

Enhancing the Durability Properties of Soft Clay Using Nano-Modified Cementitious Additives

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Abstract - This study focuses on studying the effect of nano-modified cementitious binders on soft clay soil, which is the most common type of soil in Winnipeg, Manitoba, Canada. The properties assessed were the unconfined compressive strength and durability of freezing-thawing and wetting-drying conditions. The soft clay was mixed with cement and slag; both were added at a dosage of 0 to 20% of the dried soil weight. Also, a nano-silica sol was added at a dosage of 0 to 2.4% of the dried soil weight (0 to 6% of the binder content). The unconfined compressive strength tests were done at 56 days of curing to allow for the latent hydraulic binder (slag) reactivity. Also, the freezing-thawing and wetting-drying characteristics started at 56 days of curing. In comparison to the mixtures containing slag and cement only, incorporating nano-silica in the cementitious system notably improved the properties of the treated mixtures, in terms of compressive strength, freezing-thawing durability factor, and wetting-drying durability factor. In particular, the ternary mix, compressing cement, slag, and nano-silica, has the potential to serve as an effective stabilizer for soft clay.

Keywords: Soft Clay; Cement; Slag; Nano-silica; Compressive strength; Durability; Freezing-Thawing; Wetting-Drying

1. Introduction

Constructing on soft clay is considered an engineering challenge due to the well-known problems associated with this type of weak soil. Low strength is one of the major shortcomings of this type which leads to low bearing capacity. Additionally, soft clay shows extreme deformations such as settlement, lateral movements, and heaving, making it an unstable construction base [1]. It is worth mentioning that soft clay's movements when loaded are not well predicted as it depends on the load value, load type, weathering conditions, groundwater table, etc. The aforementioned technical issues are exacerbated when weathering conditions are involved (e.g., freezing-thawing, wetting-drying) [2]. Consequently, various stabilization techniques, mechanical and chemical, have been adopted to improve the behaviour of problematic soil for various construction purposes. The mechanical techniques are working on physically stabilizing the soil without altering the chemical composition (e.g., piles, geosynthetics, fibers, dynamic compaction, etc.). On the other hand, chemical stabilization with additives is modifying the chemical composition to produce a denser structure. It is a widely used approach to treat soil within relatively a short time frame of construction. It has been proven that the chemical stabilization of problematic soil types such as soft clay with cement, lime, fly ash, or coal ash enhances their short- and long-term strengths [3].

Additives such as lime and gypsum have been applied successfully to stabilize the mechanical properties of weak soils, however, some limitations related to dimensional stability have been reported [4]. For example, gypsum has been used to stabilize poorly graded sand soil; the product was not able to survive weathering conditions when being tested under freezing-thawing and wetting-drying exposures [5]. Additionally, lime has been used to stabilize a high plasticity clay; insufficient resistance to wetting-drying cycles has been reported [6]. Thus, proper selection of chemical additives is important to achieve successful application with a satisfactory balance between mechanical and durability properties.

The most common chemical additive used is portland cement (calcium silicate-based material). It is considered an economic and efficient agent for providing adequate resistance/strength for clayey soils within a short time. In addition, it

is capable of providing satisfactory durability behaviour [7; 8]. Also, slag (alumino-silicate based agent) has been used to treat soft clayey soils. It is well-known for its latent hydraulic reactivity that provides strength gain at a late age [9]; thus, providing additional skeletal rigidity with time. Further enhancements can be achieved when blending cement and slag due to the cation exchange, agglomeration, hydration reactions, and pozzolanic reactions [10]. Other than the type of binder and its content, the water-to-soil ratio (w/s) has significant effects on the performance and the workability of the treated clay.

During the last decade, nanoparticles (e.g., nano-alumina, nano-silica, nano-clay, etc.) have been exploited in soil stabilization; they have promising potential at enhancing the engineering properties of weak soils (e.g., clayey soil), especially, when blended with cementitious binders. For instance, nano-silica can improve the performance of clayey and silty soils by achieving small intergranular spaces resulting in further interaction with the matrix [11]. Kalhor et al. [12] have stabilized clayey soil using nano-silica particles; successful enhancements have been reported for the unconfined compressive strength and the resistance to freezing-thawing cycles. Besides, clayey soil has been stabilized with 1% to 5% nano-silica and lime by the soil weight [13]. California Bearing Ratio (CBR) was increased by 2 to 8 times when compared to the parent clay. Similarly, using nano-silica at a dosage of 0.5% to 1% to stabilize a clayey soil accompanied by 0.3% polyester fibers was capable of significantly enhancing the CBR, frictional angle, and cohesion [14]. The improvements when using nanoparticles are ascribed to multiple mechanisms such as the filling effect, cation exchange, nucleation effect, and pozzolanic reactivity; hence, an extra-rigid skeleton among soil particles is achieved [12]. However, there has been no information on the potential of nano-modified cementitious binders on stabilizing soft clay. Hence, the motive of the current study focused on assessing the mechanical and durability properties of treated soil mixtures based on the key variables of cement, slag, nano-silica, and w/s.

2. Methodology

2.1 Materials and Mixtures

Complying with CAN/CSA-A3001 standard [15], General Use cement (C) and Grade 100 slag (S) were used as the main additives. Moreover, a commercial nano-silica sol (N) [50% SiO₂ solid content dispersed in an aqueous solution of water along with dispersing agents] was used. Table 1 lists the physical and chemical properties of the binders' components. To enhance the workability of the dry mixtures, a high-range water-reducing admixture (HRWRA) (based on polycarboxylic acid), ASTM C494-15 [16] Type F, was added. The HRWRA dosage was in the range of 50–200 ml/100 kg of the dried soil weight.

In this research, the soil was collected at a depth of 0.8 to 1.2 m below the ground surface to present the sub-grade layer. It was taken from a road under construction in Winnipeg, Manitoba, Canada. The soil classification was found to be high plasticity clay (CH) According to the unified soil classification system (USCS) following ASTM D7928-17 [17] and ASTM D2487-17 [18]. This represents the most common type of weak soil in this region that is characterized by a low shear strength, high plasticity, and high volumetric changes. Different properties of the sampled soil are summarized in Table 2.

Sixteen stabilized clay mixtures were prepared to be investigated (Table 3). The mix ID is composed of letters and numbers; letters refer to the additives used (C, S, and N), while the numbers after each letter refer to the additive's dosage. Two water-to-soil contents (w/s) were adopted (53% and 87%); the last number in the mix ID indicates the w/s. This represents a critical scenario (wet conditions) above the optimum moisture content of this soil. Besides, the control/reference mixtures were prepared from 100% of untreated clayey soil at the same w/s, representing the parent sub-grade material without any additives.

Table 1: Physical and chemical properties of GU cement, slag, and nano-silica.

Composition/Property	GU Cement	Slag	Nano-silica
<u>Chemical composition</u>			
SiO ₂ (%)	19.21	33.40	99.17
Al ₂ O ₃ (%)	5.01	13.40	0.39
Fe ₂ O ₃ (%)	2.33	0.76	0.02
CaO (%)	63.22	42.70	--
MgO (%)	3.31	5.30	0.21
SO ₃ (%)	3.01	2.40	--
Na ₂ O _{eq.} (%)	0.12	0.30	0.20
<u>Physical properties</u>			
Specific gravity	3.15	2.87	1.40
Mean particle size (μm)	13.15	11.45	35 × 10 ⁻³
Fineness, (m ² /kg)	390 ^a	492 ^a	80,000 ^b

^aBlaine fineness.

^bFineness was determined by titration with sodium hydroxide.

Table 2: Properties of the soil.

Properties	Value	Standard
Silt content (%)	27	ASTM D7928-17 [17]
Clay content (%)	70	ASTM D7928-17 [17]
Natural water content (%)	43	ASTM D2216-19 [19]
Liquid limit (%)	64	ASTM D4318-17 [20]
Plastic limit (%)	32	ASTM D4318-17 [20]
Plasticity index (%)	32	ASTM D4318-17 [20]
Liquidity index (%)	36	ASTM D4318-17 [20]
Specific gravity	2.57	ASTM D854-14 [21]
Maximum dry density (kN/m ³)	15	ASTM D698-12 [22]
Optimum moisture content (OMC) (%)	17	ASTM D698-12 [22]
California Bearing Ratio (CBR) at OMC (%)	5.5	ASTM D1883-16 [23]

Table 3: Proportions of mixtures.

Mixture ID.	Cement %	Slag %	Nano-silica %
<u>w/s = 53%</u>			
Control-53	0	0	0
C20-53	20	0	0
S20-53	0	20	0
N2.4-53	0	0	2.4
C20-N2.4-53	20	0	2.4
S20-N2.4-53	0	20	2.4
C20-S20-53	20	20	0
C20-S20-N2.4-53	20	20	2.4
<u>w/s = 87%</u>			
Control-87	0	0	0
C20-87	20	0	0
S20-87	0	20	0
N2.4-87	0	0	2.4
C20-N2.4-87	20	0	2.4
S20-N2.4-87	0	20	2.4
C20-S20-87	20	20	0
C20-S20-N2.4-87	20	20	2.4

2.2 Procedures

According to batches performed using a 60 rpm mechanical mixer, a specific sequence of mixing was followed to achieve homogenous mixtures. First, the clay was oven-dried following ASTM D2216-19 [19], then crushed using an 800-watt mechanical grinder with 1725 rpm until it passed Sieve #4 (4.75 mm). For the water content, 17% of dry soil weight which represents the optimum moisture content of the soil (OMC) was added to the dry soil while mixing for 60 s. Then, the cement and/or slag were added to the mixture while mixing for 30 s. The rest of the water was vigorously mixed for 45 s with the nano-silica sol and HRWRA and then added to the mixer and mixing continued for an extra 120 s. Afterwards, the mixtures were cast in three layers in the molds with compacting each layer using a 60 Hz mechanical frequency compactor for 10 seconds. This mechanical compactor was topped with a 98 mm diameter circular steel plate. The specimens were kept in the molds for 7 days before demolding, then placed in a standard curing room (a temperature of $22\pm 2^\circ\text{C}$ and a minimum relative humidity (RH) of 95%) before testing.

2.3. Testing

Unconfined compressive strength tests for each mix had been done at 56 days to allow for the reactivity of the latent hydraulic additive (slag). Triplicate 100×200 mm samples were prepared and tested following ASTM D1633 [24] at a loading rate of 1.3 mm/min. To investigate the durability of the stabilized soil samples, freezing-thawing and wetting-drying tests were accomplished for all mixtures after 56 days. Triplicate 100×116.4 mm cylinders for each mixture were cast for these tests. The freezing-thawing test consisted of 12 cycles; each cycle was composed of 24 h of freezing at -23°C , followed by thawing for 23 h in the moist curing room at $23\pm 2^\circ\text{C}$ according to ASTM D560-16, Method A [25]. For the wetting-drying test, it was 12 cycles as well, each cycle consisted of 5 hours of immersing into the water at room temperature ($22^\circ\text{C}\pm 2^\circ\text{C}$) followed by 42 hours of oven drying at $71\pm 3^\circ\text{C}$ according to ASTM D559-15, Method A [26]. After each cycle, debris, if any, was removed by applying two firm strokes covering all areas of specimens using a wire brush, and the masses were recorded. Also, a durability factor (%) was calculated based on Equation 1 following ASTM C666 [27] but with replacing the relative dynamic modulus of elasticity with the relative remaining mass of samples:

$$DF_{FT}/DF_{WD} = (PN/M) \times 100 \quad (1)$$

where, P is the remaining mass over the original mass, N is the number of cycles survived, and M is the total maximum number of cycles (12 cycles).

3. Results

3.1 Compressive strength

The results of the unconfined compressive strength for the untreated (control) specimens and treated specimens were listed in Table 4. Increasing the water content in the control mixtures from 53% to 87% significantly reduced the mechanical properties of all the mixtures. Also, the results generally indicated that improved mechanical properties can be achieved with the addition of cement, slag, and nano-silica at different levels for soil containing such moisture contents. The results indicated that the compressive strength noticeably increased when adding cement as the control-53 and control-87 yielded 40 kPa and 10 kPa, respectively, while C20-53 and C20-87 yielded 900 kPa and 640 kPa, respectively. The same trend was noted when adding slag but to a lesser extent; the mixtures S20-53 and S20-87 showed values of 490 kPa and 430 kPa, respectively.

Adding a small dosage of nano-silica only to soft clay did not show much improvement when compared to the aforementioned additives as N2.4-53 and N2.4-87 had compressive strengths of 190 kPa and 40 kPa, respectively. The binary mixtures had additional improvements as shown in Table 4. For instance, combining nano-silica with cement to produce mixtures C20-N2.4-53 or C20-N2.4-87 led to compressive strengths of 1200 kPa and 690 kPa, respectively. Similarly, S20-N2.4-53 and S20-N2.4-87 yielded 540 kPa and 450 kPa, respectively. The best binary mixture in terms of compressive was found to be C20-S20-53 and C20-S20-87 due to increasing the binder content to 40%, as the 56-days compressive strengths were 2390 kPa and 1990 kPa, respectively. Accordingly, the highest compressive strengths were recorded for the ternary mixes (C20-S20-N2.4-53 and C20-S20-N2.4-87 [Figure 1]); the compressive strengths of these mixtures were increased to 2540 kPa and 2100 kPa, respectively.

Table 4: Summary of the experimental results.

Mixture ID.	56 days compressive strength, kPa	Freezing-Thawing			Wetting-Drying		
		No. of cycles survived	Total mass loss (%)	DF_{FT} (%)	No. of cycles survived	Total mass loss (%)	DF_{WD} (%)
<u>w/s = 53%</u>							
Control-53	40	1	57	4	1	76.9	2
C20-53	900	7	51.8	28	5	56.4	18
S20-53	490	3	66.4	8	2	71.5	5
N2.4-53	190	1	50.6	4	1	72.6	2
C20-N2.4-53	1200	7	46.6	31	7	52.1	28
S20-N2.4-53	540	3	59.1	10	3	69.7	8
C20-S20-53	2390	12	4.7	95	12	6	94
C20-S20-N2.4-53	2540	12	2.9	97	12	5.1	95
<u>w/s = 87%</u>							
Control-87	10	0	100	0	0	100	0
C20-87	640	5	60.3	17	4	61.4	13
S20-87	430	2	68.9	5	2	77.2	4
N2.4-87	40	0	100	0	0	100	0
C20-N2.4-87	690	5	56.2	18	4	59.3	14

S20-N2.4-87	450	2	52.8	8	2	74.1	4
C20-S20-87	1990	10	21.6	65	9	18	62
C20-S20-N2.4-87	2100	12	14.8	85	12	19.8	80



Fig. 1: Exemplar mode of failure in the compression test at 28 days for: (a) the control specimen (Control-87), and (b) ternary binder specimen containing cement, slag, and nano-silica (C20-S20-N2.4-87).

3.2 Freezing-Thawing

Freezing-thawing results are listed in Table 4 in terms of the number of survived cycles, mass loss, and the durability factor (DF_{FT}). According to Equation 1, the freezing-thawing durability factor varied from 0 to 97%. The control mix (Control-53) only survived for one cycle with a durability factor of 4%, whilst at the high-water content (Control-87), the specimens had been disintegrated within the first cycle ($DF_{FT} = 0$). For the single binder system mixtures, both cement and slag showed a positive effect on the durability of the stabilized clay mixtures, but not to a satisfactory extent, indicating a lack of skeletal rigidity. Adding 20% cement to produce C20-53 or C20-87 led to a durability factor of 28% and 17%, respectively. Adding 20% slag to produce S20-53 and S20-87 led to a durability factor of 8% and 5%, respectively. The nano-silica alone did not provide appreciable resistance of soft clay to freezing-thawing conditions.

The durability was significantly improved when applying the binary system. Adding nano-silica to the cement mixtures (C20-N2.4-53 and C20-N2.4-87) relatively increased the freezing-thawing durability factor from 28% to 31% and

from 17% to 18%, respectively when compared to the single mixtures (C20-53 and C20-87). Comparatively, adding slag with cement (C20-S20-53 and C20-S20-87) showed a significant increase in the durability factor (95% and 65%, respectively). Similar to the compressive strength trends, the ternary binders C20-S20-N2.4-53 and C20-S20-N2.4-87 achieved DF_{FT} of 97% and 85%, respectively. The intact matrix of the ternary mix is shown in Figure 2 after applying 12 cycles of freezing-thawing.

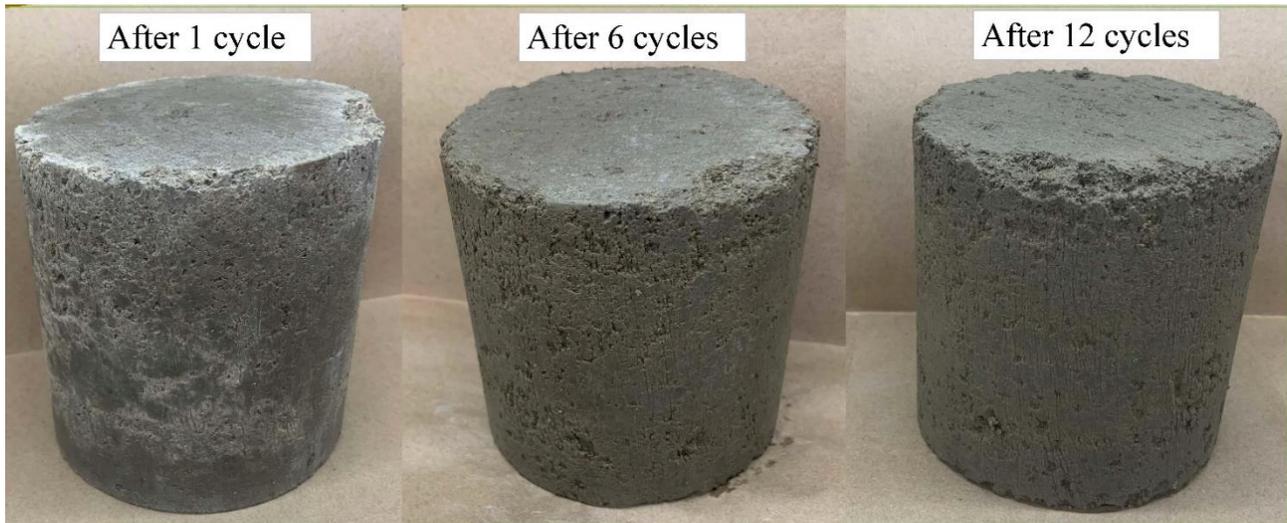


Fig. 2: Mixture C20-S20-N2.4-53 after multiple cycles of freezing-thawing (total mass loss of 2.9%).

3.3 Wetting-Drying

According to Equation 1, the wetting-drying durability factor varied from 0 to 95%. The trends of the freezing-thawing test were replicated in the wetting-drying exposure. The control mix (Control-53) only survived for one cycle with a durability factor of 2%, while Control-87 had been destroyed after the first cycle with a DF_{WD} of 0, due to the unstable nature of this soil. Cement and slag provided some improvement to the soft clay, but not to an acceptable level. For example, when adding 20% cement (C20-53 or C20-87), the DF_{WD} reached 18% and 13%, respectively. Nano-silica alone did not provide any improvement to soft clay against wetting-drying conditions. With the binary binders, the durability factor remarkably increased. For instance, adding nano-silica to the cement mixtures to produce C20-N2.4-53 and C20-N2.4-87 relatively increased the wetting-drying durability factor when compared to C20-53 and C20-87 from 18% to 28% and from 13% to 14%, respectively. At a higher binder content, mixing slag with cement to produce C20-S20-53 and C20-S20-87 showed a significant increase in the durability factor up to 94% and 62%, respectively. Again, both C20-S20-N2.4-53 and C20-S20-N2.4-87 had superior performance; these ternary binder mixtures achieved DF_{WD} of 95% and 80%, respectively. For example, Figure 3 shows that the ternary C20-S20-N2.4-53 survived the aggressive wetting-drying conditions with a mass loss of only 5.1%.

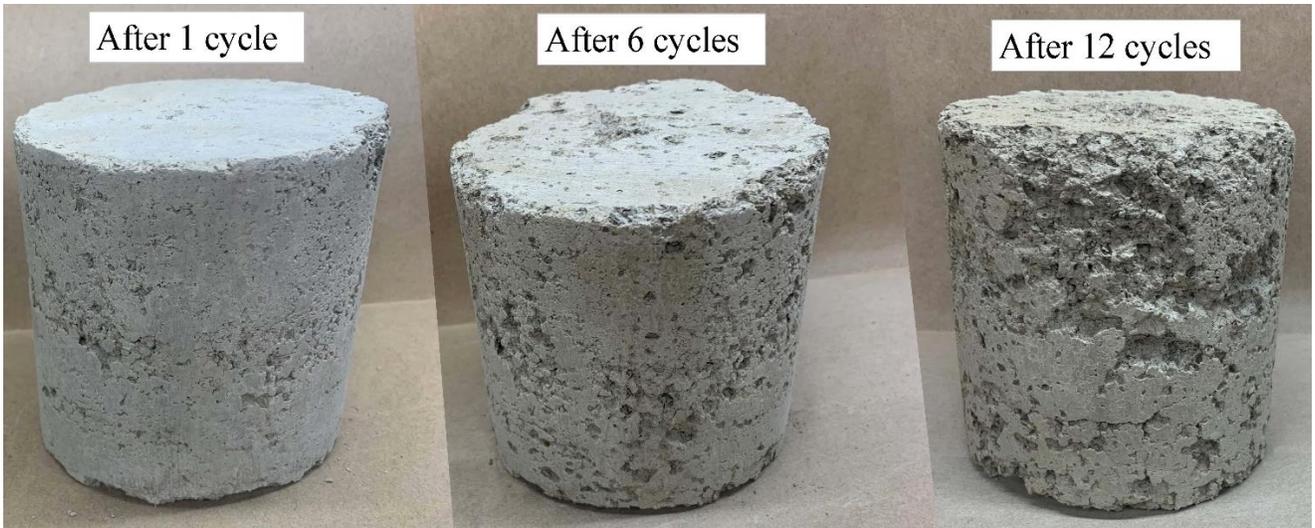


Fig. 3: Mixture C20-S20-N2.4-53 after multiple cycles of wetting-drying (total mass loss of 5.1%).

4. Discussion

Regardless of the type of binder used, the mixtures with lower water content ($w/s = 0.53$) had provided a better hydration development when compared to the mixtures with high water content ($w/s = 0.87$). Lower water-to-binder ratios reduce the volume of the capillary voids, hence densifying the hardened paste of the stabilizing binders with time [28]. This made the cementitious skeleton stabilizing the clay less vulnerable to critical moisture saturation and more resistant to the different weather exposures.

Regarding the binders' influence on treated clay, adding cement to the soft clay led to progressive production of the hydration products calcium silicate hydrate (C-S-H) with time, responsible for the strength and binding of soft clay. This is owing to the continual dissolution and reaction of the calcium silicate phases in cement (tri-calcium silicate and di-calcium silicate) with water [28]. This was shown by the performance of C20-53/C20-87 (Table 4) as the increase in cement content led to more hydration products, which stabilized flocculated fine clay particles through the formation of clay-cement bonds [29]. This explains the superior properties (DF_{FT} and DF_{WD}) of the stabilized clay mixtures compared with the untreated clay.

Similarly, adding slag to the soft clay led to progressive production of hydration products (calcium hydroxide: CH and C-S-H), but to a lesser extent due to the latent hydraulic nature of slag (less lime content relative to cement), which was the reason for evaluating the target properties at 56 days. Besides, slag has angularly shaped particles and a fine specific surface ($492 \text{ m}^2/\text{kg}$), thus acting as an interlocking filler among the soil particles hence improving the target properties when compared to the virgin clay. However, the pozzolanic reactivity (consumption of CH and additional production of secondary C-S-H) of slag is catalyzed with sufficient amounts of alkaline media (CH) [30]. Therefore, the binary binders containing 20% cement with 20% slag had high performance in terms of strength and durability due to rich binder content and hydration and pozzolanic effects of cement and slag, respectively.

The addition of nano-silica alone to the virgin clay showed minor improvement due to the filler effect as it has ultrafine nature (35 nm mean particle size at $80 \text{ m}^2/\text{g}$ surface area). The influence was found to be minor due to the absence of CH (alkaline activator) in the system, which is crucial for the chemical interaction (pozzolanic reactivity) of the nano-silica. Thereby, incorporating nano-silica with cement in particular in binary binders led to a notable increase in strength and durability factors. This can be explained as nano-silica having a nucleation effect that makes its particles act as additional surfaces for the hydration reactions, precipitating additional cement gel [31; 32]. Besides the particle packing/filling effect, nano-silica provides a vigorous pozzolanic activity that consumes the CH to produce secondary C-S-H; consequently, better development in the microstructure and skeletal rigidity can be achieved. Consequently, a more rigid skeleton with higher compressive strength and greater resistance to the freezing-thawing and wetting-drying conditions can be obtained (Table 4). This discounted the infiltration of water into stabilized clay, thus improving its resistance to weathering conditions.

The ternary nano-modified mixtures (C20-S20-N2.4-53/C20-S20-N2.4-87) showed superior performance in terms of hydration development and skeletal rigidity due to the multi-scale cementitious materials with variable reactivity. This was attributed to the high amount of dissolved Ca^{++} due to the high binder content (more than 40% by the dry mass of clay). Also, with sufficient amounts of CH produced from the hydration of 20% cement, the synergistic pozzolanic effects of nano-silica and slag had a positive impact on improving/densifying the matrix, as proven by the strength and durability factor results. It is worth mentioning that only mixtures C20-S20-53 and C20-S20-N2.4-53 have passed both the Portland Cement Association (PCA) [33] and the Federal Highway Administration (FHWA) [34] limits of the maximum allowed mass loss (6% and 7%, respectively) after the freezing-thawing and the wetting-drying tests.

5. Conclusions

Considering the materials, mixtures, testing methods implemented in this study, the following conclusions can be drawn:

- The reduction of the water content (w/s) and increasing the binder content (cement without/with slag) when treating the soft clay enhanced the compressive strength and the durability properties of the matrix (DF_{FT} and DF_{WD}) due to the efficient hydration development, the pozzolanic reactivity in case of binary cement/slag systems, and the densification of the matrix with time.
- The addition of slag and nano-silica improved the properties of the soft clay due to physical interlocking and particle packing/filler effect, respectively; however, in these single binder systems, the absence of a sufficient amount of calcium hydroxide (CH) (alkaline media) led to unsatisfactory results.

- Mixtures stabilized with blended binders containing cement (hydration development and adequate amounts of CH) with slag and/or nano-silica showed a marked enhancement in strength and durability factors due to catalyzing of the pozzolanic reactivity of slag and nano-silica.
- The ternary mixture (cement, slag, and nano-silica) was the one that achieved the highest compressive strength and durability properties. This was ascribed to the synergistic effects of those variable reactivities and multiscale constituents.
- The compendious results from this study indicate that stabilizing this weak type of soft/ expansive clay is possible in wet conditions (water contents above OMC) when applying optimized proportions of cement, slag, and nano-silica. Yet, field trials are required to substantiate the laboratory results, which are planned for future research.

Data availability statement

Some or all data, models, or codes that support the findings of this study are available from the corresponding author upon reasonable request. (Raw data for freezing-thawing tests, and wetting-drying tests).

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