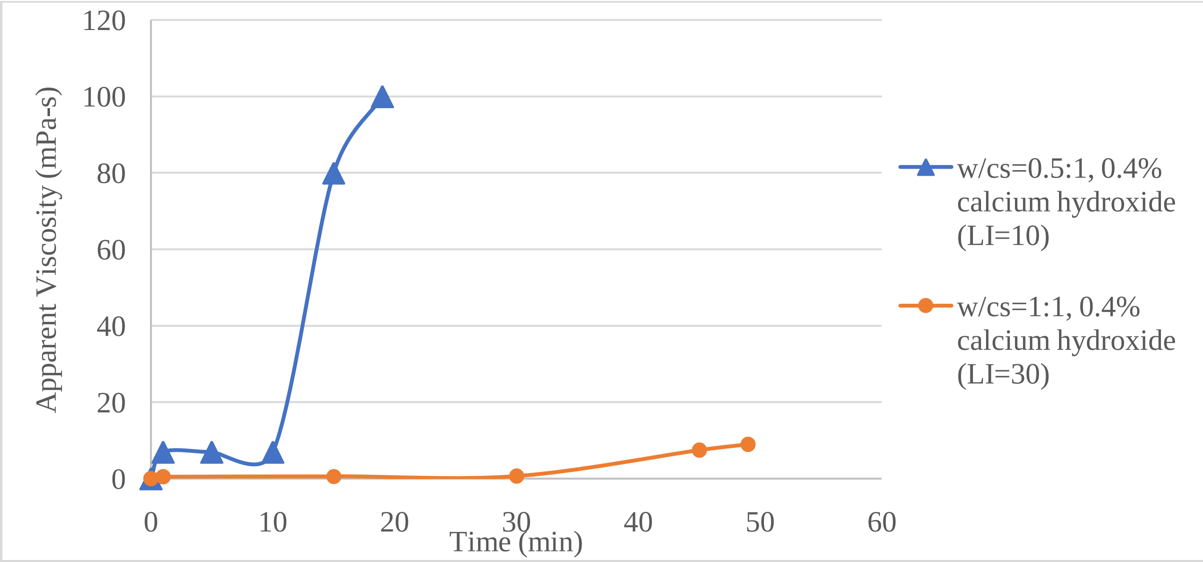

Fig.15: Time -Apparent Viscosity Characteristics for colloidal silica grouts (W/CS=0.5:1 & 1:1)

Yield value of colloidal silica grout

Fig.16 shows the variation of yield stress with time for colloidal silica grouts. For colloidal silica grouts, the yield stress increases with time. Yield value is about zero initially.

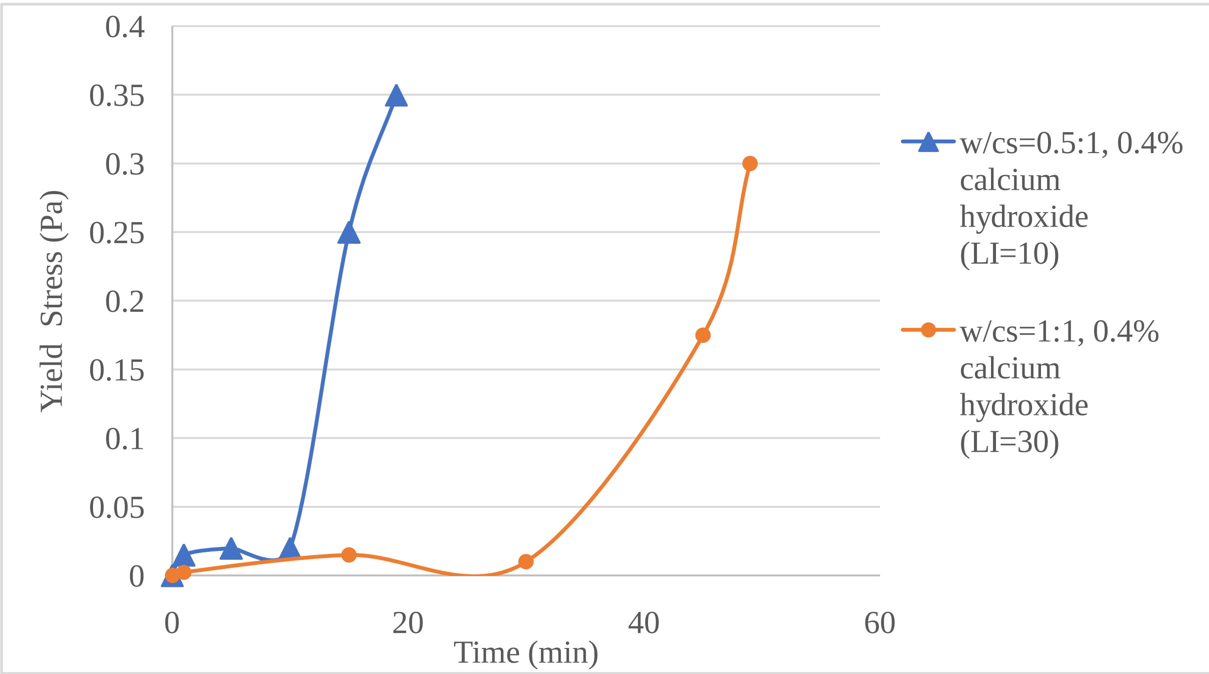

Fig.16: Yield stress V/s Time for colloidal silica grout (W/CS=0.5:1 & 1:1)

Time Strength Characteristics

Needle Penetration Resistance of Colloidal Silica Raw Grout

Fig.17 shows needle penetration resistance of colloidal silica raw Grout at a 0.4% concentration of the reactant at water to colloidal ratios like 0.5:1, 1:1, and 2:1, in which w/cs=0.5 exhibits a maximum NPR valueof 88.6 kPa while w/c=2 has a minimum NPR valueof 52.9 kPa. The needle penetration value decreases with an increase in water to colloidal silica ratio.

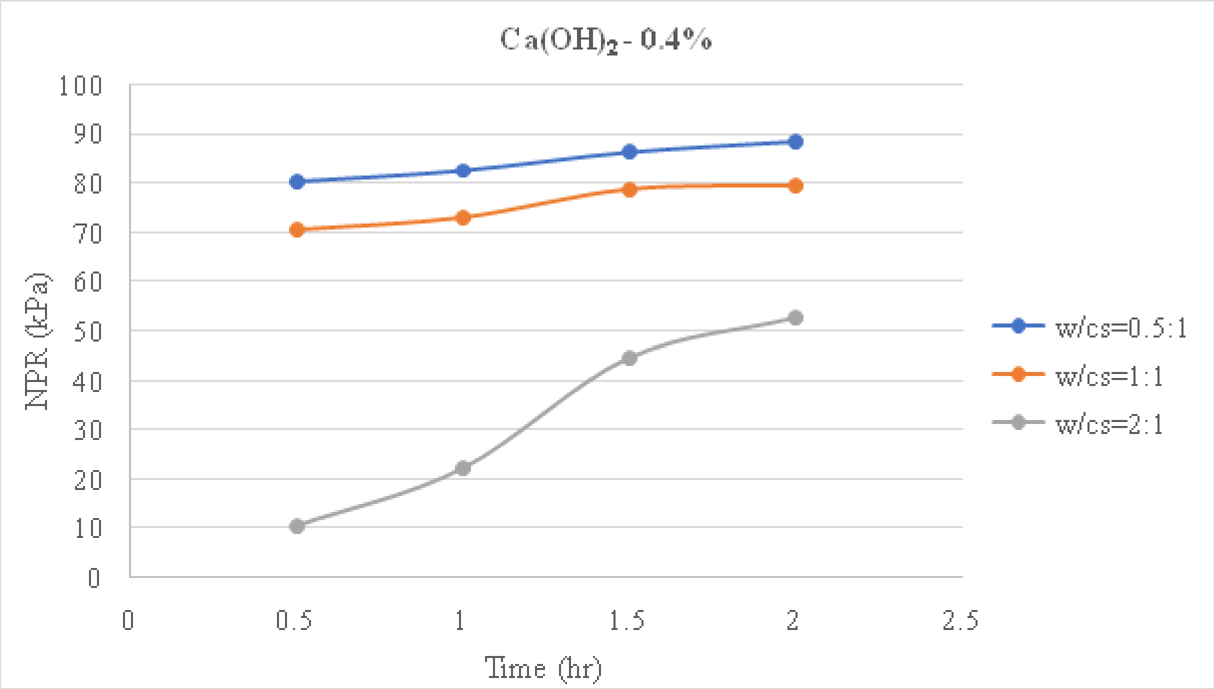

Fig. 17: NPR of Raw Colloidal Silica Grout with Concentration 0.4 %,
At (W/CS =0.5:1, 1:1, 2:1)

Time UCS Characteristics of Grouted sand for colloidal silica grout

Fig. 18 and 19Show the stress-strain curve for grouted sand dry-cured at w/cs=0.5,1 for the optimum dose of reactant**.** Fig.20 shows UCS v/s curing time. It is observed that the peak stress is higher at 90days and lower at 3 days. The stress-strain curve shows the elastoplastic behaviour. The UCS strength increases as curing time increases.

The unconfined compressive strength of grouted sand in dry and wet cured condition. The strength increases with an increase in curing time from 3 to 90 days and decreases in w:cs for 0.4% concentration of reactant; it gives maximum strength.

Grout mix with 0.4% calcium hydroxide reactant at w:cs=0.5:1 gives maximum strength of 71.43 kPa at 90days. Grout mix with 0.4% calcium hydroxide reactant at w:cs=1:1 gives a minimum strength of52.68 kPa at 90days for grouted sand.

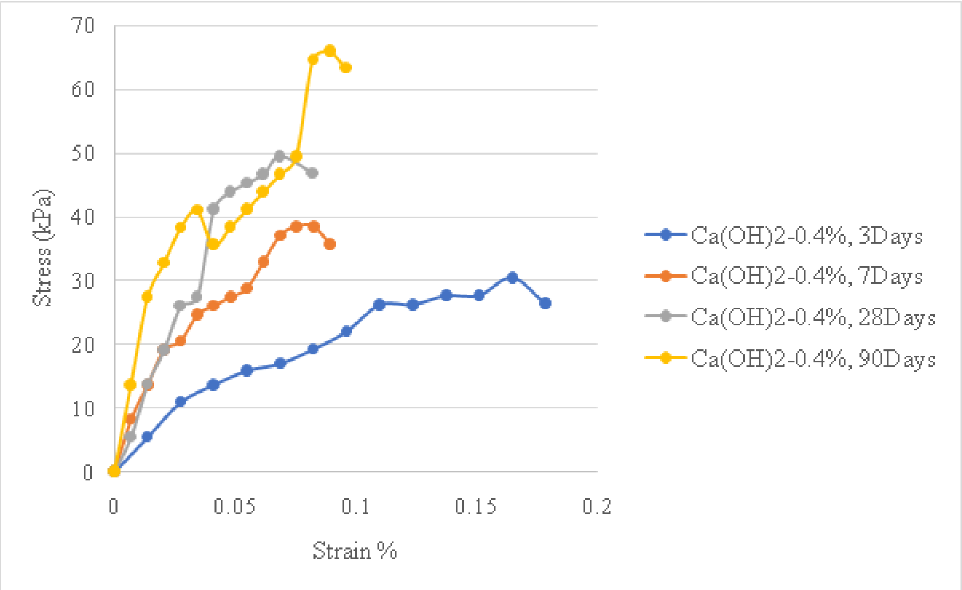

Fig.18. 90 Days stress-strain curve of Reactant 0.4% for Grouted sand wet condition (w/cs=0.5)

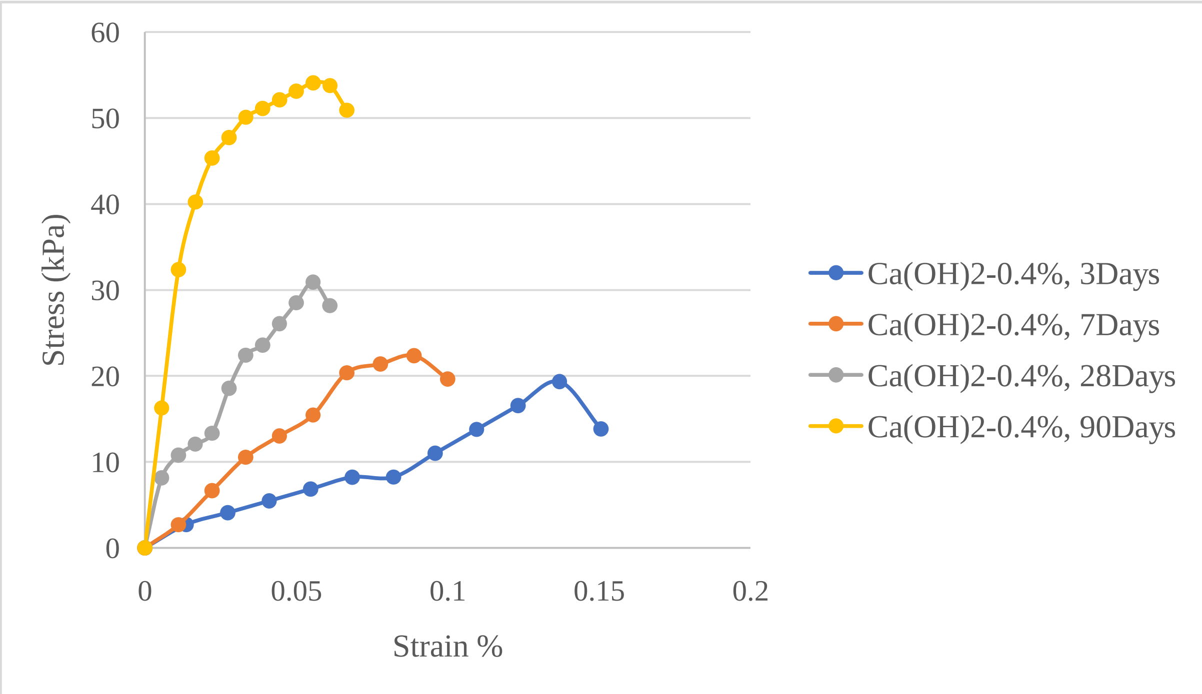

Fig.19. 90 Days stress-strain curve of Colloidal silica Grouted sand in wet cured Condition (w/cs=1:1)

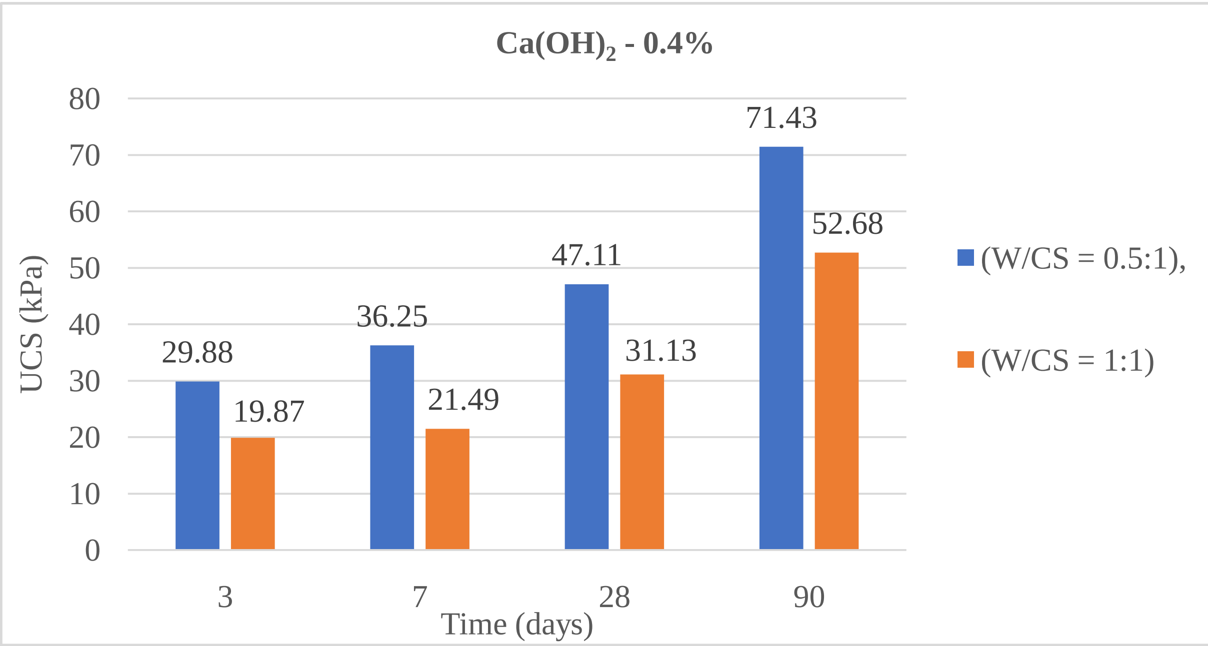

Fig.20. UCS v/s Time of curing for grouted sand (W/CS=0.5, 1)

Adherent washout strength for colloidal silica grout

Fig.14 shows variation in permeability of grouted sand at different hydraulic gradients. The test was conducted after 7 days of curing. As the w:cs ratio decreases, water pressure, time, and strength increase. Adherent washout strength is found out by measuring pressure, and pressure is converted to hydraulic gradient. Permeability of grouted sand increases as pressure increases. From the curve, it is observed that maximum adherent washout strength is 259.97kPa at w:cs=0.5:1 with reactant, and minimum strength is 79.61kPa at w:cs=1:1.

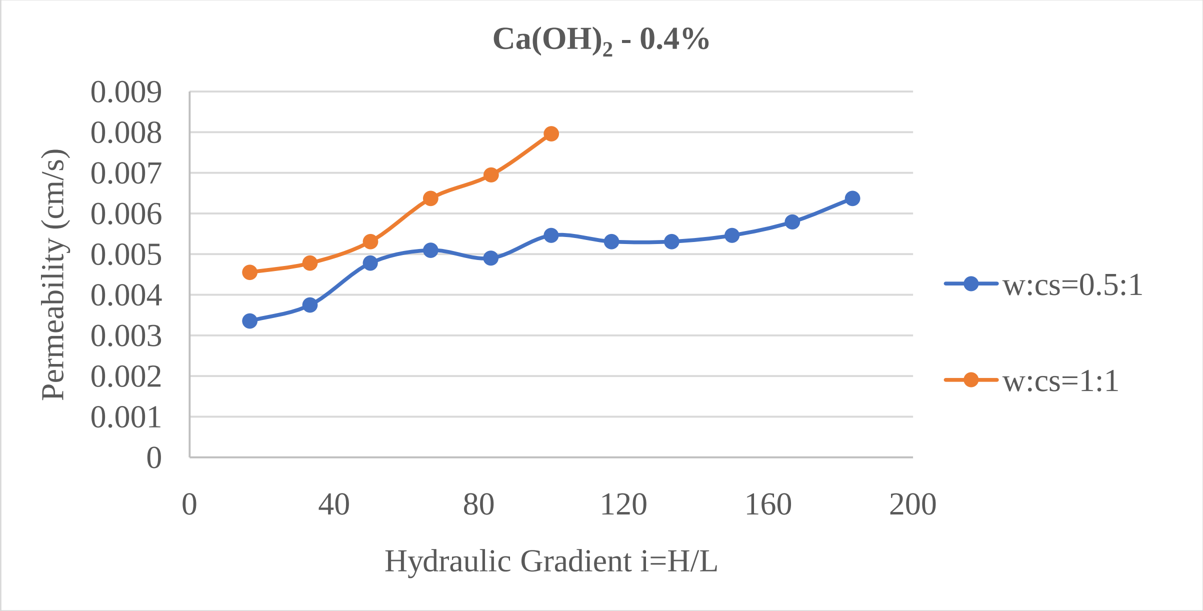

Fig.21: permeability V/s hydraulic gradient of colloidal silica grouted sand with Ca $(OH)_2$ (W:CS=0.5, 1:1)

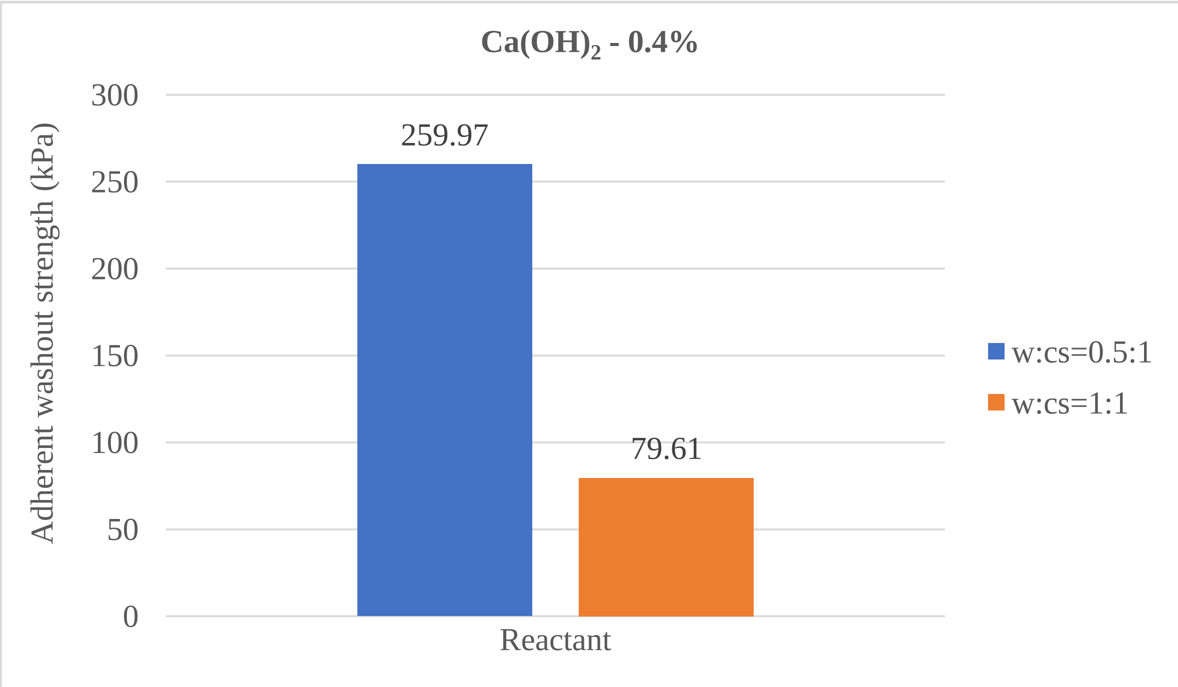

Fig.22: Adherent washout strength V/s Reactant (W:CS=0.5:1, 1:1)

CONCLUSION

Physical Properties

Gel time decreases as the percentage concentration of reactant increases in the ratio (w/cs=0.5:1, 1:1 and 2:1).
The gel time decreases from about 120 min to 0 min for different concentrations of reactant at (w/cs = 0.5:1, 1:1 and 2:1).

The pH value increases from 9.6 to 9.79 with different concentrations of reactant and increases from 0.4% to 0.8% with different (w/cs=0.5:1, 1:1, 2:1) ratios. It can be seen that pH value increases with increases in w:cs ratio. The pH value decreases with concentration of reactant increases at different w:cs ratios.

Specific gravity is increasing with decreased w/cs ratio of grout. At a lower w/cs ratio, Sp. G is greater, and at a higher w/cs ratio, it can be decreased. It can be concluded that increasing the concentration of reactant Sp. G in grout decreases.

In true permeation, it can be observed that depth of penetration increases with an increased w:cs ratio. At lower w:cs ratio penetration at lower depth in the formation & at higher w:cs ratio depth of penetration is more as compared to lower w:cs ratio.

Syneresis increases with time (days). It can be observed that % of syneresis at 1 day is 11 to 23%. At 3 days, 30 to 40%. At 28 days, 49 to 57%. At 90 days, 63 to 72%. At (w/cs = 0.5:1, 1:1, 2:1). It can be observed that the % syneresis of raw colloidal silica increases as the w:cs ratio increases.

Rheological Characteristics

Time-viscosity Characteristics Including Flow Properties

It was observed that colloidal silica grout initially undergoes Newtonian changes to pseudo-plastic, with deflection of flow curves increasing towards the shear stress axis with progress of time.

For colloidal silica grouts, the yield stress increases with time. Yield value is about zero initially.
The viscosity increases with an increase in time and decreases in the water-to-colloidal silica ratio.
At 0.4% concentration of reactant (W:CS=0.5:1) gives the highest initial viscosity at time t = 1 min.

Time- Strength Characteristics

TheNeedle penetration resistance(NPR) value for all colloidal silica raw grout after 1 hour of jellification is beyond 88.6 kPa with w:cs=0.5:1. The NPR value decreases with an increase in the water to colloidal silica ratio.

The unconfined compressive strength of grouted sand. The strength increases with an increase in curing time from 3 to 90 days and decreases in w:cs for 0.4% concentration of reactant; it gives maximum strength. Grout mix with 0.4% calcium hydroxide reactant at w:cs=0.5:1 gives maximum strength of 71.43 kPa at 90 days.

Grout mix with 0.4% calcium hydroxide reactant at w:cs=1:1 gives a minimum strength of 52.68 kPa at 90 days for grouted sand. Permeability of grouted sand increases as pressure increases. From the curve, it is observed that maximum adherent washout strength is 259.97 kPa at w:cs=0.5:1 with reactant, and minimum strength is 79.61 kPa at w:cs=1:1 ratio.

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