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Original Article

Micro and Nano-Silica Additions to Optimize the Compressive Strength of Shotcrete

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Abstract *- The reinforcement of excavated tunnels is of utmost importance for worker safety and operational efficiency in underground mining. Shotcrete significantly helps with both, and while many additions with sustainable and pozzolanic properties have been evaluated, few studies have established the upper strength limits and guidelines for the practice. This study seeks to maximize the compressive strength of shotcrete through an optimized formula with micro and nano silica additions. Three dosage combinations across four different water-cement mixtures were tested, with results presenting a compressive strength at a .45 water-cement ratio of 64.9 MPa, a 72% increase over the control. The greatest strength result was found at a .30 water-cement ratio and measured 106.7 MPa. These results present some of the highest documented compressive strength values for their respective water-cement ratios. They contribute shotcrete formulas to help the mining industry improve their underground safety structures and maintain operational efficiency.*

Keywords *- Micro silica, Nano silica, Shotcrete, Compressive strength, Underground mining.*

1. Introduction

Improving personnel safety is a paramount concern in resource mining, as in any economic endeavor. Underground mining, in particular, presents heightened risks, given the necessity for rock mass stabilization post-excavation. Proper stabilization is essential not only for worker safety but also for the uninterrupted flow of operational activities. Rock instability is a significant threat to the efficiency of extraction and the safety of mining equipment [1]. When analyzing mining accidents, most rockfalls have been attributed to mechanical strength failures in support systems [2].

In the 1970s, Canada introduced a groundbreaking method to complement traditional scaffolding – shotcrete, a concrete material that can be projected onto various surfaces. This method has proven more effective and practical in stabilizing rock formations. Shotcrete has since emerged as one of the most widely employed rock support alternatives in underground mining. This popularity is largely attributed to its versatility, ease of application, and rapid development of high strength following application [3].

Numerous studies in scientific material development, the incorporation of new pozzolanic materials as cement substitutes, and technological advancements in application equipment have all reinforced shotcrete's position as an excellent means of enhancing worker safety while maintaining operational efficiency. Currently, there are two primary application methods: dry mix, where dry ingredients are premixed, with water added and adjusted at the nozzle during application, and wet mix, where all components are mixed before application.

Advancements in shotcrete typically follow extensive research on concrete, with many developments and assessments originating in the latter. Several metrics are used to determine the ideal shotcrete, such as tensile strength, shear strength, modulus of elasticity, bond pull-out, and permeability. Among these, compressive strength emerges as a dominant variable that correlates well with other strength measures and can accurately predict the stability of structures under the stresses imposed by the surrounding rock [4]. As a result, mining companies seek methods to enhance shotcrete strength, as it directly influences performance and safety. This enhancement is achievable by incorporating various additives into the standard shotcrete mixture. These additives can be categorized as fibrous, water reducers, pore fillers, and pozzolanic constituents, with the last two providing strength improvements without the limitations of rheological application inefficiencies [5-7].

Pozzolanic constituents are utilized to replace cement while contributing to the hydration reaction. Examples of such constituents include micro silica, fly ash, granulated blast furnace slag, metakaolin, nano silica, and various other nanoparticles, all aiming to enhance concrete's rheological, physical, and mechanical properties [8,9]. Micro silica (MS), known as silica fume, stands out as a pozzolanic additive because it reduces porosity. Vijayarethinam [10] demonstrated that replacing cement with MS at a 1:3-4 ratio does not compromise its strength properties. Moreover, MS is environmentally sustainable as a by-product of silica production, which can help reduce cement content and the high emissions associated with the concrete industry.

Nanoparticles, particularly nano silica (NS), have been studied in concrete and have shown significant potential. Sobolev and Gutiérrez [11] determined that NS accelerates the hydration of C3S due to the extremely reactive surface of NS nanoparticles. In concrete, Jo [12] found that nanoparticles were more effective at increasing strength than MS. NS serves as a catalyst to the pozzolanic reaction and fills minute spaces that MS cannot, reducing porosity and improving the microstructure, both critical aspects of final compressive strength characteristics. They concluded that NS outperforms MS but did not explore their combined effects. Wu [4] observed that NS enhances homogeneity and densifies the microstructure in ultra-high-strength concrete. Zhang [13] examined various nano additions and revealed that NS reacts with the Ca(OH)2 in concrete to form a calcium silicate hydrate gel that reduces porosity, enhances the microstructure, and, consequently, improves compressive strength, recommending a maximum dosage of 1%.

Nili [14] and Masana [15] conducted early studies in concrete, while Zhang [13] focused on shotcrete. All these studies indicated a definite synergy regarding compressive strength improvements when combining MS and NS. However, these studies included superplasticizers (SP), fly ash, and other additives, making it challenging to discern the MS and NS combination's impact on compressive strength.

This study aims to advance the current state of research by leveraging insights from existing shotcrete and concrete studies to identify effective combinations of MS and NS for enhancing compressive strength in shotcrete. The study's findings demonstrate that an optimized blend of these two additives offers substantial benefits in terms of compressive strength, surpassing previous research. This breakthrough can significantly contribute to developing shotcrete practices for improved safety and efficiency in mining operations.

2. Literature Review

2.1. Method for Designing Shotcrete Mixes in Underground Mining

In the modern mining industry, there is an ongoing quest to enhance the performance of shotcrete, a vital component in ensuring the operational continuity of mineral extraction. Various researchers have delved into the properties of shotcrete through mix design and dosing, exploring the impact of various additives like micro-silica, fly ash, granulated blast furnace slag, metakaolin, nano-silica, and others. These additives aim to improve shotcrete's rheological, physical, and mechanical properties [16-17].

Furthermore, research has centered on comprehending the behavior of shotcrete's microstructure and permeability and their influence on its physical and mechanical performance. This understanding involves the inclusion of cement replacement additives in a mix design and additionally considers the combined effects of these additives and their implications on material proportions [3;18-19].

The research reveals that designing mixes with appropriately dosed additives directly impacts mechanical performance, permeability, and pore structure. Notably, a strong correlation exists between compressive strength and the microstructure of hardened shotcrete [3][18].

In addition, the inclusion of additives in shotcrete mix design affects rheological properties like torque viscosity and flow resistance, offering the potential to enhance pumping performance, rebound rates, and the application of shotcrete mix to meet specific application area requirements [16-17].

2.2. Compressive Strength of Shotcrete in the Context of Mix Design in Underground Mining

One of the primary reasons shotcrete is widely employed in underground mining for ground support is its ability to achieve early-age strength, often accomplished through the proper dosing of additives [2]. Several researchers have directed their investigations towards studying the compressive strength of shotcrete in connection with mix design. The methodology of these studies involves incorporating different additives and calculating parameters such as compressive strength, tensile strength, and modulus of elasticity.

The goal is to analyze shotcrete's behavior and assess its mechanical performance [20-21]. Another group of studies has focused on the mix design's relation to the hydration process, the microstructure of hardened shotcrete, and its connection to mechanical strength. These investigations include a series of tests with varying dosing proportions at different ages, such as 7 days, 28 days, and 56 days [3] [18].

The results consistently show that optimal mix design with the inclusion of additives significantly boosts early-age compressive strength. Furthermore, it has been demonstrated that the increased compressive strength of shotcrete is closely linked to its microstructure, which directly influences the durability of the concrete due to a more uniform distribution of pores within the concrete matrix [20-21].

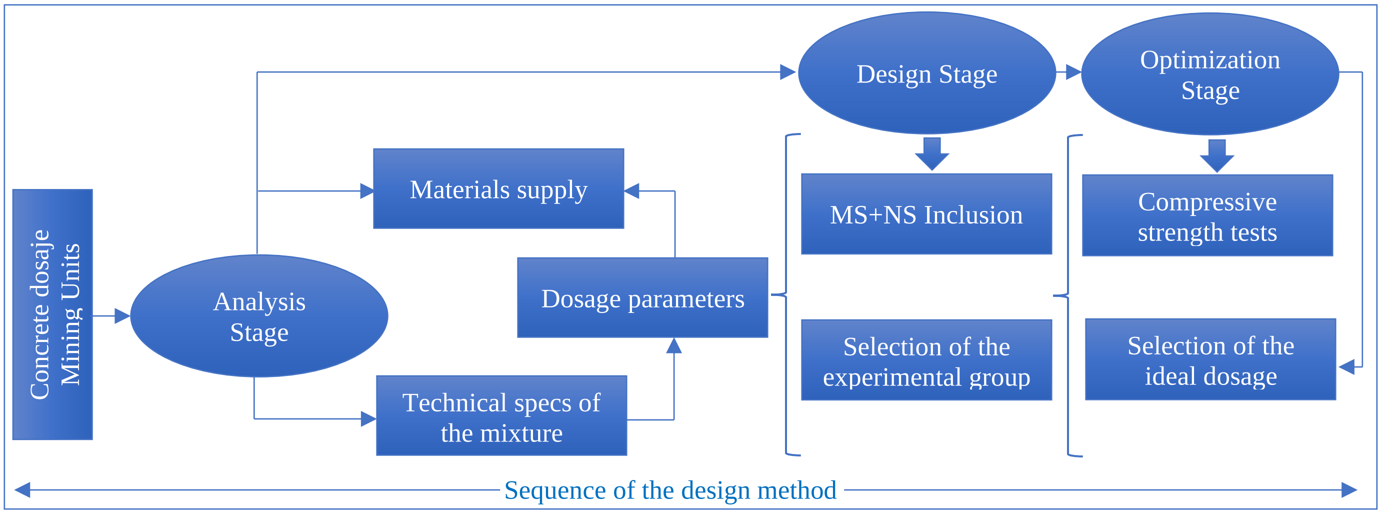

Fig. 1 Proposed method

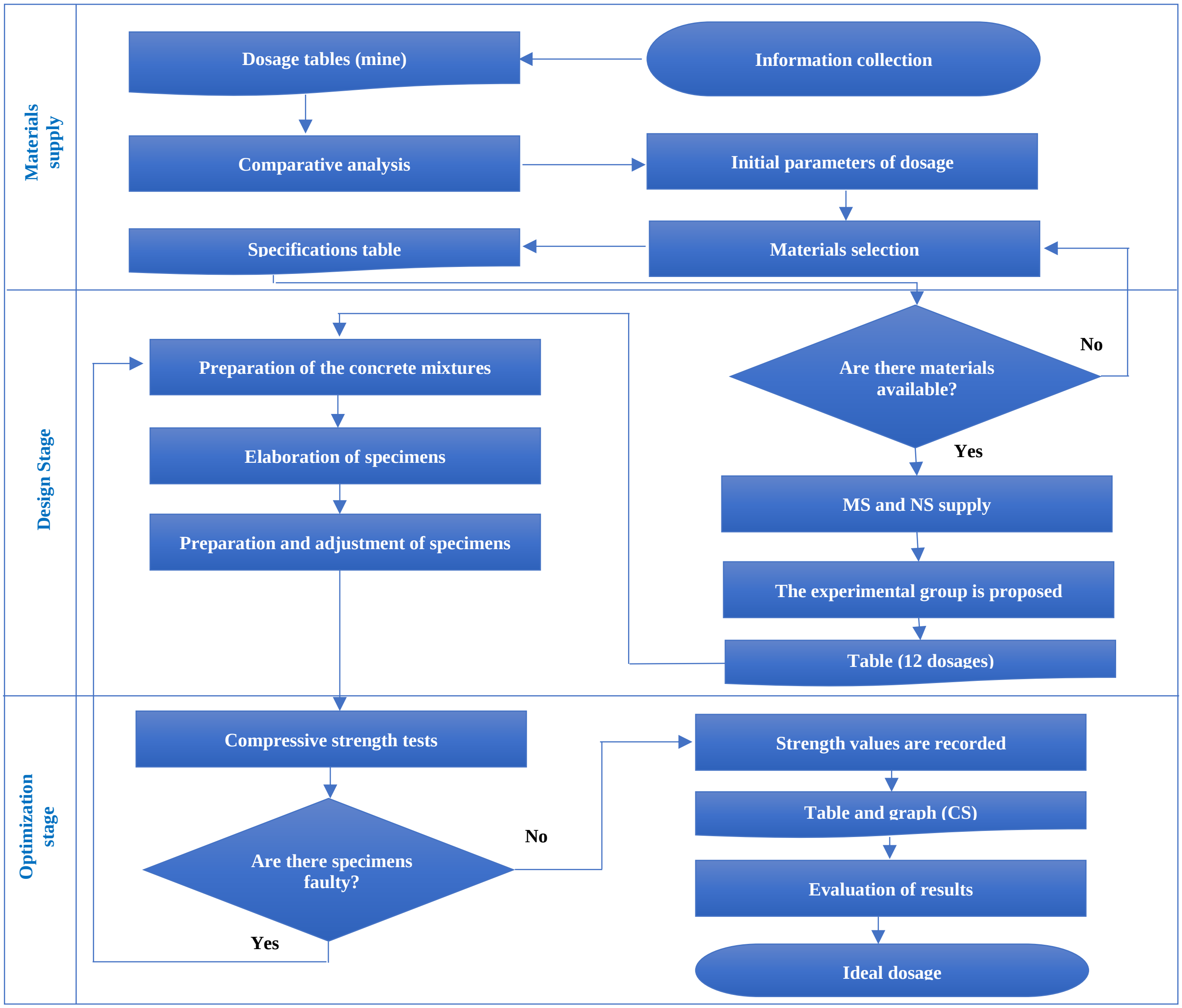

Fig. 2 Process flow of the method

Table 1. Technical specifications of solid materials

Material	Specification
Cement	Portland Type 1
Micro Silica	More than 90% purity in the form of dry powder and a density of 0.65 kg/l.
Nano Silica	More than 99% purity in colloidal solution and a density of 1.06 g/mL.
Fine Aggregate	Sand with a maximum size of 5 mm and a maximum moisture content of 0.10%.

2.3. Siliceous Additives Mix (MS and NS) in the Method of Shotcrete Mix Design in Underground Mining

Integrating siliceous additives like MS and NS into the design of shotcrete mixes for underground mining serves the growing demand for higher physical and mechanical performance in ground support. This research area has gained significant importance in recent years, and various studies have explored the combined and individual effects of MS and NS on shotcrete. Their aim is to enhance its durability and mechanical performance. The methodology of these studies relies on experimental programs that encompass a series of laboratory tests alongside the evaluation of cementing efficiency and the synergistic effects of both additives [15][19][22].

Another set of investigations has scrutinized the impact of adding MS and NS at different concentrations on the physical, mechanical, and durability characteristics of geopolymers and high-strength concretes. These comprehensive studies include compression tests, freeze-thaw resistance assessments, and microstructural characterizations [22-23]. The results of these reviewed investigations consistently demonstrate that including MS and NS significantly enhances the durability and mechanical strength of shotcrete. This enhancement is attributed to the synergistic effects of pore-filling by MS and the high pozzolanic reactivity of NS, leading to a denser packing of cementitious materials. Moreover, their combined use results in shotcrete with increased compactness, reflecting greater durability and an improved ability to resist the penetration of aggressive agents that could weaken the concrete lining matrix [15][19].

3. Methods and Materials

3.1. Overall Method

The proposed method is developed based on an experimental research model that creates a control shotcrete mixture and evaluates how different combinations of MS and NS affect the compressive strength across different water-to-cement ratios (W/C). The mixture design method encompasses three stages: Assessment stage, Design stage and Optimization stage (Figure 1), with the proposed method depicted in Figure 2. The operational shotcrete mixtures of two mining operations were assessed and compared to generate a representative experimental control. Materials selection focused on establishing the technical specifications of the main inputs for the shotcrete mixture, such as cement, fine aggregate, MS and NS (Table 1). The technical specifications of cement and fine aggregate were taken from the materials used in the mining units.

3.2. Water–Cement Ratio Indicator

The water–cement ratio indicator determines the ratio between the effective water content and the cement content in the fresh concrete mass used in each batch.

$$\mathrm{W} = \frac{\mathrm{A}}{\mathrm{C}} \tag{1}$$

Where:
A = Amount of water in Lt/m^3.
C = Amount of cement in kg/m^3.
W = Water– Cement ratio.

3.3. Dosage Indicator

The dosage indicator for MS and NS determines the percentage of replacement by weight of cement in kg/m3 for each additive in the mixture, which is represented by the following formula:

$$\mathrm{R} = \frac{\mathrm{X}}{\mathrm{C}} * 100 \tag{2}$$

Where:
X = Amount of MS or NS in kg/m^3.
C = Amount of cement in kg/m^3.
R= Percentage of cement replacement.

3.4. Selection of Dosage Experimental Groups

In order to gather sufficient data for comparison with the control and contribution to the practice, three (3) different MS-NS dosages were developed at the W/C of the control, with the same three (3) dosages tested across three (3) additional W/C ratios, for a total of twelve (12) different dosages and one (1) control.

Each of the thirteen (13) shotcrete dosages was mixed in a standard small laboratory mixer machine. First, the NS and the water are premixed in a metal container for 1 minute to obtain a better distribution of the particles. In the same way, the cement, the MS, and the fine aggregate are dry-mixed in the machine for 2 min. Then, the water and NS mixture are added to the machine and mixed for three (3) more minutes to ultimately obtain fresh concrete.

3.5. Sample Preparation

With the mixtures ready, oiled PVC column moulds are filled, and each mixture is compacted using a steel rod with a rounded tip (tamping method). The filling comprises three layers compacted with 25 strokes per layer distributed evenly throughout the cross-section of the mould. A total of 3 sample specimens were made for each batch at 150 mm in diameter and 300 mm in height each.

For design verification and quality control, the sample specimens are removed from their moulds 24 hours after being filled and placed in a curing tank at room temperature until the 28-day strength tests are conducted. Before moving on to the testing stage, the bases of the specimens are verified to ensure that they are even, and the ones without flat bases are capped so as not to affect the testing procedure.

3.6. Compressive Strength Tests

Strength tests are performed after 28 days, where each of the thirteen samples is subjected to a compression test under a load applied at a constant speed of 1.6 $kg/cm^2/s$ until the specimen fails. This process is performed using a universal testing machine following the ASTM C39/C39M testing standard. From the results obtained, the compressive strength is calculated as the average of the three tested specimens for each batch.

3.7. Calculations

The calculations used to evaluate the results of the control and experimental mixtures are specifically derived from the dosages and strength tests conducted on the concrete samples.

3.7.1. Compressive Strength Indicator

The compressive strength indicator allows us to identify the capacity of the concrete sample specimen to support a load per area unit, which is expressed in terms of effort, generally in MPa or kg/cm^2 and is calculated with the following formula:

$$\mathrm{RC} = \frac{P}{A} \tag{3}$$

Where:

P = Maximum load applied in kg.
A = Area of the cross-section in cm^2.
RC = Compressive strength of the specimen in kg/cm^2
$10\ g/cm^2 \approx 1\mathrm{Mpa}$

4. Results and Discussion

In order to establish the compressive strength effects of different combinations of MS and NS across different W/C ratios of shotcrete, a control was determined from assessing and comparing the operational shotcrete mixtures from two mining units located in the Pataz province, La Libertad department, Perú (Figure 3).

The mining units operate in the Pataz batholith, an intrusive mass located in a regional fault with NNW-SSE orientation parallel to the Maranon Valley. A corridor or structural path of mineralization, which is 160 km long with variable width that ranges between 1 and 3 km, is located on the eastern slopes of the Andes. This regional fault gave way to the formation of the vein systems found in the area. The presence of mesothermal gold vein deposits historically characterizes this intrusive mass.

While the standard shotcrete mixtures used at the two mines usually incorporate additions such as accelerators and SPs, this study chose to eliminate them to minimize variables and better isolate the effects of MS and NS. The compressive strength of this representative control at 28 days was 37.8 MPa and compares favourably to shotcrete authorities, such as the American Shotcrete Institute's Guide to Shotcrete (2016), which considers the minimum standard for compressive strength at 28.0 MPa, while Building Materials in Civil Engineering 2011 suggests that the compressive strength of shotcrete is normally between 25.0 - 40.0 MPa.

Fig. 3 Location of the province of Pataz, La Libertad, Perú

This study's control results also compare to the few studies that present standard mixtures without additions, specifically Kalhori [24], who found a compressive strength of 22.5 MPa at .5 W/C and Won et al. 2013 finding 58.4 MPa, a higher value most likely due to the .35 W/C. Considering controls with additions, Zhang [13] found a similar 56.4 MPa at W/C of .36 that included water reducers, while Smaniotto [7] achieved a maximum compressive strength after 28 days of 47.6 MPa at a W/C of just over .50 in a shotcrete mixture that included SPs and accelerators.

Cui [6] demonstrated the benefits of fibrous additions, including PET fibres to a standard shotcrete mix and finding a compressive strength between 36.0 and 47.6 MPa. For sustainable additions, such as MS, Chen [25] found a maximum strength of 31.0 MPa at a .43 W/C with a combination of coarse and fine coal gangue aggregates.

Few studies exist to aid in formulating the dosage combinations of MS and NS together. With those that do, such as Nili and Masana [14,15], both with concrete and Zhang [13] with shotcrete, they include other additions in the control. Likewise, when considering the two additions, previous research presents some debate as to which is more effective in increasing compressive strength, as Jo [12] decided that NS was preferable while Fallah and Nematzadeh [22] supported MS. As such, this study needed to follow results of previous studies of each mixture independently in order to develop dosages to test.

Likewise, existing research suggests that there is an optimized limit to the amount of MS and NS, whether separate or together. Nili [14] in concrete found a peak strength value at 6% MS and 1.5% NS with higher or lower concentrations of either yielding lower strength results. Sharaky [26] in concrete found their MS peak compressive strength at 5.0% MS. They also tested NS alone and found their peak strength at 1.5%. This coincides with Givi [27], who found a peak strength at 1.0 % NS with reductions at higher percentages and thus felt that the NS in excess of the 1.0% did not combine with the liberated lime during the hydration process and thus did not contribute to strength gains.

Many studies found that MS produced peak results at higher percentages than NS. Guided by these previous findings, this study included a 5.0%, 8.0% and 10.0% dosage of MS combined with a 1.0% and 2.0% dosage of NS, seeking to find where the limit would occur.

The first contribution of this study is to compare the three different experimental mixtures to the control to isolate the effects of the MS-NS combinations (Table 2). While it might seem counter-intuitive, at times, additions that are thought to help increase strength do not always have the theorized effect. For example, Won [5] looked to find more sustainable accelerators. While they found better results with their sustainable alternatives verses the standard, and strength was initially greater than the control between 1-7 days, this reversed at 28 days, with the greatest strength result 13% less than the control.

This study found the greatest increase in compressive strength was the mixture with 5% MS and 1% NS, although it is not excessively greater than the other mixtures (Table 2).

When consulting the Literature, few studies were found that present a wet-mix shotcrete strength at .45 W/C greater than this study's 64.9 MPa. At .5 W/C, Kalhori et al. (2020) found 39.0 MPa with 6.0 % NS, and at .42 W/C (Sharaky) et al. 2019 found 55.5 MPa with 1.5 % NS and 53.0 MPa with 5.0 % MS, both including plasticizers.

As mentioned above, definitive comparisons with existing research prove difficult due to the numerous variable combinations of W/C, additions, and additive percentages studied, and thus for general comparison, percent increase or decrease could help contribute valuable information and clarify results. This study's 71.7% strength increase at the 5% MS and 1% NS mixture compare, at the high end, with a 35-70% increased bond strength range found by Wu [4] regarding nano silica additions in ultra-high-strength concrete, where they also noted that bond strength closely correlates to compressive strength.

Table 2. Compressive strength (MPa) on the 28th day vs. control

W/C = .45	Compressive Strength (MPa)	Change (%)
Control	37.8	--
5%MS+1%NS	64.9	71.7
8%MS+2%NS	63.4	67.7
10%MS+2%NS	62.1	64.2

Table 3. Greatest compressive strength increases over control

Study	Mixture (%)	Greatest Increase (%)	SP, WR, etc.	W/C
This study	5MS, 1NS	71.7		.45
Nili *et al.* 2010	6MS, 1.5NS	26.0	X	.45
Masana *et al.* 2018	2.5MS, 2.5NS	28.1	X	.36
Zhang *et al.* 2020	5MS, .25NS	14.5	X	.36
Sharaky *et al.* 2019	1.5NS	41.3	X	.53
Fallah and Nematzadeh 2018	12MS	41.2	X	.31
Kalhori *et al.* 2020	6NS	73.3		.50

Note: In "Mixture (%)", the number represents the percentage of the ingredient that follows it.

Table 4. Proposed dosages (Kg/m3)

Mixture	*Cement*	*MS*	*NS*	*Fine Aggregate*	*Water*
MN51-0.30	459	24.1	4.6	1455	141
MN51-0.35	425	22.3	4.3	1455	165
MN51-0.40	395	20.8	4.0	1455	182
MN51-0.45	369	19.4	3.7	1455	190
MN82-0.30	478	38.2	9.6	1455	143
MN82-0.35	442	35.4	9	1455	164
MN82-0.40	412	33.0	8.4	1455	182
MN82-0.45	385	30.8	7.8	1455	191
MN102-0.30	454	46.0	9.6	1455	142
MN102-0.35	420	44	9.2	1455	161
MN102-0.40	391	41.2	8.6	1455	180
MN102-0.45	366	38.2	8.0	1455	190

Table 5. Compressive strength (MPa) on the 28th day for all dosages

Dosage per W/C	**0.30**	**0.35**	**0.40**	**0.45**
5%MS+1%NS	106.7	94.6	83.1	64.9
8%MS+2%NS	103.5	89.3	78.7	63.4
10%MS+2%NS	98.7	85.4	75.9	62.1
Mining Units	-	-	-	37.8

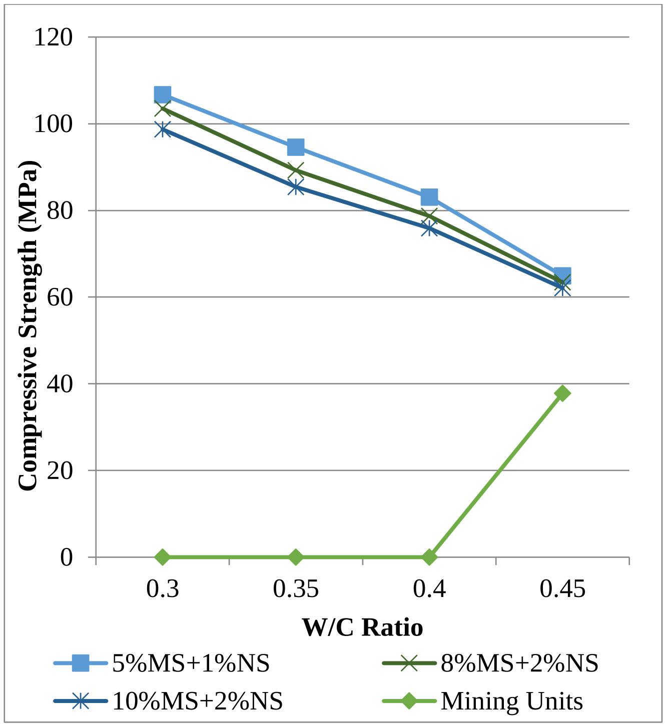

Fig. 4 Compressive Strength by dosage and W/C ratio

When comparing the percentage increase (Table 3) of the few studies mixing MS and NS together, Masana[15] in concrete found their best mixture with equal parts MS and NS, while this study, Nili [14] with concrete and Zhang [13] with shotcrete, found better results with a relatively similar relationship between MS and NS, and all with an NS dosage around the 1.0% limit found in previous studies.

We can also see that this study and the only other one that used a non-additive control, Kalhori [16], present similar and significantly greater increases in compressive strength than mixtures that included additions. Kalhori [24] with shotcrete is interesting as it ran counter to previous findings of NS losing efficacy over 1.5%, as their best percent increase over control was at 6.0% NS, and greater than its dosages of 4% and 2% at .5 W/C, achieving 39.0 MPa.

When comparing these two percent increases, this study's 71.7% and Kalhori's [24] 73.3%, with those studies that include water reducers, accelerators and superplasticizers, the results suggest that the benefit of pozzolanic materials is muted when other additions are present, as the final strength increases are less. While this may be due to the similar conditions mentioned above by Givi [27], it could suggest that it is more effective to improve strength with pozzolanic additions as opposed to other additions.

As mining operations use different W/C ratios, the study seeks to contribute by providing results for the same three MS-NS dosage levels across three more W/C ratios to bring the total number of dosages to twelve (Table 4). In the coding used for the dosages, M refers to MS, N refers to NS, the first number refers to the MS percentage, the second number refers to the NS percentage, and the number following the dash refers to the W/C ratio.

As with the control, the minimum dosage of each, 5% MS - 1% NS, proved the most effective at increasing the compressive strength across all the W/Cs tested (Table 5, Figure 4). Once again, this is in line with the findings of previous research, finding a law of diminishing returns and could be due to what Fallah and Nematzadeh [22] determined to be the high-water absorption of MS and NS that cause an incomplete hydration reaction, and in turn curb the compressive strength increase.

For absolute compressive strength achieved, we could not find results that exceed this study's for any equivalent W/C. Nili [14], in their conference study with concrete and one of the few to mix MS and NS, found a compressive strength range of around 54.0 MPa to 63.0 MPa at .45 W/C. The best result for Masana [15] was 82.2 MPa with a 2.5% MS and 2.5% NS and, including plasticizers, less than this study's 94.6 MPa at a similar W/C of .35.

Sharaky et al. (2019) found a strength of 55.5 MPa in concrete at .42 W/C at 1.5 % NS, just slightly greater than 5.0% MS at 53.0 MPa, both with plasticizers. Their greatest result was 60.0 MPa at .35 W/C with 1.5% NS. As in their MS samples, they found peak compressive strength at 1.5% NS. Suda and Rao [28] found a maximum strength of 42.9 MPa at 7.0% MS in .55 W/C concrete with ground granulated blast furnace slag as an additive. Kalhori [24] found 39.0 MPa at a .50 W/C with only NS at 6.0%.

Interestingly, for each W/C ratio, this study found the highest compressive strength occurs with the lowest levels studied of both MS and NS. For NS, this corresponds with many studies and presents the common law of diminishing returns behaviour. Wu [4] found the upper limit as well, with mixtures of both .5 % and 2.0 % nano-silica presenting similar strength values but less than at 1.0 % nano-silica.

This correlates with Shararky [26], which also saw peak values, although at 1.5 % NS. Paralleling Fallah and Nematzadeh, Wu [4, 22] similarly noted that the well-known agglomeration effects of nanoparticles, specifically with NS, would limit its catalysing effect on hydration, thus reducing compressive strength gains over the amount of 1% and, as mentioned above, Givi [27] felt that the NS in excess of 1.5% did not contribute to strength gains.

5. Conclusion

This study found that combinations of micro-silica and nano-silica can significantly increase the compressive strength of shotcrete, with results presenting some of the highest values and precent increases of shotcrete compressive strength when compared to existing studies. Like any optimum dosage parameters, the law of diminishing returns comes into play, and as previous studies have documented, the pozzolanic contributions of MS-NS show a peak dosage behaviour.

Not often mentioned is that it could be seen that when the priority is to increase the compressive strength of shotcrete, without regard to other factors, it is better to remove any admixtures and concentrate on pozzolanic materials, as the former seems to mute the effects of the later. This study contributes to the practice by presenting MS and NS shotcrete mixtures across many W/C ratios to allow mining operations to understand MS and NS effects across various operational parameters. This study was limited in focusing solely on compressive strength values and the effects of MS and NS alone. Future studies should look to analyse the microstructure, porosity, slump and additional operational factors in order to give mining operations clear guidance in ways that shotcrete with MS and NS additions can help improve the worker and operational safety of underground mining.

Data Availability Statement

All data, models, and code generated or used during the study appear in the submitted article.

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