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Bond Strength of Nano-Modified Concrete under Freezing Temperature using Hybrid Protection

A. M. Yasien[1*], M. T. Bassuoni[2]
[1]Post-doc Fellow, Civil Engineering, University of Manitoba, Winnipeg, Canada; Assistant Researcher, National Research Centre, Cairo, Egypt; yasiena@myumanitoba.ca
[2*]Professor, Civil Engineering, University of Manitoba, Winnipeg, Canada (Corresponding Author);
Mohamed.Bassuoni@umanitoba.ca

Abstract **-** Concrete and steel rebars bond strength depends on multiple factors such as concrete-steel interface and development in mechanical properties of concrete. However, concrete cured under cold temperatures (below 5°C) suffers from insufficient hydration development of cementitious paste; consequently, the mechanical properties of concrete are negatively affected. In this paper, concrete-steel bonding behaviour in concrete cast and cured under freezing temperature was explored. Three concrete mixtures were cast and cured at -20°C. The mixtures were protected using hybrid system consisting of insulation blankets and phase change materials mat. The mixtures comprised General Use cement, fly ash (20%), and nano-silica (6%) as well as calcium nitrate-nitrite as a cold weather admixture system. The mixtures were assessed in terms of internal temperature evolution, compressive and tensile strengths, as well as modulus of elasticity. In addition, the different mixtures' bond strength with steel re-bars was evaluated through a pull-out test. Thermogravimetric analysis was conducted to assess the hydration development of the adopted mixtures. Furthermore, to visualize the quality of the interface between steel-rebars and the different cementitious matrices, environmental scanning electron microscopy associated with energy dispersive x-ray analysis was performed on fracture pieces extracted from the interface. Nano-modified concrete protected using the adopted hybrid protection system produced good quality concrete-steel interface with adequate bond strength, without need for heating operations prior to casting and during curing under the adopted freezing temperature (-20°C); thus, it may present a viable option for cold weather structural applications.

Keywords**:** Nano-Silica; Cold Weather; Phase Change Materials; Freezing; Bonding; Cold Weather Admixture System.

1. Introduction

The reinforced concrete elements capacity is affected by the concrete-steel bonding, which defines their ductility and structural performance [1]. However, casting and curing concrete under cold weather negatively affect its hydration and mechanical properties development; consequently, the concrete-steel bond strength [2,3]. Guidelines and codes for concrete (e.g., ACI 306R-16 [4], CSA A23.1-19 [5]) set 4 to 5°C as the benchmark for cold weather precautions. When the ambient temperature drops to these values, hydration development of cementitious binders proceeds at slow rates and stops when the temperature reaches -2.8°C due to freezing of mixing water [6]. Concrete cured under these conditions suffers from cracked matrix and heterogonous microstructure, which negatively influences its structural performance and service life [6-7].

The aforementioned codes/guides [4,5] have stringent stipulations for cold weather applications, including limitations on mixing, placement and curing temperatures, as well as building voluminous and heated enclosures to maintain concrete temperature at an acceptable threshold (more than 10°C) during placement and protection periods. Furthermore, ACI 306R-16 [4] recommends heating any metallic embedments to at least 0°C when the ambient temperature reaches -12°C to avoid ice formation at the interface and ensure adequate development in bond strength with concrete. However, these heating practices incur considerable expenditures due to excessive energy consumption, required skilled labour and increased carbon footprint of construction [7]. Consequently, in cold regions, the construction season is limited to warmer periods (from May to October in North America), resulting in significant socioeconomic losses due to backlogged construction schedules. Therefore, extending the construction season in cold regions is essential, which requires continual development of innovative strategies to cast and protect concrete under freezing temperatures.

In cold weather applications, the bond between concrete and steel maybe inferior due to the development of inadequately hydrated interfacial zone [8]. For instance, the effect of using sodium nitrite and calcium nitrite (cold weather admixture system; CWAS) at dosages of 6% and 2% by mass of cement, respectively on bond strength with 20 mm steel bars was investigated [8]. After casting specimens, using hot water (54℃) and room temperature steel bars, they were cured at temperatures of -5 and -10℃. After 15 days of curing, the bond strength of the specimens was less than 1 MPa and increased to 5.5 MPa after 28 days, which was markedly lower than that of similar concrete cured at normal temperatures (8 to 17 MPa) [9,10].

Since the last decade, the incorporation of vigorously reactive nanoparticles (e.g. nano-silica) in concrete to improve the hydration and microstructural development of concrete as well as its hardened properties have been the subject of extensive investigations [11,12]. However, these studies were conducted for concrete cast and cured under normal temperatures (20 to 30℃), whilst research on nano-modified concrete in cold weather applications is still in early stages. For example, in recent studies by the authors [13-15], it was found that low *w/b* (0.32) concrete mixtures, cast and cured under cold temperatures down to -20 ℃, incorporating 2 to 6% nano-silica and calcium nitrate-nitrite solutions (CNAI), without and with fly ash, achieved acceptable performance in terms of hardening rates, hydration development, strength and durability properties. However, the bonding behaviour of these mixtures with unheated metallic reinforcement at freezing temperatures has not been investigated, which warrants further studies in this direction.

Concrete incorporating phase change materials (PCM), energy absorbing/release reservoirs that can reversibly store and emit heat through solidifying/melting processes [16], is a relatively new technique capable of enhancing concrete's thermal energy storage capacity. PCM can be added to the concrete by direct incorporation during mixing [17-18], micro [16], or macro-encapsulation [18] as well as saturation of lightweight aggregates [18], or nano-materials such as nano-silica [17]. Organic PCM, paraffin or non-paraffin, are preferred to be used in concrete applications due to their noncorrosive and stable nature as well as operating temperatures [18]. These materials have been commonly used to improve concrete resistance to freezing-thawing cycles by delaying ice formation [19], and mitigating thermal cracking [19]. However, the incorporation of PCM in concrete resulted in negative effects on concrete mechanical and physical properties (e.g. reduction of strength/elastic modulus [19,20], and high porosity [20], and drying shrinkage [19]), which may limit the applications of PCM-modified concrete to non-structural applications [21]. Despite the potential of PCM to retain concrete internal temperature and delay ice formation between concrete and unheated metallic embedment, the effect of using PCM as external curing on concrete bond strength has not been explored.

2. Experimental Procedure

2.1. Materials and Mixtures

General use (GU) cement and Class F fly ash, complying with CAN/CSA-A3001-18 [22], were used as the main binder component. A commercially available nano-silica solution (fully dispersed SiO_2 in aqueous solution with 50% solid content) was added to the mixtures as a partial replacement of cement. The mean particle size, specific gravity, and surface area of the nano-silica are 35 nm, 1.4 and 80,000 m2/kg, respectively. The dosages of fly ash and nano-silica were fixed at 20% and 6%, respectively. For all mixtures, the total binder content and water-to-binder ratio (w/b) were kept constant at 400 kg/m^3 and 0.32, respectively. These proportions produced concrete with balanced performance in terms of fresh, hardened and durability when cast and cured under mild and low freezing temperatures [13-15]. Well-graded river sand with fineness modulus of 2.9 and natural gravel with maximum size of 9.5 mm were used in this study. High-range water reducing admixture (polycarboxylic acid-based), complying with ASTM C494-19 and air entraining (AE) admixture, complying with ASTM C260-16, were added to achieve a target consistency and fresh air content of 175±25 mm and 6±1%, respectively.

A combination (CNAI) of calcium nitrite and calcium nitrate with a ratio of 1:1 by mass was used as CWAS, at a constant dosage of 15% by mass of the mixing water, which was selected based on the phase diagram of CNAI [13] to depress the freezing point of mixing water down to -5℃ to avoid freezing of mixing water during the first 2 to 4 hours, to allow for mixing, transporting and placement operations before applying any protection (refer to the Procedures Section). The proportions of the mixtures, which were cast and cured at -20°C, adopted in this study are shown in **Table 1**. In the mixture ID, the letters GU, F, N denote GU cement, fly ash and nano-silica, respectively.

Table 1: Mixtures proportions per cubic meter

Mixture ID.	Cement (kg)	Fly ash (kg)	Nano-silica (kg)	Water* (kg)	CNA†/CNI* (kg)	Coarse aggregate (kg)	Fine aggregate (kg)
GU	400	-	-	105.6	14+32	1156	622
GUN	376	-	48	81.6	14+32	1151	620
GUFN	296	80	48	81.6	14+32	1137	612

*Adjusted amount of water considering the water content of nano-silica (aqueous solution with 50% solid content of SiO_2) and CNI (aqueous solution with 30% solid content).

†The CNA admixture was in solid form with 70% active ingredient.

2.2. Procedures

Based on previous investigations by the authors [13-15], preparations and mixing procedures were conducted at -5ºC inside an environmental chamber, where solid constituents of concrete were kept for 24 h prior to mixing. Cold mixing water (5±1ºC) was used to mimic winter tap water condition. These temperatures simulate minimal heat conditioning for mixing concrete ingredients for cold weather applications. After mixing, concrete specimens were cast in molds, with initial temperature similar to the curing temperatures, and kept inside an environmental chamber at -20ºC till testing. The specimens were covered using hybrid protection technique (a reusable PCM mat attached to an insulation blanket of R-value of 3.5 with a reflective layer). The PCM melting point was selected to be approximately 5ºC (**Table 2**) to release stored energy (187 J/g) at the cold weather limit defined by ACI 306R-16 [4] and CSA A23.1-19 [5]. The PCM mat was replaced with another melted one after three and seven days of curing to boost the hydration of the different mixtures. Also, to simulate wind conditions, the chambers were equipped with a fan to circulate air at an average speed of 25 km/h. Properties of the adopted PCM is shown in **Table 2**.

Table 2: Properties of phase change material

Parameter	Value*
Melting point	5°C
Heat storage capacity	187 J/g
Thermal conductivity (liquid/Solid)	0.15/0.25 W/m°C
Density (liquid/Solid)	0.88/0.96 g/ml
Specific heat (liquid/Solid)	2.26/1.78 J/g°C
Number of cycles before instability	10,000 cycles
Expected service life with daily usage	27 years

*Data was provided by the manufacturer.

2.3. Testing

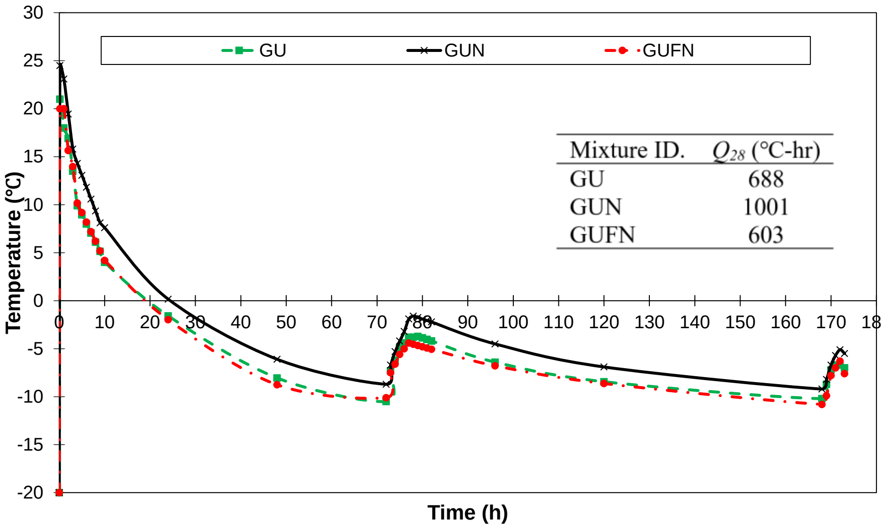
Concrete mechanical properties were investigated based on compressive and tensile strengths as well as modulus of elasticity, which were determined by testing 100×200 mm triplicate cylinders as per ASTM C3-20, ASTM C496-17 and ASTM C469-14, respectively after 28 days of curing. In addition, the bond strength of the different mixtures, with steel reinforcement at 28 days, were evaluated through pull-out tests, which were performed on triplicate 200 mm cubic specimens following RILEM 7-II-128 -94 [23] guidelines. Each specimen contained a 15M (16 mm) steel re-bar, which had an initial temperature of -20°C, with an embedment length of 200 mm). To limit the failure load below the machine capacity, half of bars embedded length (100 mm) was de-bonded using polyvinyl chloride tubing. Two linear variable displacement transducers (LVDTs) were attached to each specimen to monitor the steel bars free and loaded ends slippage under a constant loading rate of 0.5 kN/s. The machine load and LVDTs readings were collected using a data acquisition system and used to draw the force-slippage relationships of the different mixtures.

In addition, the different mixtures internal temperature was monitored using Type T thermocouples up to 28 days of curing. The thermo couples were embedded inside the bond test specimens in the steel-concrete interface. Furthermore, to assess the hydration development of mixtures, thermogravimetry (TG) at 10°C/min constant heating rate was performed on powder samples, extracted from the different mixtures. The portlandite (CH) content was calculated by multiplying the percentage drop of an ignited mass of the TG curves at the range of 400 to 450°C by 4.11 (CH-to-water molecular mass) after 1, 3, 7, 14, 28, 56 and 91 days of curing. In addition, environmental scanning electron microscopy (ESEM) associated with elemental dispersive X-ray (EDX) analysis was performed on fracture pieces extracted from the interface between the different mixtures and steel bars following the bond test to capture the effect of changing the mix design parameters on the quality of the concrete-steel interfacial transitional zone (ITZ).

3. Results and Discussion

3.1. Internal Heat Evolution

Figure 1 presents the internal temperature evolution at the concrete-steel interface in specimens during the first 7 days of curing under -20°C. In addition, the net heat produced (Q_{28}), considering concrete hydration heat generation and losses to adiabatic environment, at this interface in the different mixtures during the first 28 days of curing are tabulated in Fig.1. Q_{28} (°C-h) was integrated with respect to time, representing the area under the temperature-time curve, considering a datum temperature of -10°C, at which the hydration development was negligible. All steel re-bars had an initial temperature of -20°C similar to the curing regime. For the different mixtures, the concrete-steel interface temperature increased to reach 20 to 24.5°C after 15 min of casting concrete due to rapid heat exchange through thermal conduction between the two materials. Afterwards, the interface temperature experienced continual drop in all mixtures, which can be attributed to the heat loss to the surrounding environment (-20°C). Hence, the interface temperature, reached the freezing point of water (0°C) after 19 to 24 h and dropped to a range of -9.2 to -10.8°C, after 7 days of curing at -20°C. The different mixtures reached an average minimum temperature of -17.6 to -18.7°C after 28 days of curing, indicating near-equilibrium with adiabatic temperatures irrespective of mix design. It is worth noting that, the replacement of PCM mat after 3 and 7 days of curing led to a rise (3 to 7°C) at the concrete-steel interface temperature in the different mixtures after 4 to 6 h of replacement. This can be linked to the PCM energy release phenomenon, which re-boosted the reactivity of different binders, leading to higher internal temperature and Q_{28} as shown in **Fig. 1**.



Mixture ID.	Q_{28} (°C-hr)
GU	688
GUN	1001
GUFN	603

Fig. 1: Internal temperature profiles at concrete-steel interface in mixtures after 7 days of curing at -20°C.

Binary mixture comprising nano-silica attained the highest temperatures at the concrete-steel interface compared to other mixtures. For instance, the temperature of mixture GUN was 2 to 5°C higher than that of the corresponding reference mixture GU during the first 3 days of curing with 45% higher Q_{28} (**Fig. 1**). This can be ascribed to nano-silica nucleation effect on enhancing the kinetics of binder reactivity, as will be discussed in the TG section. In contrast, ternary mixture incorporating fly ash with GU and nano-silica showed higher temperature loss at the concrete-steel interface compared with the corresponding nano-modified binary mixture. For example, incorporating 20% fly ash in mixture GUN to produce mixture GUFN reduced Q_{28} by 40%. However, the coexistence of nano-silica in the ternary mixture partially hindered the negative impacts of fly ash on delaying the internal heat evolution of concrete, specially at the adopted curing temperature, hence the ternary binder maintained internal temperatures close to that in the single binder at the concrete-steel interface. For example, mixture GUFN had a reduction of 12% in Q_{28} compared to that of corresponding mixture GU.

3.2. Mechanical Properties and Bond Strength

The force-slip relationships of the adopted concrete mixtures, with steel are presented in **Fig. 2**. In addition, the mechanical properties; compressive strength (f_c'), tensile strength (f_t), modulus of elasticity (E), and the bond strength (τ_b), of these mixtures after 28 days are tabulated in **Table 3**. During the bond test, concrete splitting was the dominant mode of failure; consequently, the bond strength of all mixtures was governed by the mechanical properties of concrete [24]. The bonding failure load of the specimens varied between 48 to 60.9 kN corresponding to 9.6 to 12.1 MPa concrete-steel bond strength, which conforms to the bond strength values for concrete, which was cast and cured under normal temperatures, with similar strength grade and re-bar size [9,10]. It is worth noting that concrete bond strength is mainly governed by the failure mode which is a function of concrete mechanical interaction, confinement, chemical adhesion and friction with steel re-bars [24]. The splitting mode of failure of concrete dominates when the re-bar embedded length is high enough due to surrounding concrete failure by cracking [24], which is linked to the degree of hydration of the cementitious matrix. This is similar to the current case to comprehend the evolution of mechanical properties of the adopted mixtures, cured under -20°C, with respect to bonding with steel.

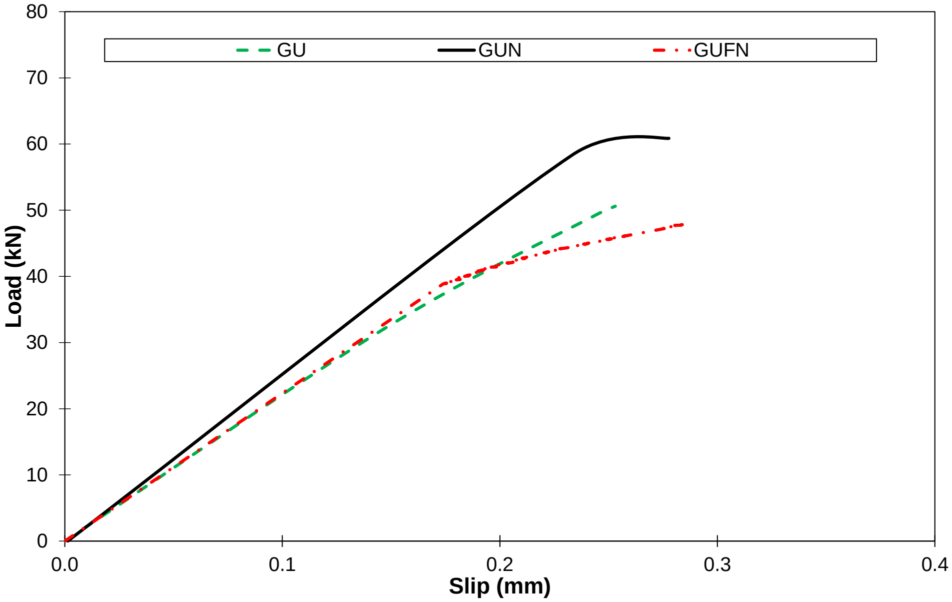


Fig. 2: Load-slip relationship (at the free end) of mixtures after 28 days of curing at -20°C.

Changing the mix design parameters had a marked effect on the mechanical properties and in turn bond strength of concrete mixtures cured under -20°C. Hence, binary nano-modified concrete attained the highest mechanical properties. For example, adding 6% nano-silica to mixture GU to produce mixture GUN increased the bond, compressive, and tensile strengths as well as modulus of elasticity by 20, 30, 43, and 31% respectively. This complies with the higher internal heat of nano-modified mixture, indicating better development in paste microstructure and ITZ with steel bars as will be discussed in the subsequent section. Comparatively, Class F fly ash has a well-documented delaying effect on the development of concrete mechanical properties at 28 days under normal and low temperatures down to -5℃ [2,13,15] due to its slow reactivity. Albeit this effect would be expected to be amplified under the freezing temperature (-20℃) adopted herein, the coexistence of nano-silica in mixtures comprising fly ash alleviated this negative influence on concrete. Hence, ternary mixture (GUFN) comprising fly ash and nano-silica yielded mechanical properties close to the corresponding single binder mixture (GU). For instance, the incorporation of 6% nano-silica and 20% fly ash in mixture GU to produce mixture GUFN led to marginal reduction in bond and compressive strengths by 5% and 3%, respectively with 3% increase in tensile strength and similar modulus of elasticity after 28 days of curing.

Table 3: Mechanical properties of concrete and bond strength with steel at 28 days

Mixture ID.	Compressive Strength, f_c' (MPa)	Tensile Strength, f_t (MPa)	Modulus of Elasticity, E (GPa)	Bond Strength, τ_b (MPa)
GU	34.6 (1.8)	3.5 (0.2)	32 (0.9)	10.1 (0.2)
GUN	45.0 (2.2)	5.0 (0.1)	42 (2.0)	12.1 (0.4)
GUFN	33.6 (1.3)	3.6 (0.1)	32 (1.0)	9.6 (0.3)

Note: numbers in parentheses represent standard deviations.

3.3. Hydration Development and Microstructure Characteristics

Figure 3 shows portlandite (calcium hydroxide, CH) trends in the different mixtures up to 91 days of curing. The different mixtures showed continual hydration, despite the gradual drop in internal temperature that reached 0℃ after 19 to 24 h of curing, as shown in the previous sections. The CH contents support the superior performance of nano-modified mixtures that produced higher CH contents at early-age due to its vigorous reactivity associated with its ultra-fineness. This can be linked to the nucleation effect of nano-silica on the cement hydration kinetics at early-age by providing the hydration products with additional surfaces to precipitate on [12], even under low temperatures. Indeed, the coexistence of CWAS was essential to keep the functionality of nano-silica especially under the freezing temperature adopted herein. This conforms to the higher internal heat of binary binder mixture compared to that of single and ternary binder mixture. Moreover, the mixtures comprising nano-silica showed consumption of CH after 14 days of curing at -20℃, indicating the initiation of delayed pozzolanic reactivity by reacting with CH to produce secondary C-S-H gel. This pozzolanic reactivity was accompanied by the filler effect in the matrix [11-15], which substantiate the notable improvement in mechanical properties, including bond strength with steel re-bars, of these mixtures at 28 days.

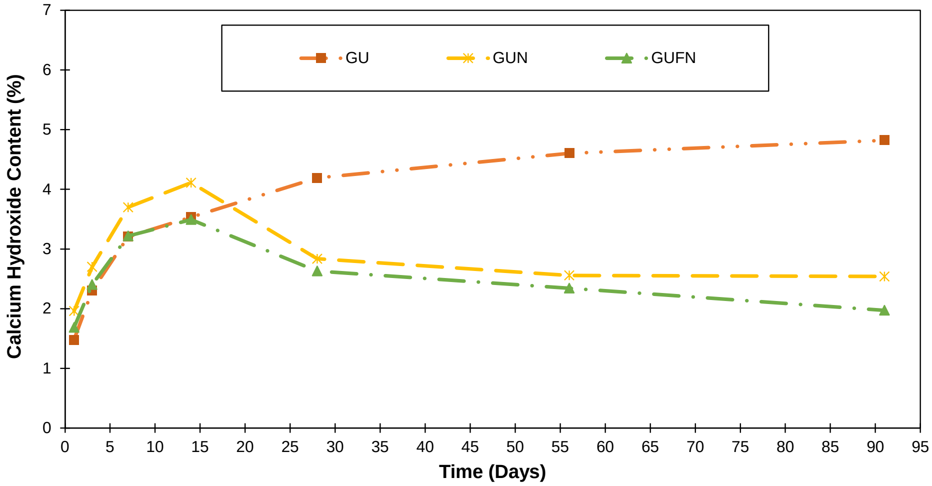


Fig. 3: thermogravimetry results for the portlandite contents in mixtures up to 91 days of curing.

Likewise, as shown in **Fig. 3**, ternary mixture showed continual hydration development, even with the presence of 20% Class F fly ash, due to the synergistic effects of nano-silica and CWAS which catalysed the slow reactivity of fly ash. Hence, nano-modified fly ash concrete, cast and cured under -20°C showed acceptable performance in terms of mechanical properties and hydration development. This highlights that ACI 306R-16 [4] and CSA A23.1-19 [5] recommendations against the use of slow reactivity supplementary cementitious materials under cold weather concreting can be mitigated by the addition of nano-silica.

To corroborate the TG findings, ESEM analysis was performed on fracture pieces extracted from the concrete-steel interface of the different mixtures after the bond test (**Fig. 4**). In addition, the average calcium-to-silicate ratio (*C/S*) within the concrete-steel ITZ was determined by EDX, which was performed at multiple points. It was reported that the *C/S* of conventional and secondary/pozzolanic C-S-H were approximately 1.7 and 1.1, respectively [25]. The microstructural features of the different mixtures were dissimilar, which indicated the dominant effect of changing the mix design on concrete cast and cured at the freezing temperature adopted herein. It is worth noting that the micro-cracks appearing in the images are associated with mechanical loading during the pull-out test which led to concrete failure.

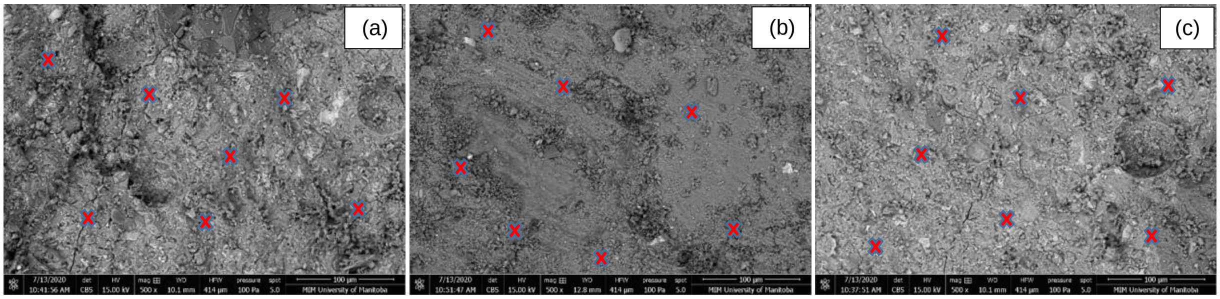


Fig. 4: ESEM micrographs for mixtures (a) GU (avg. *C/S* = 1.6), (b) GUN (avg. *C/S* = 1.1), (c) GUFN (avg. *C/S* = 1.4). Note: The *C/S* was calculated based on the marked locations.

The inclusion of nano-silica in concrete cast and cured under -20°C produced more homogenous microstructure compared to single and ternary mixture. For example, the incorporation of 6% nano-silica in mixture GU to produce mixture GUN reduced the *C/S* from 1.6 to 1.1 as shown in **Fig. 4 (a) and (b)**, which highlights the pronounced effect of nano-silica pozzolanic reactivity, even under freezing temperatures, on densifying concrete-steel ITZ by producing secondary C-S-H gel, which improved the bond strength of the binary binder mixture. Accordingly, as shown in **Fig 4. (c)**, the negative effects of incorporating 20% slowly reactive material such as Class F fly ash was alleviated by the co-existence of nano-silcia. Hence, nano-modified fly ash mixture showed better microstructure characteristics (densified concrete-steel ITZ) and lower (*C/S*) compared to single binder mixture. This supports the previously discussed synergistic effect of nano-silica and fly ash which produced concrete mixtures with comparable/ better performance than the corresponding single binder mixture.

4. Conclusions

Considering the mixtures' design, mixing/curing temperature, protection and test methods implemented in this study, the following conclusions can be drawn:

- Without heating, the hybrid protection system (insulation blankets + PCM mat) produced concrete mixtures with similar mechanical properties and bond strength with steel compared to conventional concrete cured under normal temperatures. The PCM acted as a renewably external curing aid through its heat release phenomenon, which improved the hydration and microstructure development of concrete cast and cured under -20°C.

- The synoptic experimental results showed that nano-modified concrete comprising CWAS, without or with fly ash (20%) and protected using the hybrid system, achieved adequate hydration development, mechanical properties and bonding with steel re-bars due to the nucleation, pozzolanic and filler effects of nano-silica.
- The integrated strategy of nano-modified concrete and hybrid protection system presents a viable option to produce reinforced concrete elements suitable for cold weather applications down to -20°C without the need for heating practices. This still needs to substantiated by field trials on large-scale elements, as well as investigating the effect of increasing the re-bar diameter and concrete cover on concrete Q_{28}, τ_b as well as mode of failure, which are recommended for work.

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