

# Development of Ultrafine Slag-Based Grout with Magnesium Oxide and Calcium Hydroxide Activator

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## ABSTRACT

**This. aims to enhance ultrafine slag-based grout by incorporating magnesium oxide, calcium hydroxide activator, and polycarboxylate ether super slump retainer 50% superplasticizer. Pure slag (Alcofine-1203) is solely utilized in the experiment, with additional components added at specific percentages relative to the slag weight: magnesium oxide (2.5%), calcium hydroxide activators (4%), and superplasticizer polycarboxylate ether super slump retainer (0.5%). These compositions are tested across different water-to-solids (W/S) ratios of 0.8, 2, and 5. Various physical, rheological, and strength properties including gelification, specific gravity, bleeding potential, fluidity, syneresis, true permeation, viscosity, needle penetration resistance, unconfined compression strength, and washout strength are comprehensively analysed in this study. It is observed that an increase in the W/S ratio results in a decrease in specific gravity, strength, afflux time, and viscosity, while it leads to an increase in gel time, bleeding, syneresis, pH, and penetration of grout.**

**Index Terms:** Slag based grout, Particulate, Permeability, Strength,

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## INTRODUCTION

Civil engineers frequently consider grouting to address foundation issues, involving the injection of cementitious slurries or similar substances into inaccessible areas beneath structures.(1) Grouting materials fall into two main categories: (a) suspension-type or particulate grouts (coarse grout), and (b) solution-type or chemical grouts (fine grout). Particulate grouting materials typically include cement, cement with bentonite clay to reduce bleeding, and cement with admixtures for further design properties. Natural pozzolans like finely ground shale, pumicite, diatomite, and artificial pozzolans such as fly ash (a coal combustion byproduct) and blast furnace slag are commonly used.(2)Recently, Alcofine-1203, a form of blast furnace slag, has gained attention as a grouting material. In ultrafine slag-based grout, the finely ground slag, boasting a remarkable fineness of 12,000 cm<sup>2</sup>/kg, plays a pivotal role in enhancing its soil penetration capabilities by virtue of its expanded surface area. While raw slag lacks self-binding properties, it can be activated by alkaline additives, which dissolve silicate and aluminate compounds to form calcium silicate hydrates (C-S-H) and calcium aluminate hydrates (C-A-H). These hydrates enhance the strength and binding capacity of slag-based grouting materials.(3)

D.H.Kim et al, investigate the enhancement of grout strength by incorporating blast furnace slag powder and reinforcing fibres. Their study highlights significant improvements in compressive strength, particularly when using aramid fibers.(4)E.Tkaczewska et al, the impact of different superplasticizers on fly ash blended cement, underscoring the superior efficacy of polycarboxylate ethers over conventional varieties. They emphasize the reduction in water content and enhancement of hydration heat, setting time, and mechanical properties.(5)Yijie Zhang et al. explore tailored anti-scouring grouts for karst water control, utilizing a phosphate cement-derived condensate with diverse additives, highlighting the influence of MgO-to-KH<sub>2</sub>PO<sub>4</sub> ratio, waterglass, and fly ash levels on crucial properties, offering guidance for efficient karst conduit sealing.(6) Ketaki B. Patel et al. investigate eco-friendly cement-based grouts, employing various activators and slag blends, favouring NaF activation for enhanced strength and viscosity. Their study offers insights into the rheological characteristics of chemically activated microfine slag cement grouts(7).

This study examines the enhancement of grout properties through the use of slag-based grout with magnesium oxide (2.5% by slag weight) and calcium hydroxide (4% by slag weight) activators, alongside a polycarboxylate ether super slump retainer (PCE-SSR) (0.5 % by slag weight) superplasticizer. To overcome the inherent challenge of the slag's limited self-binding properties, magnesium oxide (MgO) and calcium hydroxide (Ca (OH)<sub>2</sub>) are introduced as activators. These compounds catalyse the hydration process, fostering robust strength development within the grout matrix. Furthermore, the inclusion of a PCE-SSR (Polycarboxylate Ether - Slump Retention) superplasticizer serves to counteract flocculation during the mixing phase, ensuring uniform dispersion and optimizing the slag's reactive surface area. This synergy promotes efficient interaction between the slag and activators, culminating in a meticulously crafted grout solution tailored for superior performance in diverse construction settings. The effectiveness of these compositions is evaluated across varying water-to-slag (W/S) ratios of 0.8, 2, and 5.

### MATERIALS AND METHOD

#### Materials

##### Alcofine-1203 or GGBFS

Ground Granulated Blast Furnace Slag (GGBFS), obtained from Ambuja Cements Limited (Adani Group), is a byproduct of iron and steel production, created by quenching melted blast furnace slag with water. Widely substituted for Portland cement in concrete production, GG-BFS enhances durability, reduces heat of hydration, and improves workability, promoting sustainable construction by repurposing industrial waste. Table 1 describes the physical properties &

Table 2 describe the chemical composition of slag.

**Table 1 Physical properties of Slag**

Property	Unit	Value
Average particle size	µm	4 to 6
D <sub>10</sub>	µm	1.9
D <sub>50</sub>		4.5
D <sub>85</sub>		8.2
D <sub>90</sub>		8.9
Fineness	cm <sup>2</sup> /kg	12000
Bulk density	(kg/m <sup>3</sup> )	685
Specific gravity	-	2.81

**Table 2 Chemical composition of slag**

Chemical	%
SiO <sub>2</sub>	34.8%
Al <sub>2</sub> O <sub>3</sub>	21.6%
Fe <sub>2</sub> O <sub>3</sub>	1.8%
CaO	34.0%
SO <sub>3</sub>	0.13%
MgO	6.6%

#### Magnesium oxide

Rawpharma Biz Private Limited, located in Ahmedabad, Gujarat, procures magnesium oxide, This compound exhibits a specific gravity of 3.70 and a pH range of 8–10. Table 3 Describe the chemical composition of MgO.

**Table 3 Chemical composition of MgO**

Sr. no	Specification	Result
1	Magnesium oxide (MgO)	85-87 %
2	Silica (SiO <sub>2</sub> )	6.50%
3	Calcium Oxide (CaO)	3.00%
4	Aluminium oxide (Al <sub>2</sub> O <sub>3</sub> )	0.25%
5	Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	0.25%
6	Loss of Ignition (LOI)	3.00%
7	Mg content	5.2%
8	Mesh size	200 mesh fine powders

### Calcium hydroxide

Jay Chemicals, located in Nandesari, Vadodara, Gujarat, supplies calcium hydroxide, this compound exhibits a specific gravity of 2.2 and a pH range of 12.4 - 12.8. And Table 4 Describe the Chemical Composition of Ca(OH)<sub>2</sub>.

**Table 4 Chemical composition of Ca(OH)<sub>2</sub>**

Sr. no.	Nature of hydrated lime	Free flowing fine white powder
1	Assay as Available Ca(OH) <sub>2</sub>	70.00+
2	Active CaO%	+52
3	Magnesia as MgO	2
4	Acid Insoluble As (SiO) <sub>2</sub>	4
5	iron	In trace
6	Aluminium	In trace
7	Sieve test	In trace

### Polycarboxylate Ether-super slump retain 50%

Fair Mate Chemical Private Limited, Vadodara supplies polycarboxylate superplasticizers, which confer numerous benefits including enhanced slump retention, increased early and ultimate strength, improved durability, and reduced permeability in concrete. These superplasticizers are often preferred over traditional lignosulfonate or naphthalene-based alternatives due to their lower water-to-cement ratio and reduced CO<sub>2</sub> emissions, making them environmentally friendly. Table 5 Describe the properties of PCE-SSR.

**Table 5 properties of PCE-SSR**

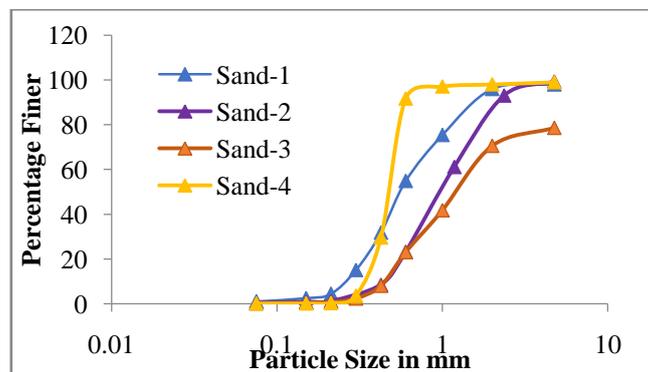
Parameter name	Q. C standard		Q. C check and ok
	Water white colour liquid	-	
Physical appearance	Water white colour liquid	-	Clear cover viscous liquid
Specific gravity	1.100	1.140	1.109
Solid content	48.0%	52.0%	48.92%
pH	5.0	7.5	6.1

### Sand

The test procedure utilized four types of sand, with their respective gradation curves illustrated in

Fig. 1. Additionally,

Table 6 provides an overview of the properties associated with each type of sand.



**Fig. 1 Particle distribution size curve of sand**

**Table 6 properties of sand**

Property of sand	Sand-1	sand-2	sand-3	sand-4
Specific gravity	2.55	2.68	2.62	2.63
Type of sand as per sieve analysis	SP- poorly graded sand			
Coefficient of Uniformity (C <sub>u</sub> )	2.71	2.63	1.49	2.70

Coefficient of Curvature ( $C_c$ )	0.94	0.96	1.108	1.1
Maximum Density ( $\text{gm/cm}^3$ )	1.83	1.75	1.67	1.87
Minimum Density ( $\text{gm/cm}^3$ )	1.5	1.50	1.43	1.68
Density ( $\text{gm/cm}^3$ )	1.62	1.61	1.52	1.72
Angle of internal friction in degree (from Direct shear box test)	35°	35°	38°	36°

### Preparation of grout using ultrafine slag-based materials

In this research, water and polycarboxylate ether (PCE) were initially diluted and mixed in a grout mixer for 30 seconds. Following this, magnesium oxide (MgO) and calcium hydroxide ( $\text{Ca(OH)}_2$ ) were added in specified proportions and mixed for an additional 30 seconds. Subsequently, slag was added in three equal portions, and the blending process was completed within 3 minutes. Finally, the entire mixture underwent a final mixing cycle lasting 1 minute.

### Experimental details

The research encompasses a thorough assessment of diverse physical, rheological, and strength properties inherent in slag-based grout. Physical properties including gelification time, specific gravity, bleeding potential, true permeation test, pH, and syneresis test are scrutinized. Rheological properties such as fluidity and viscosity are also meticulously analysed. Furthermore, strength properties including the needle penetration resistance test, unconfined compression strength test, and Adherent washout strength test are conducted.

Gelation time, 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. For water-cement ratios exceeding 2, a larger container is employed to form a 7 cm grout cake post-bleeding, with subsequent monitoring at 15-minute intervals until deformation ceases.(1) The specific gravity of grout is determined using the mud balance method (

Fig. 2) across water-solid (W/S) ratios of 0.8, 2, and 5. Grout samples are placed in a 140 cc cup attached to a calibrated metal beam, balanced by adjusting a sliding weight, and specific gravity is recorded upon achieving equilibrium.(8)



Fig. 2 Mud balance

Assessing bleeding potential is crucial for grout stability, aiming to prevent segregation and maintain its original properties. Grout is poured into a 1000 cc measuring cylinder, and the height of water above settled grout is measured at intervals up to 48 hours, with early readings taken more frequently to capture initial bleeding stages.(9)

The permeation test evaluates grout penetrability in various soil gradations under gravity. A glass cylinder filled with sand (26 cm height) is used to introduce 200 ml of grout, left undisturbed until gelation. After dismantling, the maximum grout penetration depth is measured for performance analysis, offering insights into its effectiveness in different soil types.(1)

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 (Fig. 4.11) provided real-time pH readings, ensuring precise monitoring throughout the study.(1) 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: 3, 7, 14, 28 & 90 days, providing insights into stability and performance.(10)

The fluidity of grout, evaluated via the Marsh cone test (See

Fig. 3), The afflux time, measuring how long it takes to fill a 1000 cc cylinder, determines fluidity based on viscosity, following the principles of the Hagen-Poiseuille law, where the time taken (in seconds) for a fixed volume (1000 cc) to pass through a capillary (0.47 cm) is directly proportional to the fluid's viscosity. The cone features a capacity of 2000 ml, a diameter of 15 cm, a height of 30 cm, and an orifice diameter of 4.75 mm.(11)

The viscosity of the grout was determined using a Brookfield RVT (See

Fig. 4), which assesses the torque needed to rotate an immersed element 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.(12)



Fig. 3 Marsh cone apparatus



Fig. 4 Brookfield RVT viscometer

The needle penetration resistance test assesses the gel's resistance by applying a specific weight in grams. This weight is exerted on a Vicat wooden needle with a cross-sectional area of 1.18 cm<sup>2</sup>, which is inserted to a depth of 2 cm into 100 cc of gel placed after the gelation period.(1)

Unconfined compressive strength (UCS) testing involved cylindrical specimens prepared in PVC pipes (See **Error! Reference source not found.**), cured for durations ranging from 3,7,14,28 and 90 days. Tests were conducted using a strain-controlled triaxial testing machine (See **Error! Reference source not found.**) under unconfined compression conditions, providing insights into stress-strain behaviour and specimen resilience under compressive stress.(13)

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/cm<sup>2</sup> up to 8 kg/cm<sup>2</sup>, 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.(1)



Fig. 5 UCS sampler



Fig. 6 UCS test on specimen

## RESULT AND DISCUSSION

### Physical properties

Assessing the durability of grout relies heavily on its physical properties. These evaluations provide crucial insights into the grout's performance and longevity.

#### Gelification time

Fig. 5 illustrates the W/S ratio increases, the gel time extends, indicating a more diluted mixture. This dilution results in lower reactant concentrations, greater particle separation, and a slower precipitation of hydration products. These combined factors lead to a prolonged gel time in slag-based grout.

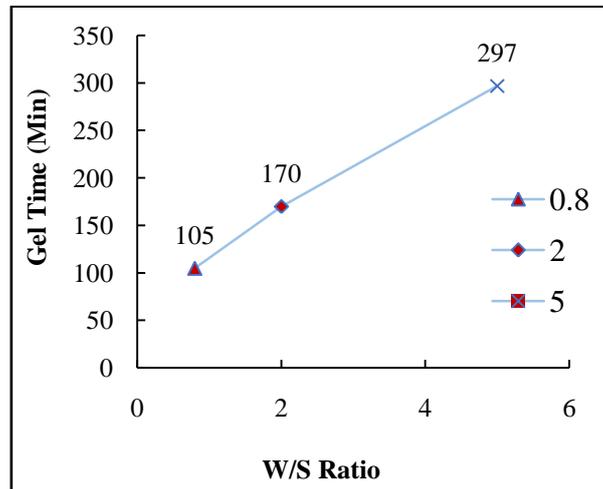


Fig. 5 Effect of W/S Ratio on Gel Time of Slag-Based Grout

### Specific gravity

Fig. 6 illustrates the W/S ratio increases, the specific gravity of slag-based grout decreases. This decrease is primarily due to the dilution effect of the additional water, which has a significantly lower density than slag. Consequently, the overall density of the mixture is reduced, leading to a lower specific gravity.

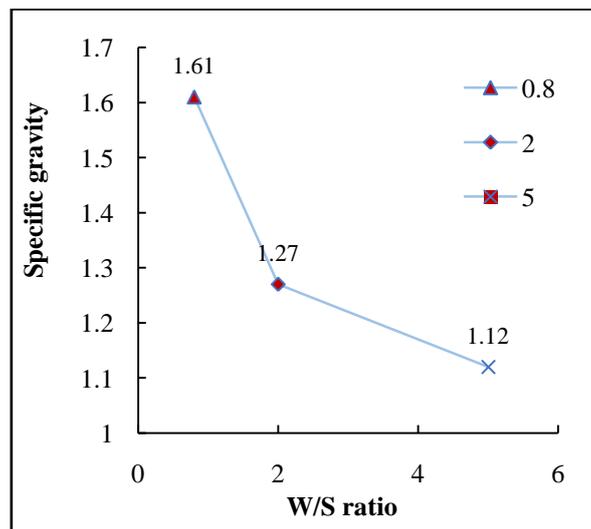


Fig. 6 Effect of W/S Ratio on Specific gravity of Slag-Based Grout

### Bleeding potential

Fig. 7 shows the sedimentary settlement of slag-based grout and

Fig. 8 illustrates that an increase in the W/S ratio leads to greater sediment settlement and increased bleeding. This is because a higher W/S ratio introduces more free water into the mixture, reducing its viscosity and solid content. This reduction in viscosity decreases particle interlocking, slows hydration reactions, and promotes the settlement of solids. Consequently, these factors collectively cause excess water to move to the surface of the grout, resulting in increased bleeding.

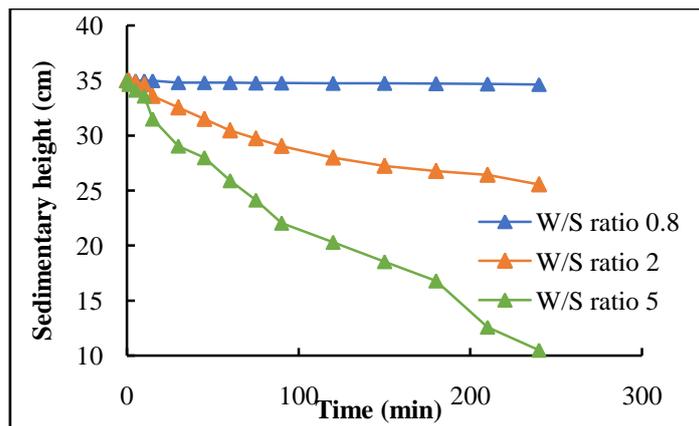


Fig. 7 Effect of W/S ratio on Sediments height of Slag-Based Grout

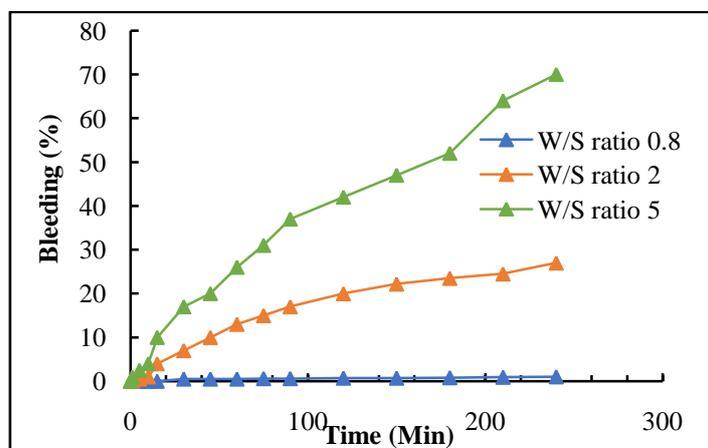


Fig. 8 Effect of W/S ratio on Bleeding potential of Slag-Based Grout

**True permeation**

Fig. 9 illustrates the true permeation results, showing that an increase in the W/S ratio enhances the grout's penetrability. This improvement is due to several factors: the reduction in viscosity and internal friction, increased fluidity and pumpability, decreased particle interference, improved workability, and the utilization of capillary action. Together, these factors enable the grout to flow more freely and penetrate finer and more complex spaces.

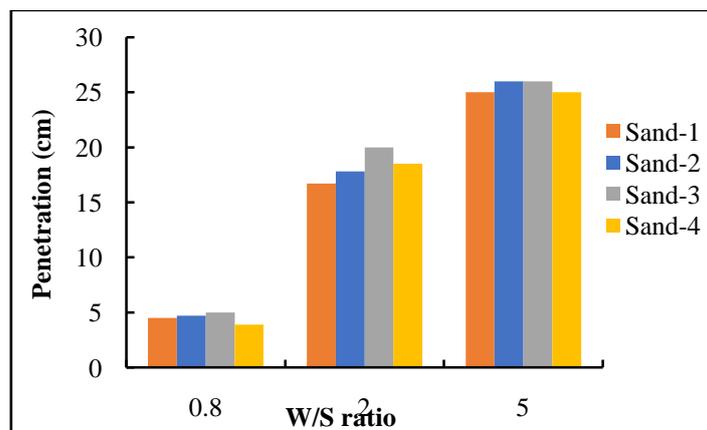


Fig. 9 Effect of W/S ratio on Penetrability in different sand of Slag-Based Grout

**Syneresis**

Fig. 10 illustrates the syneresis results, indicating that increasing the water-to-slag ratio in slag-based grout enhances the syneresis potential. This effect is attributed to several factors: the introduction of more bleeding, resulting in a weaker initial gel structure; the slowdown of hydration reactions; and the consequent greater shrinkage.

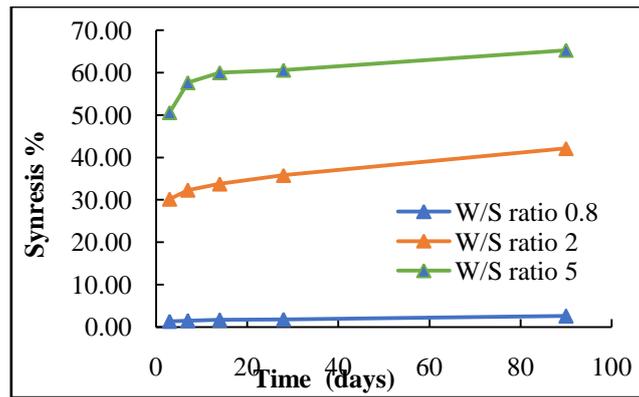


Fig. 10 Effects of W/S ratio on Syneresis of slag-based grout

### pH

Fig. 11 illustrates the pH results, indicating that an increase in the water-to-slag ratio in grout leads to higher pH levels. This elevation primarily occurs due to the enhanced dissolution and dispersion of alkaline activators and slag components, resulting in a greater release of hydroxide ions into the solution. Consequently, this increase in  $\text{OH}^-$  ions contribute to the overall rise in pH.

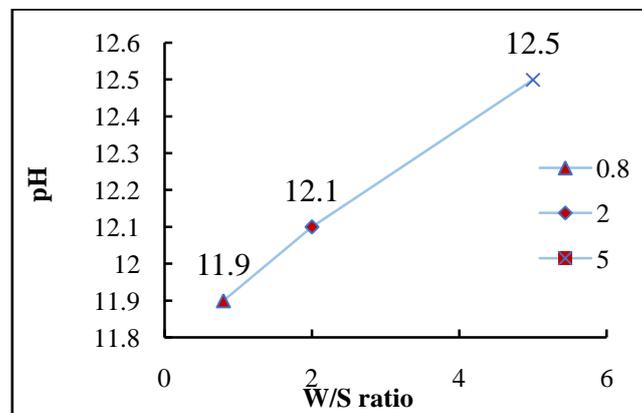


Fig. 11 Effects of W/S ratio on pH of Slag-based grout

### Rheological properties

Rheological properties are crucial for assessing the workability and flow characteristics of grout. The study encompassed tests like viscosity and fluidity to gauge the flow ability and consistency of grout samples. These tests furnished vital insights into the grout's viscosity and yield stress, facilitating a comprehensive understanding of its rheological performance

### Fluidity by marsh cone

Fig. 12 illustrates the afflux time results for different water-to-slag (W/S) ratios, demonstrating a decrease in afflux time with an increase in the W/S ratio. This reduction primarily stems from several factors: the decrease in viscosity, improvement in fluidity and workability, decrease in cohesion, and lower frictional resistance. Collectively, these factors enhance the flow characteristics of the grout, enabling it to spread and settle more rapidly.

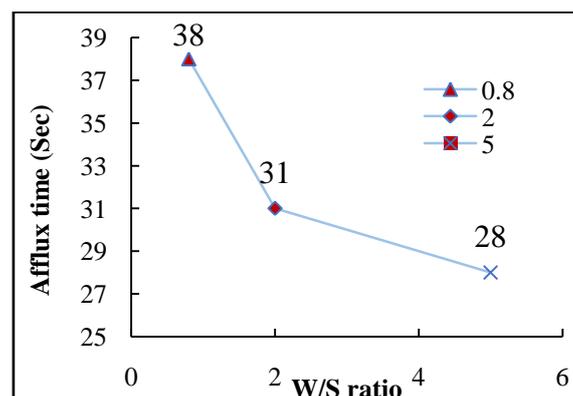


Fig. 12 Effects of W/S ratio on Afflux time of Slag-based grout

### Viscosity

Both under agitation and without agitation reading taken at grout's cake, augmenting the water-to-solids (W/S) ratio consistently apparent viscosity, plastic viscosity, and yield stress, as illustrated in The decline in apparent viscosity, plastic viscosity, and yield stress with increasing W/S ratio underscores the necessity of regulating water content in formulations to attain desired flow traits, particularly in scenarios where flow behavior profoundly influences product efficacy. This scrutiny furnishes valuable insights for industries reliant on mastering and adjusting rheological properties for enhanced product development and process streamlining.

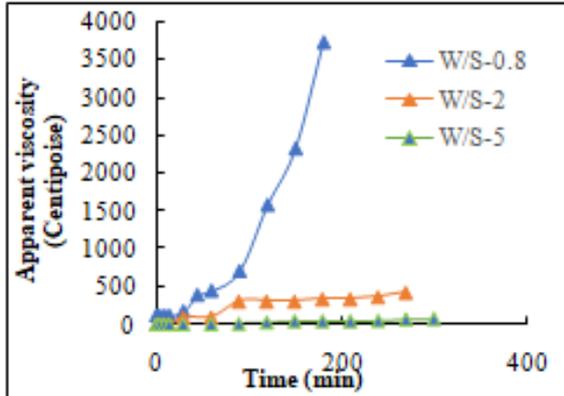


Fig. 15 Effects of W/S ratio on Apparent viscosity time characteristics of slag-based grout with agitation

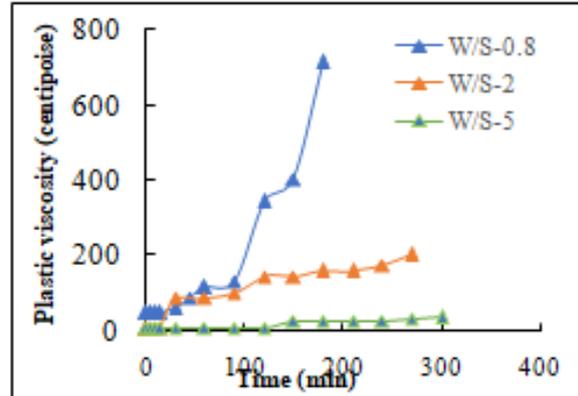


Fig. 16 Effects of W/S ratio on Plastic viscosity time characteristics of slag-based grout with agitation

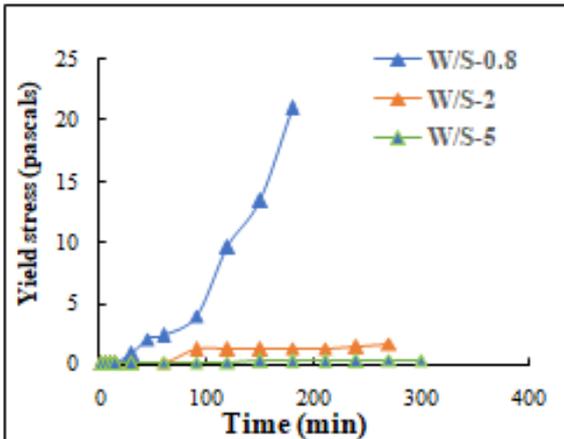


Fig. 17 Effects of W/S ratio Yield stress time characteristics of slag-based grout with agitation

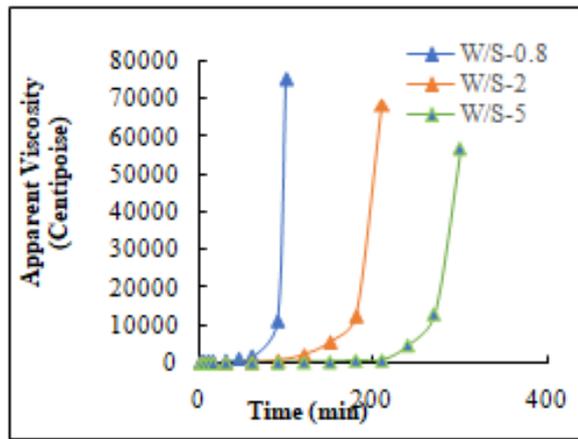
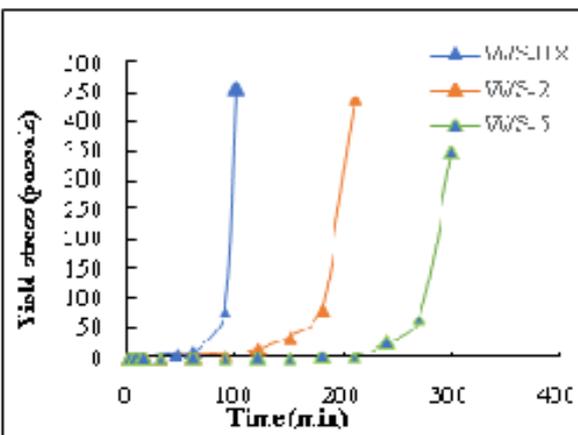
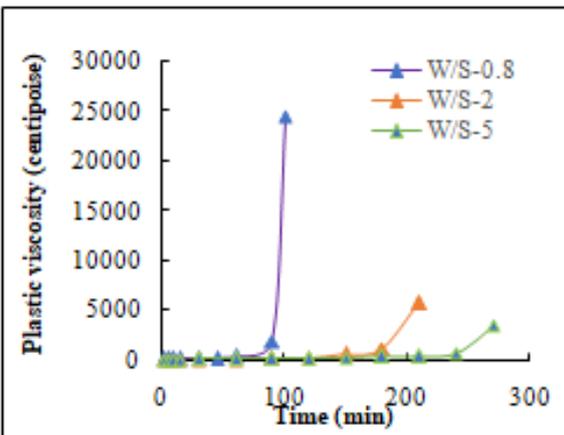


Fig. 18 Effects of W/S ratio on Apparent viscosity time characteristics of slag-based grout without agitation



### Strength properties

Assessing grout strength and durability involves conducting tests across different W/S ratios. Comparison of compressive strengths elucidates W/S ratio effects on mechanical performance, supplemented by needle penetration resistance tests for surface strength evaluation, Adherent washout test for washout strength of the grout.

### Needle penetration test

The Needle Penetration Resistance (NPR) test was employed to assess the surface strength of the samples.

Fig. 13 displays the NPR results for different water-to-slag (W/S) ratios. Notably, tests conducted at W/S ratios of 0.8 and 2 were executed 30 minutes after the gel time, whereas for W/S -5, the tests extended up to 90 minutes post-gel time. The observed decrease in NPR with an increase in the water-to-slag ratio in grout primarily arises from multiple factors: a weaker initial gel matrix, delayed and less efficient hydration, reduced concentration of hydration products, microcracking, segregation and settlement of particles, and bleeding. Collectively, these factors contribute to the formation of a less robust and weaker surface in the hardened grout.

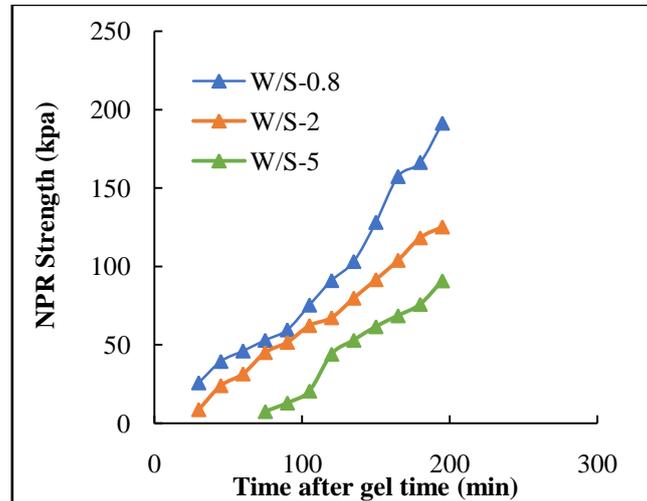


Fig. 13 Effects of W/S ratio on Needle penetration resistance(NPR) of slag-based grout

### Unconfined compression test

Unconfined Compression Strength (UCS) tests were carried out on both raw grout and grouted sand samples.

Fig. 14 illustrates the relationship between time of curing (days) and UCS (kPa). It is observed that an increase in the W/S ratio results in a decrease in strength. This decline is attributed to the increased W/S ratio leading to a reduction in material proportion and an increase in water content. Additionally, it's worth noting that the UCS of grouted sand is lower compared to raw grout. This discrepancy stems from inherent disparities in composition and structure between the two materials. Grouted sand, characterized by its porous and heterogeneous nature, inherently exhibits lower strength attributes compared to the more homogenous and cohesive raw grout.

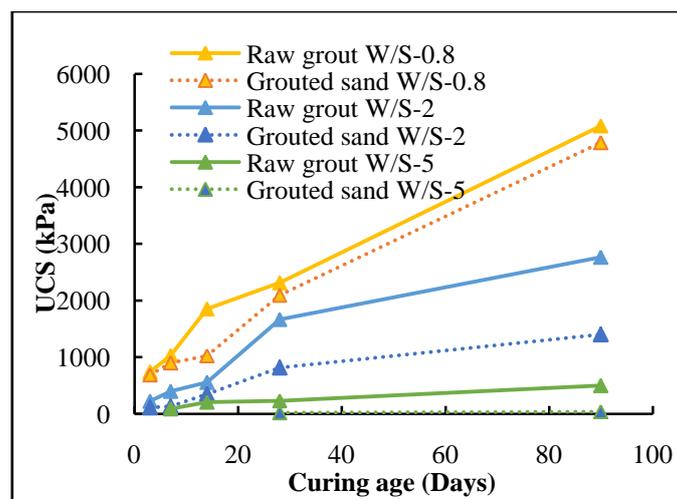


Fig. 14 Effects of W/S ratio on Unconfined Compressive Strength (UCS) of Raw Grout and Grouted Sand in Slag-Based Grout

### Adherent washout test

Washout strength tests were conducted on poorly graded sand at water-to-slag (W/S) ratios of 0.8, 2, and 5, with a permeability of 0.675 cm/s. Washout strength could not be determined at W/S ratios of 0.8 and 2 due to the air compressor's maximum capacity limitation of 8000 Kpa.

## CONCLUSION

In conclusion, this study demonstrates that the integration of magnesium oxide, calcium hydroxide, and polycarboxylate ether superplasticizer significantly enhances the performance of ultrafine slag-based grout. The optimized compositions and water-to-solid (W/S) ratios offer valuable insights for the development of advanced grouting materials with improved physical, rheological, and strength properties. A comprehensive analysis of the influence of water-to-slag (W/S) ratio on the performance of slag-based grout provides vital insights for formulating and enhancing product efficacy. As the W/S ratio increases, several significant changes occur across various aspects of grout behaviour. These changes include prolonged gel time, reduced specific gravity, increased sediment settlement and bleeding, enhanced permeability, elevated pH levels, heightened syneresis potential, and decreased afflux time. Additionally, rheological properties such as apparent viscosity, plastic viscosity, and yield stress consistently decrease with higher W/S ratios, highlighting the pivotal role of water content control in achieving desired flow characteristics. Furthermore, assessments of strength through Needle Penetration Resistance (NPR), Unconfined Compression Strength (UCS) tests, and washout strength evaluations illuminate the complex interplay between W/S ratio, material composition, and structural integrity. Notably, higher W/S ratios are associated with diminished strength attributes, primarily due to weakened gel structures, delayed hydration, increased porosity, and particle segregation.

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