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Hybrid Nucleation Acceleration Method with Calcium Carbonate and Calcium Silicate Hydrate for Fast-Track Construction

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Abstract: This research focuses on achieving early strength of cement-based materials through the hybrid nucleation acceleration method. Through the study of various mortar mixtures, which incorporate components such as ordinary Portland cement (OPC), fine limestone powder (with a particle size of d_{50} : 1 µm), coarse limestone powder (with a particle size of d_{50} : 12 µm), calcium silicate hydrate (C-S-H) nucleation seeding agent, and calcium nitrate (CN), the effect of the hybrid nucleation acceleration method was investigated. When OPC was substituted with 20% fine limestone powder, a strength of 13.5 MPa was achieved at 6 h, whereas the use of coarse limestone powder only yielded 3.5 MPa within the same time frame. The mortar containing 2% C-S-H nucleation seeding agent reached an impressive 16 MPa at 6 h. Meanwhile, through the synergistic combination of fine limestone powder and C-S-H nucleation seeding agent, the 6 h early strength attained an impressive 19 MPa. The micrograph revealed that the hybrid nucleation acceleration method significantly promoted the formation of a dense network of C-S-H within the paste, thus enhancing the packing density. Measuring the heat release demonstrated that the samples accelerated with the C-S-H nucleation seeding agent and fine limestone reached the peak 160 min earlier than the OPC sample, indicating a faster hydration process. The hybrid nucleation accelerated concrete (HNAC) achieved strengths of 20 MPa and 27 MPa within 6 and 8 h, respectively, whereas the 28-day strength surpassed 70 MPa. The concrete equivalent mortar (CEM), derived from concrete, attained a compressive strength of 25 MPa within 8 h, making it suitable for repair applications. The modulus of rupture (MOR) was 7.31 MPa at 8 h and increased to 17.27 MPa at 28 days. Overall, the developed concrete and CEM with the novel hybrid nucleation acceleration method allowed for high early and long-term strength for fast-track construction to be attained.

Keywords: fast-track construction; high early strength; limestone powder; accelerator; hybrid nucleation

1. Introduction

Fast-track construction has become the standard practice for a wide range of structures, including shopping malls, warehouses, factories, semiconductor fabrication facilities, hospitals, and any project with stringent time constraints. These constructions can take the form of precast or in-situ concrete applications. Traditional normal-strength concrete (NSC) is readily available but exhibits a slow rate of strength gain, typically achieving only 30 to 40% of its ultimate strength within a day and approximately 80% within seven days [1]. In contrast, high early-strength concrete (HESC) emerges as the preferred choice for expeditious concrete building projects. HESC can attain 50 to 70% of its characteristic strength within a day [2] and is usually also accompanied by high later strength.

Both precast and in-situ construction methods can benefit significantly from using HESC. In precast applications, HESC can accelerate the de-molding time of the precast elements. The de-molding of concrete elements is only permissible once the required de-molding strength is achieved. According to the Hong Kong Buildings Department [3],



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the de-molding strength for precast reinforced concrete elements typically falls within the range of 10 MPa to 20 MPa, whereas for prestressed structures the de-molding strength exceeds 30 MPa. The conventional way to achieve the required de-molding strength of precast elements in a shorter amount of time is through steam curing [4]. Unlike HESC, concrete strength acceleration through steam curing is affected by several factors, such as temperature and the curing duration. Inappropriate control of these parameters hampers the concrete from attaining the intended performance [5]. Moreover, steam curing is recognized as a curing method that consumes substantial energy [6]. Avoiding this process can contribute to reducing the carbon emissions associated with concrete throughout its lifecycle. On the other hand, for in-situ concrete placement, the ability of HESC to achieve early strength enables the quick rotation of formwork, while the high later strength of HESC ultimately contributes to cost savings due to the reduced wall, slab, and column size and reinforcement. According to The Concrete Centre [7], the use of HESC in conjunction with self-compacting concrete results in a cost reduction of up to 8.4% for the structural frame of buildings, attributed to reduced labor and material expenses.

For repair and maintenance purposes, HESC can be advantageous in reducing downtime. In cases where the enhancement of strength, stiffness, and durability is necessary, such as retrofitting vertical elements like columns, using HESC for concrete jacketing may help with rapid repair. Shehab et al. [8] noted that using high compressive strength for concrete jacketing can reduce the section of a repaired member, making the high strength of HESC particularly advantageous for reducing the repaired section. For horizontal member repair, particularly concrete flooring, which affects large areas of construction, normally the concrete strength must reach a minimum of 12.5 MPa (partial-depth repair) and 17.3 MPa (full-depth repair) before being open to traffic. The use of HESC in such applications would speed up the repair process.

HESC generally has a high amount of very fine OPC, a low water–cement (w/c) ratio, and accelerators. Cement blended with supplementary cementitious materials is not an appropriate selection for HESC due to the slow pozzolanic effect [9]. On the other hand, the utilization of limestone filler in cement has been shown to be a more attractive solution to achieve high early strength in concrete. A recent finding by Bentz et al. [10] shows that fine limestone particles, the size of which is smaller than that of cement particles, may act as nucleation sites and accelerate the hydration of C₃S. In general, the rate of acceleration is influenced by the particle size, crystal structure, and amount of limestone. Galan et al. [11] reported that incorporating fine limestone (d_{50} : 1.2 µm) into the samples improved the early-age mechanical properties. The early strength at 4 h improved by 150% when limestone powder was incorporated into the concrete. Limestone powder in calcite crystalline form can serve as a nucleation agent. It has the ability to enhance the formation of calcium silicate hydrate (C-S-H) precipitation on the limestone's surface due to the similarity between the planar configuration of Ca and O atoms in calcite and CaO layers in C-S-H, which resembles tobermorite [12].

Besides adding a conventional accelerator to develop HESC, a new generation of accelerators based on a C-S-H nucleation seeding agent has been developed [13]. C-S-H nucleation seeding agents accelerate the cement hydration by modifying the pore solution ion concentration (e.g., Ca^{2+} , Al^{3+} , Na^+ , K^+ , SO_4^{2-} , etc.), enabling these ions to easily absorb the seed. The introduction of a C-S-H nucleation seeding agent leads to the dissolution pathway in the cementitious material being altered. Furthermore, the C-S-H growth mechanism is changed, and the C₃S dissolution is delayed or impacted due to the change in Al^{3+} and SO_4^{2-} concentration process. Furthermore, the introduction of a C-S-H nucleation seeding agent provides additional nucleation sites, which benefit from the low interfacial energy. This nucleation process yields two distinct outcomes. Firstly, it can accelerate the hydration of calcium silicate, known as the filler effect, which is attributed to the interplay between C-S-H precipitation and C₃S dissolution. Secondly, C-S-H nucleation seeding shifts the nucleation and growth of C-S-H gel away from dissolving clinker particles.

This enhances secondary heterogeneous nucleation within the capillary porosity [15]. The repositioning of C-S-H gel from the surfaces of C_3S particles to the pore space results in a more uniform distribution of C-S-H throughout the paste [16]. Consequently, this reduces the porosity, improves the mechanical strength, and diminishes the permeability of the final binder [17].

The present study thus focuses on the innovation and advancement in early-strength technology utilizing the hybrid nucleation acceleration method by incorporating fine limestone and a C-S-H nucleation seeding agent. The aim of the research is to obtain concrete with a compressive strength of at least 15 MPa within 6 h, which would be highly beneficial to precast and in-situ building construction. By using the same mix design, a mortar repair material is also derived for fast-track repair application.

2. Materials and Methods

2.1. Raw Materials

The cement (OPC) used for this research was CEM I 52.5 N from YTL Cement Bhd., which fulfilled the requirement in BS EN 197-1. The Blaine fineness of the cement was measured as $364 \text{ m}^2/\text{kg}$, and its average particle size was 12.15 µm. Two types of limestone powder were used as nucleation agents. The first was Betocarb F-IP, and this limestone powder (LSP1) has an average particle size of 1.37 µm. The second limestone powder (LSP12) was Betocarb HP-MI, with a measured average particle size of 14.43 µm. Both products were from OMYA Malaysia Sdn. Bhd. The field emission scanning electron microscope (FESEM) images of the OPC and LSP are shown in Figure 1, while the properties of the OPC and limestone powder are given in Table 1. Additionally, the particle size distribution of the OPC and limestone powder is presented in Figure 2.





Figure 1. FESEM image under 5000× magnification: (a) OPC; (b) LSP1; (c) LSP12.

Property	OPC	LSP 1	LSP 12
SiO ₂	19.18	-	-
Al_2O_3	5.07	-	-
Fe ₂ O ₃	3.60	0.1	0.1
CaO	63.34	-	-
MgO	0.46	-	-
SO_3	2.79	-	-
CaCO ₃	-	95.0	97.5
MgCO ₃	-	4.0	1.5
HCl insoluble content	-	0.9	0.9
Total alkalis	0.63	-	-
Loss on ignition	3.30	-	-
$C_3 \tilde{S}$	49.96	-	-
C ₂ S	19.10	-	-
C ₃ A	7.34	-	-
C ₄ AF	10.95	-	-
Blaine fineness (m ² /kg)	364	-	-
Specific surface area (m^2/g)	0.88	7.47	0.95

Table 1. Property of cement and limestone powder in %.



Figure 2. Particle size distribution of OPC and limestone powder.

The coarse aggregate used was 20 mm graded granite. The fine aggregate used was river sand with a maximum particle size of 4 mm. The basic properties of the aggregates are shown in Table 2.

Tab	le 2	2.	Basic	properti	ies of	the	aggre	gates.
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Property	Coarse Aggregate	Fine Aggregate
Designation (mm)	4/20	0/4
Grading	Graded	Graded
Category	Gc90/15	G _F 85
Particle fineness	-	MP
Fineness modulus	-	2.8
Fines content	f_4	f ₃
Specific gravity	2.65	2.63
Water absorption (%)	0.65	0.30
Los Angeles coefficient	LA30	-

The polycarboxylate ether-based superplasticizer (SP) used was Master Glenium 8522, which complied with BS EN 934-2 and had a solid content of 35%, with a water-reducing power ranging from 30% to 40%. The liquid-based calcium nitrate (CN) was Nitcal LQ50.

It had a solid content of 50%, and the pH ranged from 7 to 9. The C-S-H nucleation seeding agent was Master X-Seed 100, with a pH ranging from 10 to 12 and a density of 1.15 g/mL. The measured solid content of the C-S-H nucleation seeding agent was 22%.

2.2. Mix Proportioning and Sample Preparation

To investigate the accelerating effect of the fine limestone powder and accelerating admixture, the compressive strength (using mortar samples) and heat release measurement (using paste samples) were investigated. The amount of cementitious material and w/c were fixed for the samples. Additional water from the aggregate and admixtures were corrected to ensure the w/c was constant throughout the process.

As given in Table 3, for the mortar test samples, the level of cement replacement with limestone powder was investigated up to 20%, while both accelerating admixtures (CN and C-S-H nucleation seeding agent) were dosed up to 2%. In the initial stage of mortar preparation, the OPC, limestone powder, and sand were mixed for 1 min at a low speed. Subsequently, water and SP were added to the mixture and mixed for an additional 1 min at high speed. The resulting mortar was then cast into cubic molds with 50 mm edges and compacted with a vibrating table. The cubic samples were tested at intervals of 6 h, 8 h, 24 h, and 28 days. The cubes for the 6 h, 8 h, and 24 h tests were stored at room temperature (27 ± 3) °C and covered with plastic sheets before undergoing the tests. The cubes meant for the 28-day test were de-molded after 24 h and immersed in water before undergoing further tests. For each compressive strength measurement, a set of three 50 × 50 × 50 mm³ samples was tested in accordance with BS EN 12390-3 [18] at the respective ages.

Table 3. Mortar mix for the early age compressive strength test.

Mix	Cement (g)	LSP1 (g)	LSP12 (g)	Sand (g)	C-S-H (g)	CN (g)	SP (g)	w/c
OPC	450	-	-	1350	-	-	4.5	0.35
10LSP1	405	45	-	1350	-	-	5.0	0.35
20LSP1	360	90	-	1350	-	-	5.3	0.35
10LSP12	405	-	45	1350	-	-	4.8	0.35
20LSP12	360	-	90	1350	-	-	5.0	0.35
C-S-H1	450	-	-	1350	4.5	-	4.5	0.35
C-S-H2	450	-	-	1350	9.0	-	4.5	0.35
CN1	450	-	-	1350	-	4.5	4.5	0.35
CN2	450	-	-	1350	-	9.0	4.5	0.35
C-S-H2-10LSP1	405	45	-	1350	9.0	-	5.0	0.35
C-S-H2-20LSP1	360	90	-	1350	9.0	-	5.3	0.35
CN2-20LSP1	360	90	-	1350	-	9.0	4.5	0.35

The early strength of the mortar served as the basis for determining the optimal level of limestone powder and accelerator dosage. By utilizing the optimal dosage of limestone powder replacement and accelerators, a combination of both materials was employed to investigate the synergistic acceleration effect. Then, paste samples (Table 4) were selected based on the corresponding mortar samples and were prepared to examine the early-age hydration rate. No superplasticizer (SP) was introduced in the paste sample preparations to eliminate the potential influence of an acceleration effect from the SP.

Table 4. Paste mix for heat release measurement.

Mix	Cement (g)	LSP1 (g)	C-S-H (g)	CN (g)	w/c
P-OPC	450	-	-	-	0.35
P-20LSP1	360	90	-	-	0.35
P-C-S-H1-20LSP1	360	90	4.5	-	0.35
P-C-S-H2-20LSP1	360	90	9.0	-	0.35
P-CN2-20LSP1	360	90	-	9.0	0.35

All of the powder was mixed for 1 min, followed by the addition of water and accelerators. Then, the mixture was mixed for another 1 min. The paste samples were kept in a thermal flask to prevent the heat from escaping to the surroundings. Thermistors from NTC, measured in a temperature range of -55 °C to 125 °C, were inserted into the thermal flask to measure the temperature. The data were logged with Campbell Scientific CR10X Datalogger every 10 min up to 2000 min.

After determining the optimum mixture, the HESC mix was designed with the absolute volume method for real-world in-situ and precast applications. This concrete mix was denoted as hybrid nucleation accelerated concrete (HNAC). For repair application purpose, a concrete equivalent mortar (CEM) was developed based on the method by Ghorbel et al. [19] by removing coarse aggregate from the HNAC. The mix designs for HNAC and CEM are given in Table 5. For practical application, the mixes were designed based on BS 8500-1 to meet the DS-2 class, while the chemical resistance was designed to meet the ACEC-4z and DC-4z classes.

Table 5. HNAC and CEM mix designs.

Mix	Cement (kg/m ³)	LSP1 (kg/m ³)	Coarse Aggregate (kg/m ³)	Fine Aggregate (kg/m ³)	C-S-H (kg/m ³)	SP (kg/m ³)	w/c
HNAC	360	90	820	965	9	5	0.35
CEM	570	140		1295	14.2	12.0	0.35

For HNAC preparation, the coarse aggregate and fine aggregate were mixed for 1 min, followed by the addition of OPC and limestone powder, and further mixed for 1 min. Subsequently, water and SP were added and mixed for another 1 min. The workability of the concrete was designed for slump class S3, and a slump test, conducted according to BS EN 12350-2, was employed to verify that the concrete achieved the desired workability. The fresh concrete was cast into cubes with 100 mm edges and compacted using a vibration table. For each compressive strength test, a set of three samples of $100 \times 100 \times 100 \text{ mm}^3$ was tested according to BS EN 12390-3 at the respective age. The concrete cubes underwent testing at specific intervals of 6 h, 8 h, 24 h, 7 days, and 28 days. The samples designated for testing at 6 h, 8 h, and 24 h were stored at room temperature (27 ± 3) °C, covered with plastic sheets, and then subjected to tests. Cubes scheduled for testing at 7 days and 28 days were de-molded after 24 h and immersed in water before undergoing additional testing.

CEM is considered a mortar-based repair material without coarse aggregates like commercial preblended repair material. The preparation of CEM was similar to that of normal mortar, and all samples were mixed until homogenous and molded into different samples. For compressive strength measurement, three samples measuring $50 \times 50 \times 50$ mm³ were tested in accordance with BS EN 12390-3. In the modulus of rupture (MOR) test, the mortar was cast into prisms measuring 40 mm \times 40 mm \times 160 mm. Three prisms were tested for each measurement in accordance with BS EN 196-1. The compressive strength was determined and MOR tests carried out on the samples examined at 6 h, 8 h, and 24 h, which were maintained at room temperature (27 \pm 3) °C and covered with plastic sheets before testing. Conversely, all other samples were de-molded at 24 h and immersed in water before proceeding with additional testing. For the modulus of elasticity (MOE) test, the mortar was cast in cylinders with a diameter of 100 mm and a height of 200 mm. Three samples were prepared and tested at 28 days according to BS EN 12390-13. To ensure the results were precise, the end preparation of the cylinder was carried out via the grinding method. The surface was checked with a filler gauge and an engineering straight edge to ensure the surface for loading was flat. For drying shrinkage measurement, three samples were prepared in prisms with sizes of 25 mm \times 25 mm \times 285 mm and tested according to ASTM C157 up to 28 days under dry-air (relative humidity between 50% to 60%) and moist conditions. To analyze the morphology of the paste, images of specific fractured surfaces were taken using a Zeiss Gemini Auriga FESEM 24 h after the process. The fractured

specimens were initially sliced into smaller pieces and securely attached to specimen stubs. Following that, a thorough cleaning was carried out to eliminate dust using a blower. Ultimately, the samples were examined under a FESEM at a magnification of $5000 \times$.

3. Results and Discussion

The early strength measurement was carried out on blends containing different replacement levels of limestone powder ranging from 10% to 20%. The following sections provide insights into the influence of the fine limestone powder, C-S-H nucleation seeding agent, and CN, as well as the synergistic effects of the fine limestone powder and C-S-H nucleation seeding agent. Based on the outcome of the hybrid nucleation acceleration, a concrete mix was designed to show the possible application of hybrid nucleation in precast and in-situ concrete work. A CEM was also developed to study the behavior of the repair material for vertical and horizontal member repair work.

3.1. The Effect of Limestone Powder

A comparison was made between the two types of limestone powder at various replacement levels, and the outcomes are detailed in Table 6. Using limestone powder mortar with different fineness and replacement levels enhanced the early-age compressive strength of the mortar. The positive effect of limestone powder was noticeable at as early as 6 h. Specifically, at 6 h, the highest early strength of 13.6 MPa was achieved with 20% replacement of LSP1, whereas the sample with coarser limestone powder (20% LSP12) recorded a strength of 3.5 MPa. At this age, the strength of the OPC sample was recorded as zero and negligible but began to exhibit strength gain after 6 h, with a recorded strength of 5.1 MPa at 8 h. At 8 h, the strength of the 10LSP1 sample and 20LSP1 sample increased by 282% and 68%, respectively. The average 1-day strength of the sample with 10% and 20% LSP1 replacement was approximately 45 MPa, marking a notable 5 MPa or 11% improvement over the OPC sample. Moreover, the 20LSP1 sample exhibited the highest strength at 28 days, surpassing the OPC sample by 10.2%. Although a similar trend of strength increment was observed for the LSP12 sample, both the early and later strength measurements were inferior to those of the LSP1 sample.

Mix		Compressive S	Strength (MPa)	
IVIIX	6 h	8 h	24 h	28 Days
OPC	0.0	5.1 (0.3)	40.8 (0.8)	62.3 (0.6)
10LSP1	4.5 (0.4)	17.2 (0.4)	44.8 (0.5)	63.4 (0.7)
20LSP1	13.6 (0.7)	22.8 (0.5)	45.3 (0.6)	65.9 (0.4)
10LSP12	0.0	10.2 (0.3)	39.1 (0.5)	57.8 (0.3)
20LSP12	3.5 (0.6)	15.9 (0.2)	41.2 (0.3)	58.2 (0.8)

Table 6. Effects of limestone powder on compressive strength.

Standard deviation is presented in parentheses.

According to Vance et al. [20], limestone powder finer than cement particles may act as a nucleation site and accelerate the cement hydration. The OPC has an average particle size of d_{50} , normally ranging from 10 µm to 15 µm [10,21]. The d_{50} of the OPC used was 12.15 µm. Hence, LSP1 effectively boosted the early strength of mortar by providing a nucleation site for nucleation seeding. On the other hand, since the average particle size of LSP12 was measured as 14.43 µm, the role of LSP12 to serve as a nucleation site for seeding was deemed insignificant. Due to the larger particle size, the dilution effect was more prominent [21]. Hence, lower later strength was observed when using LSP12 as a cement replacement material.

In contrast to the use of coarse limestone powder, the mortar containing the finer LSP1 exhibited a notable improvement in both early and later strengths. It was noted that fine crystalline calcite also facilitated the nucleation effect for cement hydration [10]. Berodier and Scrivener [22] explained that the enhanced precipitation of C-S-H is due to the similarity

between the planar configuration of Ca and O atoms in calcite and CaO layers in C-S-H. The nucleation seeding effect increased with the increase in LSP1 content, demonstrated by the 20LSP1 sample, which exhibited the highest acceleration effect. According to Cao et al. [23], increasing fine limestone powder leads to more nucleation sites, which can aid the formation of C-S-H. The enhanced later strength observed in samples replaced with LSP1 can be attributed to the improved packing density of the paste. Bosiljkov [24] similarly reported that the inclusion of finer limestone powder can increase the concrete strength by enhancing the packing density.

3.2. The Effect of Conventional Admixture and C-S-H Nucleation Seeding Agent

Table 7 illustrates the effects of the C-S-H nucleation seeding agents and CN on the strength of the mortar. The sample incorporating a 2% C-S-H nucleation seeding agent demonstrated the highest early strength, which was recorded as 16.1 MPa at 6 h and 23.7 MPa at 8 h. A similar trend was observed for the sample with a 2% CN addition, achieving a strength of 4.3 MPa at 6 h and 15.8 MPa at 8 h. The optimal dosage for both the C-S-H nucleation seeding agent and CN was determined to be 2% due to the highest early strength achieved. When comparing the two accelerators at their optimal dosages, the C-S-H seeding agent exhibited significantly superior performance compared to CN. Specifically, at 6 h, the strength of the C-S-H sample was 3.7 times greater than that of CN, and at 8 h, it showed an improvement in strength by 1.5 times. The impact of the C-S-H seeding agent was less significant at 24 h, for which the improvement was recorded at about 3%. At a 2% dosage, the 24 h strength of CN improved by 13.2%. Similarly, Kičaitė et al. [25] also reported that additional CN improved the early strength by about 4% to 12%. It was noted that the samples containing C-S-H nucleation seeding agent and CN exhibited higher compressive strength than the OPC sample at 28 days. The maximum improvement for the C-S-H sample was about 5%, whereas it was around 3% for the CN sample. The findings aligned with those of Cuesta et al. [14], who concluded that the addition of a C-S-H nucleation seeding agent improved the later strength of the samples.

	Compressive S	Strength (MPa)	
6 h	8 h	24 h	28 Days
0.0	5.1 (0.3)	40.8 (0.8)	62.3 (0.6)
7.1 (0.5)	19.5 (1.0)	42.1 (0.5)	64.0 (0.9)
16.1 (0.3)	23.7 (0.9)	41.4 (0.8)	65.3 (0.4)
3.2 (0.1)	14.1 (0.3)	47.0 (0.5)	63.5 (0.4)
4.3 (0.4)	15.8 (0.6)	49.9 (0.4)	64.1 (0.3)
	6 h 0.0 7.1 (0.5) 16.1 (0.3) 3.2 (0.1) 4.3 (0.4)	6 h 8 h 0.0 5.1 (0.3) 7.1 (0.5) 19.5 (1.0) 16.1 (0.3) 23.7 (0.9) 3.2 (0.1) 14.1 (0.3) 4.3 (0.4) 15.8 (0.6)	Compressive Strength (MPa)6 h8 h24 h0.05.1 (0.3)40.8 (0.8)7.1 (0.5)19.5 (1.0)42.1 (0.5)16.1 (0.3)23.7 (0.9)41.4 (0.8)3.2 (0.1)14.1 (0.3)47.0 (0.5)4.3 (0.4)15.8 (0.6)49.9 (0.4)

Table 7. Effects of the C-S-H seeding agent and CN on compressive strength.

Standard deviation is presented in parentheses.

In the presence of CN, the rate of hydration of calcium silicate phases such as C₃S and possibly C₂S increases [26]. Hence, the early strength of the sample with CN increased. Unlike conventional CN accelerators, C-S-H nucleation seeding agents are more effective at accelerating cement hydration through a shortening of the induction and dormant period of the cement hydration process [27]. C-S-H seeding particles accelerate the hydration by modifying the ion concentration of the pore solution and altering the dissolution pathway in the cementitious system. As a result, the C-S-H growth mechanism is changed. The nucleation seeding agent also provides secondary nucleation sites to promote the growth of hydration products [15]. After incorporating a C-S-H nucleation seeding agent, the C-S-H gels were distributed more uniformly throughout the paste, reducing porosity within the binder and enhancing its compressive strength [17].

3.3. The Effect of Hybrid Nucleation Acceleration

Upon considering the influence of fine limestone powder on early strength and identifying the optimal accelerator dosage, a study was conducted on three mixes combining LSP1 with C-S-H nucleation seeding agent or CN. The hybrid acceleration effects of these mixes are presented in Table 8. At 6 h, the C-S-H2-20LSP1 sample achieved the highest early strength at 19.4 MPa, followed by the C-S-H1-20LSP1 sample, with a recorded strength of 16.9 MPa. In comparison, the acceleration effect of CN2-20LSP1 was lower than the hybrid nucleation seeding effect, resulting in a recorded strength of 14.3 MPa.

Min		Compressive S	Strength (MPa)	
	6 h	8 h	24 h	28 Days
OPC	0.0	5.1 (0.3)	40.8 (0.8)	62.3 (0.6)
C-S-H2-10LSP1	16.9 (0.2)	24.7 (1.1)	45.4 (0.7)	64.9 (1.2)
C-S-H2-20LSP1	19.4 (0.3)	27.9 (0.9)	46.6 (0.7)	64.5 (0.9)
CN2-20LSP1	14.3 (0.3)	20.6 (0.8)	47.9 (1.0)	65.8 (0.6)

Table 8. Effects of hybrid nucleation acceleration on compressive strength.

Standard deviation is presented in parentheses.

Figure 3 also shows that C-S-H2-20LSP1 exhibited higher strength than the samples independently containing LSP1 or C-S-H nucleation seeding agent. Compared to the 20LSP1 sample, the 6 h strength exhibited a remarkable 43% improvement, whereas the 8 h strength increased by 22%. Additionally, compared to the sample with the 2% C-S-H nucleation seeding agent (C-S-H2), there was a 20% and 18% increment in the 6 h and 8 h strength, respectively.



Figure 3. Early strength of the samples with hybrid nucleation acceleration compared to the samples with only 20% LSP1 or 2% C-S-H nucleation seeding agent.

In summary, there is a synergistic effect between fine limestone powder and the C-S-H nucleation seeding agent. It was noted that the CN2-20LSP1 sample with 20% LSP1 replacement and 2% CN exhibited higher early strength compared to CN2, further highlighting the nucleation effect of limestone powder in contributing to the early strength.

3.4. Surface Morphology Study with FESEM

The surface morphologies of various mixtures are shown in Figure 4. It is important to note that strength is inversely related to porosity, meaning that a denser surface structure indicates higher strength [28]. When compared to the OPC sample, the sample with LSP1 had a denser surface and hence higher compressive strength. Additionally, it is worth mentioning that there was evidence of the development of C-S-H on the surface of the limestone in the sample. Conversely, in the 20LSP12 sample, weak calcium hydroxide crystals and pores were observed, which adversely impact early strength. This finding

aligns with the results reported by Briki et al. [21], where finer limestone powder resulted in better packing, while coarser limestone powder increased porosity. Finer limestone powder appeared to enhance the packing density of the paste and accelerate the hydration of C-S-H crystals.



(**g**)

Figure 4. FESEM of sample (**a**) OPC; (**b**) 20LSP1; (**c**) 20LSP12; (**d**) CN2; (**e**) C-S-H2; (**f**) C-S-H2-20LSP1; (**g**) 20LSP1-2CN.

Furthermore, the presence of CN in the sample (CN2) promoted the growth of needlelike C-S-H, which usually appears during the early stage of cement hydration. In contrast, mesh-type C-S-H, characterized by its shorter fibrous structure, forms later in the hydration development process [29]. Notably, the presence of a C-S-H nucleation seeding agent and LSP1 led to the growth of mesh-type C-S-H on the fracture surface of the sample (C-S-H2-20LSP1) at an early age, indicating improved packing. The dense mesh-type C-S-H was seen to be growing on the cement particle, particularly in C-S-H2-20LSP1, owing to the hybrid nucleation effect. In contrast, the C-S-H growth on CN2-20LSP1 was less dense compared to that on the sample accelerated with LSP1 and C-S-H nucleation seeding agent (C-S-H2-20LSP1).

3.5. Heat Release

By measuring the heat release of the paste sample (Figure 5), the hydration rate could be determined. For the P-OPC control sample, the peak of the heat release curve occurred at 500 min. When OPC was replaced with limestone powder, the heat release curve shifted earlier than that of the control sample. It was noted that the peak of the heat release curve for P-20LSP1 occurred at 380 min, which was 120 min earlier than that of the P-OPC sample, indicating faster hydration (Figure 5). Galan et al. [11] reported similar findings when replacing OPC with 5 to 15% limestone powder with a size of 1.2 μ m. Moreover, the heat release was affected by the type of limestone powder. The heat release of the sample containing 1 μ m limestone powder (P-20LSP1) was higher than that of the sample containing 12 μ m limestone powder (P-20LSP12), and this difference in heat release could have been a contributing factor to the difference in strength between the two samples. Vance et al. [20] similarly observed that the sample containing 0.7 μ m limestone powder exhibited a higher level of heat release compared to the sample containing 15 μ m limestone powder. It should be noted that the recorded maximum temperature for samples with limestone powder was lower than that of the control sample due to the dilution effect.



Figure 5. Heat release measurement of the samples.

The heat release curve of the sample featuring hybrid nucleation (P-C-S-H2-20LSP1 and P-C-S-H1-20LSP1) shifted to the left, indicating a faster hydration process than that of samples without a C-S-H nucleation seeding agent (P-20LSP1) and the standard OPC sample. Specifically, the peaks of P-C-S-H2-20LSP1 and P-C-S-H1-20LSP1 occurred 40 min and 30 min earlier, respectively, than the peak of P-20LSP1. A similar trend was observed for P-CN2-20LSP1, with its peak occurring only 10 min earlier than that of P-20LSP1. These results demonstrate a significant enhancement in accelerating cement hydration in samples utilizing the hybrid nucleation acceleration method.

3.6. Application of HNAC in Building Construction

Based on the earlier findings, HNAC was formulated using the hybrid nucleation acceleration method, incorporating 20% cement replacement with LSP1 and the addition of a 2% C-S-H nucleation seeding agent. The compressive strength of HNAC is presented in Table 9. HNAC was able to achieve 20 MPa within 6 h and more than 25 MPa in 8 h. For precast concrete, generally, the de-molding strength lies between 10 MPa and 20 MPa. NSC can only achieve more than 10 MPa after 12 h to 18 h [30]. The usage of HNAC can therefore significantly reduce the de-molding time by at least half. Thus, with 6 h demolding strength, the precast production can be doubled [31]. If the de-tensioning strength of a pre-stressing structure is 30 MPa, the de-tensioning process can safely be estimated to commence after 12 h. For in-situ construction, the high early strength of HNAC can help to reduce the formwork rotation time, whereas the high later strength (>70 MPa) can help to decrease the column size and increase the lettable floor area of the building. Similar benefits have been reported by The Concrete Centre [7] when utilizing HESC.

Table 9. Compressive strength of HNAC.

Mix -		Compressive Strength (MPa)						
IVIIX	6 h	8 h	24 h	7 days	28 Days			
HNAC	20.3 (1.1)	27.7 (0.8)	46.4 (0.9)	61.4 (1.4)	72.1 (1.2)			

In terms of OPC consumption, the concrete achieved a strength of 27.7 MPa within 8 h using only 360 kg/m³ of OPC. In other fast-track concretes, especially repair materials, the OPC content varies from 386 kg/m³ to 564 kg/m³, and the 8 h strength ranges from 17.0 MPa to 27.3 MPa [32]. The strength-to-OPC (S/O) ratio of HNAC stands at 0.08 MPa/kg OPC, whereas fast-track concrete typically falls within the range of 0.04 to 0.05 MPa/kg OPC. For the 28-day strength of concrete, the S/O ratio was 0.20 MPa/kg OPC. In comparison, when examining concrete with a characteristic strength exceeding 60 MPa, the S/O ratio ranged from 0.12 to 0.13 MPa/kg OPC [33]. This indicates that the concrete prepared using the hybrid nucleation accelerated method can achieve high early and later strengths but with a lower carbon footprint than conventional HESC.

3.7. Application of CEM as a Repair Material

By removing the coarse aggregates of HNAC, CEM was developed. As depicted in Table 10, the CEM exhibited impressive compressive strength comparable to HNAC. It achieved an early strength of 18.6 MPa in just 6 h, whereas the 8 h strength was recorded as 25 MPa, signifying that most repair tasks can be accomplished within 8 h. CEM can be considered a rapid repair material due to the ability to achieve 25 MPa within 8 h [34]. When dealing with concrete slab repairs that affect extensive areas, employing NSC can result in work disruptions due to slow strength gain. Therefore, the application of rapid repair material can be highly advantageous. The required minimum compressive strength varies depending on the depth of the repair, ranging from 12.5 MPa to 17.3 MPa. CEM can be added with coarse aggregate for full-depth repair when necessary. Repairs can be conducted during nighttime to minimize work interruptions, ensuring completion within 6 to 8 h.

Table 10. Compressive strength of hybrid nucleation-accelerated CEM.

Mix		Comp	ressive Strength	(MPa)	
IVIIX	6 h	8 h	24 h	7 Days	28 Days
CEM	18.6 (0.9)	26.8 (1.1)	47.5 (0.8)	60.4 (1.0)	73.2 (1.3)

Standard deviation is presented in parentheses.

The MOR or flexural strength is a crucial parameter, particularly for the repair of horizontal concrete members. The MOR of the sample was studied using the CEM (Table 11). According to Teo and Lee [35], the MOR of OPC concrete is about 10% to 20% of the concrete compressive strength. In this study, the MOR of the CEM was approximately 25% of its compressive strength, which is attributable to the hybrid nucleation effect, which led to the formation of a denser C-S-H within the paste.

Table 11. MOR of the CEM.

Mix			MOR (MPa)		
Mix -	6 h	8 h	24 h	7 Days	28 Days
CEM	5.3 (0.4)	7.3 (0.5)	9.7 (0.3)	15.8 (0.6)	17.3 (0.4)

Standard deviation is presented in parentheses.

The MOR-to-compressive strength (m/c) ratio is an essential parameter in assessing the toughness and cracking resistance of concrete materials, as reported by recent research [36]. A higher m/c ratio indicates superior resistance to cracking, signifying the ability of a concrete mix to withstand tensile stresses and maintain structural integrity. In the present study, the CEM repair material achieved an m/c ratio of 0.27 in 8 h. A typical C32/40 concrete has an m/c ratio of around 0.17 [37], and a high-strength concrete of more than 80 MPa has an m/c ratio of 0.08 [38]. Previous findings showed that higher-strength concrete is more brittle than lower-strength concrete. Hence, the m/c ratio decreases when compressive strength increases.

For rapid repair materials such as calcium sulfoaluminate (CSA) and magnesium phosphate (MP) repair materials, the m/c ratios were recorded as 0.16 [39] and 0.10 [40], respectively. The m/c at 8 h in the present study indicates that the crack resistance of the CEM was 1.7 times superior to that of CSA and 2.7 times better than MP repair material. It was reported that repair materials like polymer-modified cementitious (PMC) material enhanced by styrene acrylic had an m/c ratio range of 0.13 to 0.16 [41]. Similarly, PMC modified with vinyl acetate yields an m/c ratio ranging from 0.14 to 0.20. Thus, the m/c of CEM is at least 1.35 times better than PMC material. Thus, it is worth noting that the 28-day m/c ratio in the current study (0.24) surpassed that of other cementitious materials, equating its toughness to that of polymer-modified repair materials. The findings have important implications for the overall performance and longevity of the repaired section, contributing to a more resilient and long-lasting structure.

The MOE of the developed material in the present study was recorded as 40.5 ± 0.4 GPa, similar to the MOE of the C60/75 concrete [1]. The CEM also shared an MOE comparable to that of MP concrete (15 to 42 GPa [42,43]), CSA concrete (38 to 42 GPa [44]), and calcium aluminate cement (CAC) concrete (25 to 45 GPa [45]). According to Kiani et al. [46], the MOE plays a critical role in avoiding compatibility issues; excessive differences in MOE between the repair material and the original substrate can cause compatibility issues. When undergoing surface repair, the ACI Committee 364 [47] explained that repair material with a lower MOE than the substrate might not be able to support as much load as the original substrate. Hence, there are chances that failure may occur in the original substrate. On the other hand, if a repair material with a higher MOE than the substrate is selected, it can carry a greater load than the substrate and potentially concentrate thermal and shrinkage-induced stresses in the repair material. However, this concentration of stress could potentially result in cracking or the detachment of the repair.

Although the drying shrinkage for moist-cured CEM samples was insignificant, the air-dried CEM samples exhibited higher shrinkage (Figure 6). The 28-day drying shrinkage for the water-cured sample was recorded as $-52 \ \mu\epsilon$. On the other hand, in the air-dried CEM samples, the drying shrinkage was $-770 \ \mu\epsilon$. It was noted that more than 80% of shrinkage occurred within 14 days, and almost half of the shrinkage was observed within the first 7 days.



Figure 6. Drying shrinkage of the CEM.

According to Emmons and Vaysburd [48], the drying shrinkage of the CEM is categorized as moderate shrinkage material, where the shrinkage falls within the range of $-500 \ \mu\epsilon$ and $-1000 \ \mu\epsilon$. Based on the ASTM C928 requirement, the maximum shrinkage for rapid hardening repair material is limited to a maximum of $-1500 \ \mu\epsilon$. The results of the CEM indicate that the shrinkage of this material falls within this specified limit, measuring at approximately half of the designated threshold. Consequently, it is deemed to be a suitable and acceptable repair material for practical application. However, compared to other repair materials, such as CSA-based material (shrinkage of $-250 \ \mu\epsilon \ to \ -600 \ \mu\epsilon \ [49])$ and PMC material (shrinkage of $-200 \ \mu\epsilon$ [50]), the CEM recorded shrinkage that was 1.3 to 3 times higher and 3.85 times higher, respectively. The shrinkage of the CEM was also slightly higher compared to that of traditional OPC concrete, which lies within the range of $-500 \ \mu\epsilon$ to $-600 \ \mu\epsilon$ [51]. Morgan [52] emphasized that, more importantly, the shrinkage property of the repair material should be less than that of the original substrate to prevent compatibility issues. Shrinkage within the new repair material will lead to stress developing on the bonding interface and cause debonding or failure. Thus, in real-world applications, when utilizing CEM for repair, proper curing of repair is required to prevent debonding issues.

4. Conclusions

The current study was designed to determine the effect of hybrid nucleation acceleration on cement hydration. Based on the results and findings, the following conclusions can be drawn:

- Fine limestone powder enhances cement hydration more effectively than coarse limestone powder. Substituting 20% fine limestone powder for OPC in mortar resulted in a 6 h strength of 13.5 MPa, whereas using coarse limestone powder achieved only 3.5 MPa within the same time frame. Mortar containing a 2% C-S-H nucleation seeding agent achieved strength of 16 MPa at 6 h, surpassing the CN-based mortar, with 4.3 MPa. A synergistic combination of 20% fine limestone powder and 2% C-S-H nucleation seeding agent reached an impressive 6 h early strength of 19 MPa.
- 2. The surface morphology observation of samples produced with the hybrid nucleation acceleration method revealed the formation of a dense mesh of C-S-H network within the paste, contributing to enhanced mechanical performance.
- 3. By incorporating a 2% C-S-H nucleation agent and 20% fine limestone powder in the mixture, the peak of heat release was observed to shift 160 min earlier compared to the OPC reference mix, indicating an accelerated hydration process.
- 4. HNAC achieved 20 MPa and 27 MPa strengths within 6 and 8 h, respectively. The 28-day strength of the concrete surpassed 70 MPa, which means that it can be used as

high-strength concrete. These findings are expected to yield cost savings in precast and in-situ construction projects by reducing mold and formwork turnover times.

- 5. CEM repair material derived from HNAC attained a compressive strength of 25 MPa within 8 h, making it suitable for structural applications. The MOR of the CEM was 7.31 MPa at 8 h and increased to 17.27 MPa at 28 days. Thus, the m/c ratio for the CEM was 0.27 at 8 h and 0.24 at 28 days, demonstrating its superior resistance to cracking.
- 6. The MOE of the CEM was 40.5 GPa, comparable to normal to high-strength concrete and similar to other repair materials such as MP, CSA, and CAC concretes. In contrast, the air-dried drying shrinkage of the CEM was measured at $-770 \ \mu\epsilon$, which was slightly higher than that of typical concrete. However, it is within the $-1500 \ \mu\epsilon$ limit given by ASTM C928.
- 7. Overall, the HNAC and CEM developed with the hybrid nucleation acceleration method allow high early and long-term strength to be attained for fast-track construction, with potential cost savings and superior resistance to cracking.
- 8. The current research mainly focused on investigating the impact of the hybrid nucleation acceleration method on OPC binder within the temperature range of 24 °C to 30 °C. In the future, research could explore the effect of the hybrid nucleation acceleration method across a wider temperature range (-20 °C to 40 °C) and its compatibility with other supplementary cementitious materials. These efforts aim to enhance understanding and have the potential to expedite project timelines in the repair and construction industry.

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