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Nanomaterial-Enhanced Self-Consolidating High-Performance Concrete: Investigating the Effects of Nano POFA Incorporation

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ABSTRACT

Palm oil fuel ash (POFA) is one of the agricultural wastes that negatively impacts the environment and health because it is abundantly disposed of in landfills without commercial value. The primary objective of this research is to investigate the potential incorporation of Nano-POFA (NaPOFA) as a cement replacement into Self- Self-Consolidating High-Performance Concrete (SCHPC) through an experimental study to produce concrete with improved durability and strength. The NaPOFA is prepared using high-energy milling and introduced as cement replacement from 1% - 10%. The evaluation of the SCHPC NaPOFA mix encompasses four tests: physical properties, assessed using a particle size analyser; fresh properties, evaluated through a flow table test; and mechanical properties, determined through a splitting tensile strength test, with supported software analysis on Design Expert. The results reveal that the optimal percentage of Nano-POFA replacement is 1%, resulting in an impressive 18.18% increase in flowability and a substantial 12.12% increase in splitting tensile strength compared to the control mix. In conclusion, the SCHPC NaPOFA mix developed in this study demonstrates potential applications for constructing durable and sustainable structures, contributing to reduced cement consumption and mitigating the carbon footprint in the construction industry.

INTRODUCTION

The construction industry's reliance on concrete has soared in recent years, with global cement consumption surpassing 1.5 billion tonnes in 2000, coinciding with an average per capita usage of about one cubic meter of concrete. Concrete has solidified its status as a cornerstone material in the construction industry of the 21st century due to its cost-effectiveness, straightforward manufacturing processes, and production efficiency. Concrete's versatility knows no bounds, catering to specialized applications, including self-

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compacting variants, underwater compositions, high-performance roller-compacted forms, and reactive powders. The evolution of modern concrete has resulted in a refined construction substance tailor-made for specific functions, achieving both ease of use and environmental economy.

For a concrete blend to be deemed high strength (HSC), its compressive strength must fall within the range of 40 N/mm² to 100 N/mm². In Malaysia, high-strength concrete is extensively utilized due to its superior strength and endurance compared to conventional concrete. Employing high-strength concrete in structurally efficient and eco-friendly designs confers manifold advantages, such as enabling the construction of expansive bridges and augmenting the longevity of bridge decks. The water-to-binder ratio is pivotal in shaping high-strength concrete, as a lower ratio increases strength. Ultra-high-performance concrete typically incorporates a cement content ranging from 700 kg/m³ to 1100 kg/m³, nearly threefold that of standard concrete (Bandukwala & Sonkusare, 2016). According to Jagana & Vinod (2017), concrete's low permeability necessitates a reduced water-to-cement ratio, which is pivotal for exceptional durability. This underscores the requirement for increased cement content to maintain the necessary water and cement paste for workable concrete. At the same time, superplasticizers play a crucial role in sustaining workability within low water-to-cement ratio compositions.

Numerous strategies have been investigated, focusing on enhancing the paste binder to bolster concrete durability. In the early 1980s, Japan faced concrete durability challenges due to inadequately skilled labour, which compromised construction quality (Adebayo et al., 2020). In response, self-compacting concrete (SCC) emerged to ensure durable concrete structures independent of construction quality. Empirical investigations by Xie et al. (2002) demonstrate that self-consolidating concrete (SCC) imbued with cementitious materials exhibits optimal workability, robust mechanical properties, and heightened durability. Contemporary concrete research has embraced nanotechnology in cement-based materials, ushering in a new era of exploration. Diverse nanomaterials have been harnessed within concrete and cement-based products. Recent attention has turned to the substantial silica oxide content and other fine particle materials within the waste, igniting studies on nano-sized Palm Oil Fuel Ash (POFA) as a source material for nano POFA concrete development (Halim et al., 2021; Hamada et al., 2022; Liew et al., 2023). POFA, originating from palm oil fields and processes supplying the palm oil industry, yields substantial solid waste. This waste, comprising fibers, shells, and empty fruit bunches, serves as fuel for steam generation, powering palm oil production. Approximately 5% of solid waste post-combustion translates to POFA producing significant production volume (Sata et al., 2004).

Consequently, the disposal of this POFA waste necessitates vast expanses of landfill, incurring substantial transportation and maintenance costs for the government. Integrating pozzolanic materials as partial cement replacements is a novel advancement in concrete technology (Abu Aisheh, 2023; Hasan et al., 2023; Sarangi & Suganya, 2024). Studies by (Adamu et al., 2024; Alharthai et al., 2024; Hamada et al., 2023; Hamada et al., 2022; Malaysian Palm Oil Board, 2022; Zhang et al., 2024) have yielded findings indicating that POFA can be integrated into concrete to produce a novel modified concrete type, with POFA's properties enhancing the composition. Although POFA's large particle size and porous structure lead to a less pronounced pozzolanic reaction, grinding can accentuate this reaction (Mashri et al., 2022).

This research introduces a promising strategy to address the considerable waste generated by the palm oil industry through its transformation into a viable substitute for cement, thus contributing to sustainability efforts. By repurposing this waste material, the study aims to mitigate its environmental impact effectively. Incorporating POFA into concrete advocates for environmentally conscious practices and encourages innovation within the construction sector. Additionally, using POFA can yield cost savings by reducing cement expenditures and optimizing concrete production processes. Integrating Nano-sized Palm Oil Fuel Ash (NaPOFA) as partial replacements in SCHPC holds significant promise in enhancing conventional SCHPC to meet the intricate demands of high-rise building structures. Furthermore, this research aligns with Sustainable Development Goal (SDG) 9, underscoring the importance of resilient infrastructure, inclusive and sustainable industrialization, and innovation. By investigating alternative cementitious

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materials like NaPOFA, the study aims to advocate for more environmentally friendly construction practices, contributing to SDG 11 (Sustainable Cities and Communities) by fostering the development of durable and sustainable concrete solutions tailored for urban environments in Malaysia.

MATERIALS AND METHOD

Material preparation

The specimens and materials utilized in this research encompass 50 mm concrete cubes, Ordinary Portland Cement (OPC), coarse and fine natural aggregates, POFA, water, and glenium employed as admixtures. The OPC employed in this investigation was sourced from Tasek Sdn. Bhd. For aggregates, coarse aggregates with a maximum size that passes through a 10 mm sieve were chosen, while river sand with a maximum size that passes through a 4 mm sieve was employed as fine aggregate. The concrete mixing process adhered to BS EN 1008, 2002, utilizing potable tap water. In the mixing procedure to fabricate NaPOFA concrete, MasterGlenium ACE 8538 was partially incorporated as an admixture. This glenium was procured from BASF (Malaysia) Sdn. Bhd. The purpose of integrating glenium in this study was to enhance the mechanical properties of SCHPC, complementing the influence of NaPOFA within the concrete mix. The materials employed include OPC, coarse aggregates, fine aggregates, glenium, and POFA. POFA was used as cement replacement in this study and was collected from the palm oil mill at United Palm Oil Sdn. Bhd. factory at Sungai Kecil, Nibong Tebal, Pulau Pinang, Malaysia.

Sample preparation

This research highlighted using NaPOFA as a substitute for cement in concrete, at varying proportions of 1%, 3%, 5%, 7%, and 10%. Six different concrete mixes were created: one without NaPOFA (control) and five others with increasing levels of NaPOFA, labelled SCHPCNP1, SCHPCNP3, SCHPCNP5, SCHPCNP7, and SCHPCNP10. The initial step in the concrete mixing process involved the blending of cement and POFA within the mixing apparatus. After this, potable tap water and the designated admixture were introduced into the mixture and blended until a uniform cement paste was achieved. Once this uniformity was attained, the fine aggregate was carefully added to the mixture, succeeded by adding coarse aggregate. Subsequently, the mixture was left for several minutes to concrete to mix well before it was poured into the mould. The concrete cubes underwent curing for 1, 3, 7, 14, and 28 days after casting. The mix proportion employed in this study referred to the concrete design mix study by (Norhasri et al., 2017) and was slightly modified since there was no direct or conventional design for the SCHPC mix. The design mix was selected as a point of comparison because it can produce high-strength concrete using regular coarse and fine aggregate, a more affordable and widely accessible material in Malaysia. The specific design mix utilized for the concrete can be found in Table 1 for reference.

Test method and procedure

High-energy ball milling

The initial step involves subjecting the gathered raw POFA to an extensive oven-drying process for 24 hours. This drying process occurs at a controlled temperature of 110 ± 5 ºC, eliminating excess moisture from the raw material. A meticulous procedure is employed to remove coarse particles from the now-dried POFA. This is achieved by sieving the ash through a 212 µm sieve, resulting in smaller particles better suited for precise control over the filler effect. The subsequent transformation of the sieved POFA into NaPOFA involves grinding using a high-energy ball milling machine. This machine operates in conjunction with 10 mm-sized zirconium balls for a specific duration, ensuring the reduction of NaPOFA particles to their smallest attainable size. In this milling setup, the rotational speed is fixed at 200 rpm, and the weight

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ratio of zirconium balls to powder is maintained at a ratio of 1:10 comprising 50 grams of balls to 500 grams of POFA.

Table 1. Concrete mix design

Mixes	Cement (kg/m^3)	POFA (kg/m^3)	Water (kg/m^3)	Coarse aggregate (kg/m^3)	Fine aggregate (kg/m^3)	Super plasticizer (kg/m^3)
SCHPC	1000	0	160	800	433	16
SCHPCNP1	990	10	160	800	433	16
SCHPCNP3	970	30	160	800	433	16
SCHPCNP5	950	50	160	800	433	16
SCHPCNP7	930	70	160	800	433	16
SCHPCNP10	900	100	160	800	433	16

Particle size analyser

Upon completion of the milling process, the NaPOFA powder samples undergo a comprehensive analysis utilizing a zeta potential analyzer to determine their particle size characteristics. The size of the cementitious material was assessed using dynamic light scattering (DLS) on a Malvern Zetasizer Nano-ZS instrument. This method involved measuring particle size by analyzing the scattering of light collected at a 175° angle to generate an autocorrelation function. Measurements were taken in disposable sizing cuvettes at a constant temperature of 25 °C, maintained within a narrow range of 0.1 °C. The software (DTS v5.03) analyzed the data according to ISO 13321:1996 standards, providing mean size values through cumulant analysis and size distribution based on intensity, volume, and number. Once this optimal size is attained, the milling and analysis procedures are promptly concluded. Subsequently, this milling NaPOFA, now at its smallest particle size, is employed as a replacement for cement to investigate its impact on the hardened concrete properties.

XRF and XRD analysis

Subsequently, a comprehensive chemical assessment of the NaPOFA powder was conducted through X-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses. These analytical techniques were employed to gain insight into the inherent properties of NaPOFA. The chemical composition of both MicPOFA and NaPOFA underwent scrutiny through an X-ray fluorescence instrument (S1 TITAN Handheld XRF Analyzer), providing valuable data regarding their elemental composition. Furthermore, the chemical properties specific to NaPOFA were meticulously analyzed via X-ray diffraction (XRD) instrument (RIGAKU ULTIMA, IV) was used to identify the major phases in the cementitious materials, shedding light on its crystalline structure and composition. Collectively, these analytical methods contribute to a comprehensive understanding of the properties and characteristics of NaPOFA.

Flow table test

To assess the fresh properties of the SCHPC mix, the Slump Flow and T50 tests were employed, adhering to the guidelines set forth by ASTM C143 as a benchmark. The testing apparatus included a flow slump cone, a sturdy base plate, a precision stopwatch, and a scale for accurate measurements. To initiate the test, the center of a 500 mm diameter circle was meticulously aligned with the midpoint of the plate. Subsequently, the freshly mixed concrete was carefully poured into the slump cone. The T50 measurement was obtained as the slump cone was gradually raised until the concrete naturally flowed to reach a diameter of 500 mm. At this point, the largest diameter of the concrete spread was determined, with precision being ensured through an average calculation based on three separate readings.

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Splitting tensile test

Six (6) different mixes with utilization percentages of NaPOFA as a partial replacement for cement, ranging from 1% to 10%, were subjected to a comprehensive assessment of their mechanical properties. The splitting tensile test was performed with an axial load with a specified loading rate of 0.02 mm/s. The casting procedure involved the production of three identical cylindrical samples, each measuring 100 x 200 mm in size, tested at intervals of 1, 3, 7, 28, and 365 days, enabling the acquisition of a comprehensive dataset that accounted for the concrete's evolving strength characteristics over time. The results of these tests were based on the average values derived from the three cylindrical samples. As a reference, the compression strength test was conducted following BS EN 12390-6, 2000.

Design an experiment by using RSM

This study used Design-Expert software (DE) version 11 and Response Surface Methodology (RSM) to determine the optimum mix design for high-strength concrete incorporating Nano-POFA (NaPOFA). The historical data design (HDD) under the RSM was conducted to evaluate the previously obtained experimental data. The percentage of NaPOFA replacement to cement and ages was chosen as the input variable, and splitting tensile strength was chosen as the predicted response.

RESULTS AND DISCUSSION

The development of particle size of NaPOFA

The study presents data on the particle size of the subsequent production of nano-size POFA (NaPOFA) through milling as depicted in Fig 1. Initially, sieve POFA had a size of 1484 nm before milling, and after 12 hours of milling, it reached its smallest size of 255 nm. However, over 36 hours of milling, the particle size increased to 1106 nm due to agglomeration, a phenomenon attributed to van der Waals forces. This led to the cessation of milling at 120 hours, with the smallest recorded size being analyzed. Based on the criterion that nanoparticles in nano concrete should be less than 500 nm in size, NaPOFA with a size of 255 nm was selected for the concrete mix.

The significance of particle size is emphasized in the study, as it profoundly influences the physical and mechanical properties of concrete. Due to their ultra-filler effect, finer POFA particles containing reactive silica (SiO_2) contribute to higher concrete strength and durability. Previous research, such as studies by Birgisson et al. (2012) and Rajak et al. (2015), supports the idea that smaller particles enhance cement paste properties by reducing voids and filling pore systems, ultimately improving mechanical properties. The study's findings underscore the importance of reducing particle size to enhance concrete properties, paving the way for further investigations using NaPOFA as partial cement replacements to explore their properties and potential applications in concrete development.

Chemical analysis of NaPOFA

This investigation analyzed cementitious materials using XRF in the limestone-limestone mode. This analytical technique was employed to precisely identify the individual elements comprising the chemical composition of various raw materials. The resulting chemical compositions of cement and NaPOFA have been detailed in Table 2, shedding light on their elemental constituents. In accordance with the standards set by ASTMC618-92a, palm oil fuel ash (POFA) is classified as falling within the range of Classes C and F, as elaborated by Ali et al. (2015).

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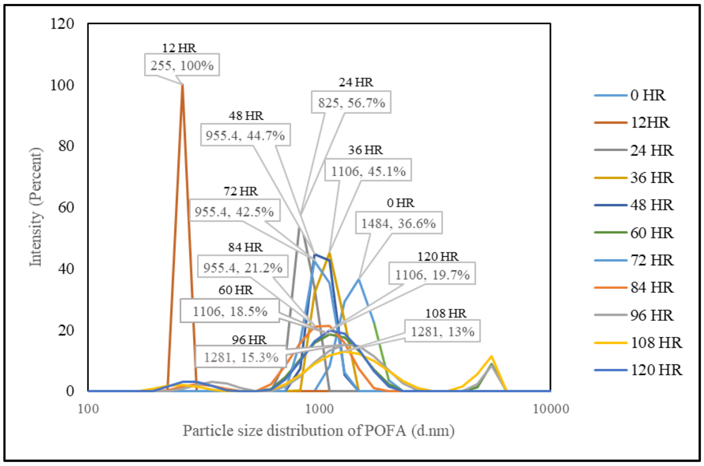

Fig. 1. Particle distribution of POFA over 120 hours.

Table 2. Chemical composition of materials

Chemical composition	Mass percentage %	
	OPC	NaPOFA
Silicon Oxide (SiO_2)	16.13	33.16
Aluminum Oxide (Al_20_3)	2.17	2.66
Ferric Oxide (Fe_2O_3)	2.50	2.32
Calcium Carbonate ($CaCO_3$)	71.78	38.44
Sulfur Trioxide (SO_3)	2.87	1.79
Titanium dioxide (TiO_2)	0.13	0.18
Potassium Oxide (K_2O)	0.31	7.29
Remaining constituent	4.11	14.14
Total compound	100	100
Calcium Oxide (CaO)	60.15	39.71

Table 2 reveals that NaPOFA primarily consists of silica, constituting an impressive 33.16%, surpassing the SiO_2 content in cement, which stands at 16.13%. Silica plays a pivotal role in the hydration process, forming calcium silicate hydrate (C-S-H) when it combines with calcium hydroxide (CaOH) during pozzolanic reactions. This results in stronger and less porous contact zones between aggregates and the cement paste matrix, as substantiated by Altwair et al. (2012). Calcium carbonate ($CaCO_3$) ranks as the second most abundant element in cement and NaPOFA, with cement leading at 71.78%, followed by NaPOFA at 38.44%. $CaCO_3$ in concrete offers significant advantages, including early strength enhancement, accelerated reactions, and reduced porosity, as highlighted by Ali et al. (2015).

Moreover, NaPOFA exhibits higher Al_2O_3 content (2.66%) than cement (2.17%). Al_2O_3 plays a critical role in setting time and strength, and the SiO_2/Al_2O_3 ratios further influence concrete performance. NaPOFA boasts a SiO_2/Al_2O_3 ratio of 12.47, surpassing cement's ratio of 7.43. This higher SiO_2/Al_2O_3 ratio contributes to greater strength, as Al_2O_3 exhibits higher solubility than SiO_2 (Faradilla et al., 2020).

Thus, the chemical composition of NaPOFA closely resembles that of cement, classifying it as Class C fly ash, as defined by materials with CaO contents above 10% and silica and alumina contents below 50%. In conclusion, NaPOFA's chemical makeup aligns with cement properties, making it a suitable partial replacement for cement in this study. Additionally, other physical attributes of POFA, such as fineness and shape, can enhance concrete properties when used as a partial substitute.

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Fig 2 provides an insight into the mineral composition of NaPOFA, which was derived from the XRD analysis. The graph's trend in Fig 3 reveals that NaPOFA primarily comprises three constituents: Quartz (Q) (SiO_2), Mullite (M) ($3Al_2O_3.2SiO_2$), and Grossular (G) with OH-bearing properties ($Ca_3Al_2(SiO_4)_2(OH)_4$). These mineral components were identified using reference codes 00-033-1161, 00-006-0259, and 00-031-0250, respectively.

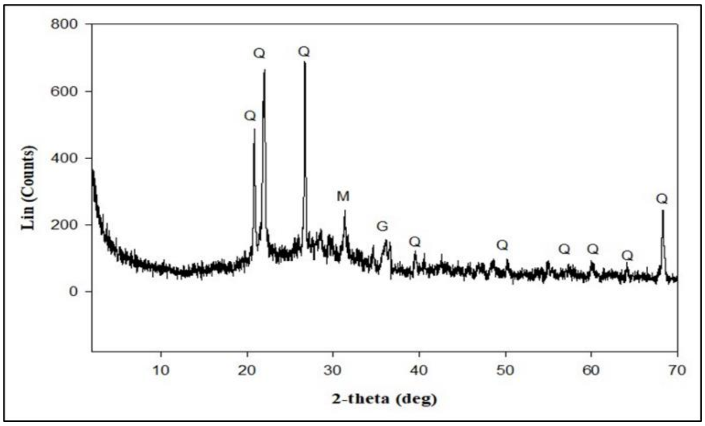

Fig. 2. XRD patterns for NaPOFA.

The XRD diffractograms for NaPOFA exhibit strikingly similar diffractive halo structures, spanning from 20° to 30° (2-theta). This region signifies a low crystallinity zone, often associated with the presence of a glassy phase within the material. Interestingly, Bandukwala & Sonkusare (2016) noted that the presence of a peak between 15º and 40º in the XRD pattern indicates the presence of non-crystalline material to a certain extent. It's worth mentioning that this XRD pattern for NaPOFA aligns closely with patterns observed in POFA samples studied in previous research by Kroehong et al. (2011), Mulizar et al. (2020), and Zeyad et al. (2017). Upon closer examination, the XRD analysis identified major crystalline phases in NaPOFA, with SiO_2 dominating and minor peaks representing mullite and grossular.

Anticipations are high for NaPOFA to exhibit superior compressive strength results, primarily attributed to its stable silica content. This enhanced strength potential is underscored by the presence of Mullite ($3Al_2O_3.2SiO_2$) and Grossular with OH-bearing characteristics ($Ca_3Al_2(SiO_4)_2(OH)_4$) within its mineral composition, notable for their alumina and silica content. Supporting evidence for this assertion emerges from the XRF analysis data presented in Table 2, revealing an increment of alumina and silica content compared to cement. This phenomenon aligns with the XRD findings, which depict that the highest peak for NaPOFA was Quartz. In essence, XRD analyses validate the material properties of NaPOFA, facilitating a cohesive comprehension of their compositional alterations and prospective performance attributes.

Effect of slump flow T50 cm over different percentages of NaPOFA as partial replacement to cement replacement

The assessment of fresh properties in self-consolidating high-performance concrete (SCHPC) involved comprehensively examining various mixture batches to ascertain their workability and flowability characteristics. Six mixture batches were labelled SCHPC, SCHPCNP1, SCHPCNP3, SCHPCNP5, SCHPCNP7, and SCHPCNP10. These formulations were subjected to slump flow and T50 cm slump flow tests to evaluate their performance.

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As illustrated in Fig 3, the slump flow results for all mixtures ranged from 550 mm to 850 mm, indicating a consistent level of workability and flowability across the formulations. This range aligns well with the criteria established by EFNARC 2002 for self-consolidating concrete. It suggests that all formulations possessed the commendable filling ability, crucial for achieving proper consolidation without mechanical vibration.

Upon closer examination, SCHPCNP1 and SCHPCNP3 emerged as particularly notable formulations due to their remarkable stability during testing. SCHPCNP1 exhibited a flow time of 4.8 seconds, while SCHPCNP3 achieved a slightly faster flow time of 4.4 seconds. These results indicate that these mixtures met the workability requirements and demonstrated excellent stability, essential for preventing segregation and maintaining the uniform distribution of aggregates and other constituents within the concrete matrix.

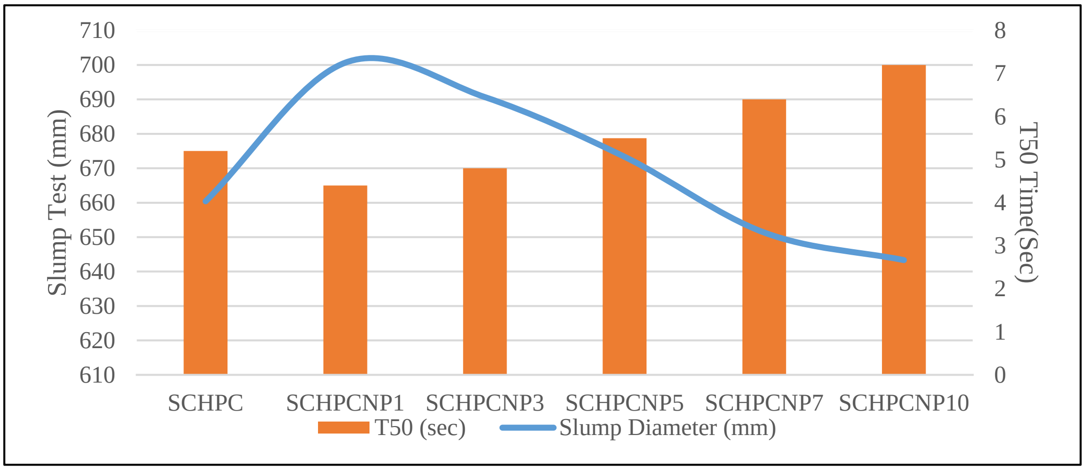

Fig. 3. Relation between slump flow T50 cm of six (6) series of concrete mixes with different percentages of NaPOFA as partial replacement to cement replacement.

Although all mixtures displayed promising filling abilities, SCHPCNP1 demonstrated exceptional filling ability and viscosity performance. This was evident from the observed slump flow characteristics, with SCHPCNP1 displaying the highest slump flow among all formulations. Fig 3 visually represents this trend, showing a peak in slump flow with SCHPCNP1 followed by a gradual decrease through SCHPCNP10. SCHPCNP1 exhibited a notable 18.18% increase in flowability compared to the base SCHPC mixture, with the fastest T50 flow time recorded at 4.4 seconds.

Further analysis revealed that a maximum of 1% NaPOFA replacement was optimal for maintaining excellent filling ability in SCHPC. This finding underscores the importance of the percentage replacement of supplementary materials to achieve desired fresh properties without compromising the overall performance of the concrete. Additionally, SCHPCNP1 emerged as the standout performer in fresh properties, showcasing a 0.34% increase in slump flow and a significant 6.38% improvement in T50 time compared to the base SCHPC formulation.

The evaluation of fresh properties in SCHPC highlighted the importance of meticulous testing and formulation optimization to achieve desired workability, flowability, and stability. The findings underscored the potential benefits of incorporating NaPOFA while identifying SCHPCNP1 as a promising formulation for further investigation and application in practical construction scenarios.

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Splitting tensile test of concrete mixes with different percentages of NaPOFA as partial replacement

Table 3 provides valuable insights into the relative splitting tensile strength of SCHPC mixes incorporating NaPOFA. The data reveal a consistent and progressive increase in relative strength for SCHPCNP1, spanning from day one to day 365, with relative strengths recorded at 122.3%, 117.3%, 116.1%, 122.5%, 114.6%, and 114.6%, respectively, in comparison to SCHPC. This trend underscores the positive influence of NaPOFA on the splitting tensile strength of the concrete mixture. Table 3 shows that the nano-sized POFA achieves a remarkable 5% partial replacement compared to the control SCHPC. However, a decline in relative splitting tensile strength is evident when the partial replacement of NaPOFA escalates to 7% and 10%, with SCHPCNP10 (10% partial replacement) recording the lowest relative splitting tensile strength at 88.9%.

Table 3. Relative splitting tensile strength for SCHPC specimens containing NaPOFA

Days Mixes	1	3	7	14	28	365
	Relative splitting tensile strength (%)					
SCHPC	100.0	100.0	100.0	100.0	100.0	100.0
SCHPCNP1	122.3	117.3	116.1	122.5	114.6	115.6
SCHPCNP3	113.2	109.8	126.2	117.1	104.7	105.3
SCHPCNP5	111.2	105.9	104.9	102.3	100.0	101.6
SCHPCNP7	120.4	111.4	111.6	102.7	92.7	99.0
SCHPCNP10	90.9	93.9	109.3	101.5	88.9	89.5

In the case of splitting tensile strength, NaPOFA-based concrete demonstrates an enhancement in its strength properties compared to SCHPC. Notably, 1% partial replacement yields the highest strength, while 10% partial replacement results in the lowest performance in hardened properties. It's worth highlighting that the relative splitting tensile strength outpaces the relative compressive strength by a significant margin, with a 12.12% increase observed. This suggests that the incorporation of nano-sized POFA as a partial cement substitute has a more pronounced positive impact on tensile strength in concrete structures compared to control SCHPC.

The observed trends in the splitting tensile strength of NaPOFA-based concrete compared to SCHPC can be attributed to several factors related to the characteristics and behavior of NaPOFA in the concrete mixture. Firstly, NaPOFA exhibits pozzolanic activity, engaging in a chemical reaction with calcium hydroxide in the presence of water to form additional calcium silicate hydrate gel. This reaction enhances the densification of the concrete matrix and contributes to improved mechanical properties, including tensile strength. Secondly, when finely ground to nano-sized particles, NaPOFA acts as a filler material, filling voids between larger aggregate particles and resulting in a more compact and homogenous concrete structure. This improved packing density enhances resistance to cracking and improves tensile strength. Additionally, the nano-sized particles of NaPOFA have a higher surface area, allowing for better interaction with cement hydration products and leading to more efficient pozzolanic reactions, further enhancing tensile strength. However, at higher replacement levels of NaPOFA, such as 7% and 10%, there may be a decline in splitting tensile strength due to excessive substitution of cement by the pozzolanic material, limiting available cementitious material for hydration and bonding. Overall, the positive influence of NaPOFA on splitting tensile strength is attributed to its pozzolanic activity, filler effect, particle size and distribution, and reduction of porosity within the concrete matrix, emphasizing the importance of determining an optimum replacement level to maximize benefits on concrete properties.

Optimization of NaPOFA as partial replacement to cement through splitting tensile test by RSM

The optimization of the mixed proportions in SCHPC incorporating NaPOFA was achieved through the software's optimization tool, which offers various optimization options, such as maximizing,

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minimizing, or reaching specific target values through the splitting tensile strength test. Within the framework of Response Surface Methodology (RSM), an Analysis of Variance (ANOVA) was employed to investigate the connection between the input variables (% of NaPOFA as a substitute for cement and aging time) and the output variable (splitting tensile strength). The summarized results are presented in Table 4, which demonstrates that the model exhibits a significant F-value of 26.92 and an exceedingly small p-value of 0.0001. These findings underscore a substantial relationship between the proportion of NaPOFA as a partial cement replacement and the age of the concrete, impacting its splitting tensile strength. Furthermore, the p-value of the model is less than 0.05, confirming the rejection of the null hypothesis concerning the two input factors. Based on the ANOVA results, a quartic model equation was derived to predict the splitting tensile strength of NaPOFA concrete on the 365th day, as expressed in Equation (1). It's important to note that the levels of each factor should be specified in their original units when using this equation for predictions.

$$\begin{aligned} Splitting\ Tensile\ Strength \\ = {} & 3.08087 + 0.132606 * Ages + 1.15456 * NaPOFA \\ & + -0.00264427 * Ages * NaPOFA + -0.00674871 * Ages^2 \\ & + -0.497607 * NaPOFA^2 + -0.000181191 * Ages^2 * NaPOFA \\ & + 0.000582812 * Ages * NaPOFA^2 + 0.000165617 * Ages^3 \\ & + 0.0734038 * NaPOFA^3 + -1.51331e-06 * Ages2 * NaPOFA^2 \\ & + 5.13629e-07 * Ages^3 * NaPOFA + -8.0096e-07 * Ages \\ & * NaPOFA^3 + -4.05713e-07 * Ages^4 + -0.00352772 \\ & * NaPOFA^4 \end{aligned} \tag{1}$$

Table 4. ANOVA compressive strength of SCHPC with the utilization of NaPOFA

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	13.04	14	0.9317	26.92	< 0.0001	Significant
A-Ages	0.0620	1	0.0620	1.79	0.1952	
B-NaPOFA	0.0742	1	0.0742	2.15	0.1578	
AB	0.0613	1	0.0613	1.77	0.1974	
A^2	0.0847	1	0.0847	2.45	0.1327	
B^2	0.4510	1	0.4510	13.03	0.0016	
A^2B	0.0697	1	0.0697	2.01	0.1705	
AB^2	0.0168	1	0.0168	0.4864	0.4932	
A^3	0.0619	1	0.0619	1.79	0.1955	
B^3	0.0832	1	0.0832	2.41	0.1359	
A^2B^2	0.0810	1	0.0810	2.34	0.1409	
A^3B	0.0603	1	0.0603	1.74	0.2012	
AB^3	0.0002	1	0.0002	0.0067	0.9355	
A^4	0.0650	1	0.0650	1.88	0.1850	
B^4	1.06	1	1.06	30.62	< 0.0001	
Residual	0.7267	21	0.0346			
Cor total	13.77	35				

The comprehensive evaluation of model fit, as illustrated in Table 5, offers valuable insights into its predictive performance. The predicted R^2 coefficient, a cornerstone metric gauging the extent of variance explained by the model, yields a robust value of 0.9472. This signifies a high degree of predictive accuracy, with approximately 94.72% of the variance in the dependent variable being effectively captured by the model's independent variables. Moreover, the Adjusted R^2 value of 0.9120 serves as a crucial refinement, integrating considerations of model complexity and sample size into the assessment. The marginal

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discrepancy of less than 0.2 between the predicted R^2 and the Adjusted R^2 underscores the model's resilience in preserving its explanatory power across diverse analytical contexts. Adeq. Precision measures the signal-to-noise ratio. The ratio in this study is 19.8263, indicating an adequate signal since a ratio greater than four is desirable.

Table 5. Fit Statistic

Std. Dev.	0.1860
Mean	4.25
C.V. %	4.38
R^2	0.9472
Adjusted R^2	0.9120
Predicted R^2	0.8465
Adeq. Precision	19.8263

To analyze the intricate relationship between the NaPOFA replacement percentage, aging time, and compressive strength of POFA, we employed a 3D representation of the response surface using Design-Expert 11.0.5 software, as demonstrated in Fig 4. This graphical representation distinctly highlights the substantial impact of substituting cement with NaPOFA on the splitting tensile strength of POFA concrete as it evolves over time. Interestingly, the 3D data diverges from the typical linear trend in concrete splitting tensile strength data over time. Instead, it suggests a cubic model, as evidenced by the R^2 value approaching 1. This departure from linearity is likely influenced by the unique properties of POFA, as discussed in previous sections. Specifically, the agglomeration of NaPOFA particles during milling and the subsequent increase in water absorption with higher replacement percentages contribute to a decline in strength.

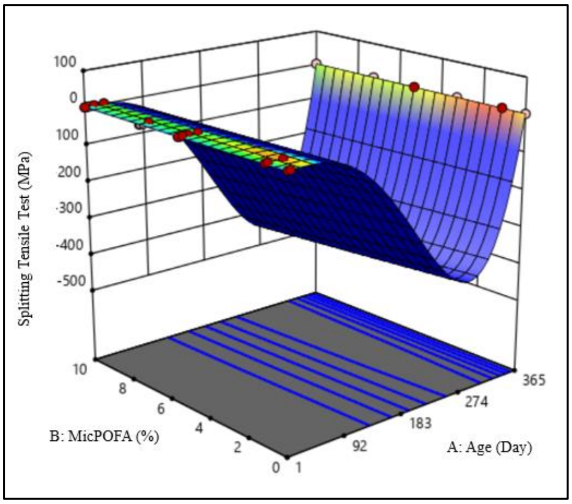

Fig. 4. 3D representation of the response surface of the splitting tensile strength.

Based on the results discussed above, these residual analysis tools must verify that the model's predictions and observations are accurate and appropriately explained. Fig 5 depicts the studentized normal plot for this study in which the normal probability plots predict the residuals following a normal distribution. The straight line in the studentized residual plot indicates that the distribution error is normal. Even though some scattering is expected even with normal data, most data approach a straight line. Thus, the data can normally be distributed in the responses of the model.

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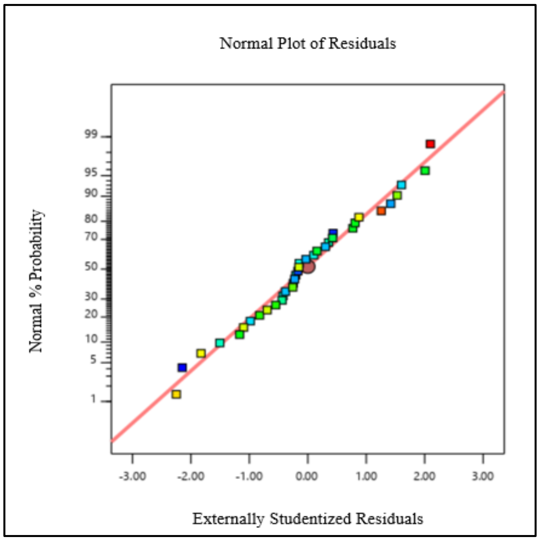

Fig. 5. Normal probability plot of the studentized normal plot for splitting tensile strength.

The analysis aimed to assess the goodness of fit between predicted and actual values, gauging how well the data aligns with the model. Fig 6 depicts the alignment between the experimental and predicted values of the splitting tensile strength for SCHPC, both with and without incorporating NaPOFA. Points that closely approach the straight line demonstrate accurate predictions. Notably, among the 36 runs conducted, none exceeded a 10% percentage difference, affirming that 100% of the experimental data aligns with the predicted values. Consequently, the prediction model stands as a reliable tool for anticipating the splitting tensile strength of SCHPC incorporating NaPOFA.

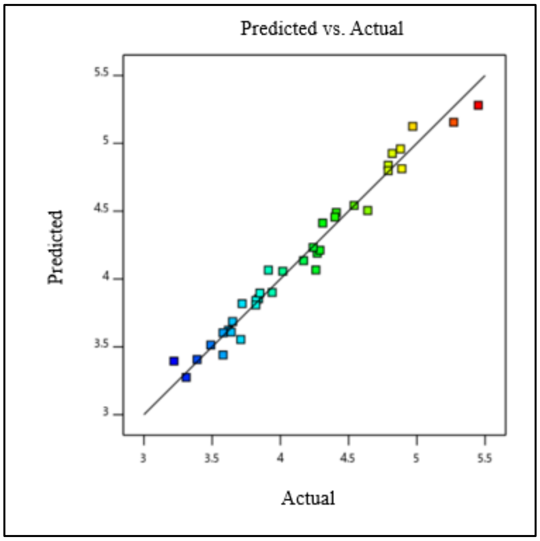

Fig. 6. Predicted vs. actual value of SCHPC splitting tensile strength.

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The proportions of SCHPC incorporating NaPOFA were optimized using the software's tool for optimization, which offers various options such as maximizing, minimizing, or achieving specific targets. The results indicate that, according to RSM predictions, the maximum compressive strength of POFA concrete at day 28 is 5.46 MPa, and this is achieved with a recommended NaPOFA replacement percentage of 1.38%, which approaches 1%. This finding is illustrated in Fig 7.

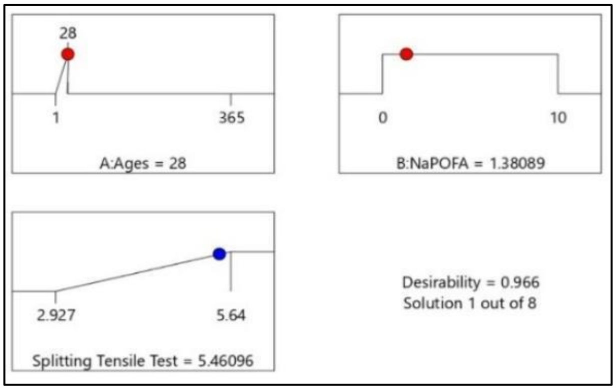

Fig. 7. Optimum percentage of NaPOFA as partial replacement to cement for maximum strength at maturity day (28 days).

Furthermore, the predictions from RSM suggest that the maximum compressive strength of POFA concrete at day 365, set to its maximum value, is 5.64 MPa, with a suggested NaPOFA replacement percentage of 1.48%, which also approaches 1% as in Fig 8. In this study, we investigated the mixed proportions of NaPOFA in SCHPC, aiming for the highest strength, and found that SCHPCNP1 consistently leads to splitting tensile strength results for up to 365 days.

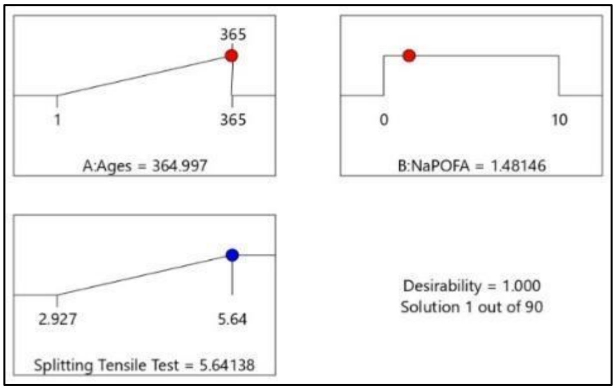

Fig. 8. Optimum percentage of NaPOFA as partial replacement to cement for maximum strength at maturity day (365 days).

CONCLUSION

The key findings and conclusions drawn on the experimental investigation of producing SCHPC by incorporating NaPOFA are as follows:

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(i) The milling process of NaPOFA revealed a 12-hour duration milling showing the smallest particle size, measuring 255 nm. The particle size fluctuated during milling due to the agglomeration process.

(ii) NaPOFA, as a cement replacement in concrete, improved fresh and hardened properties. Notably, there is an inverse relationship between the percentage of NaPOFA used as cement replacement and parameters like slump flow and splitting tensile strength.

(iii) Based on experimental findings and a mathematical model supported by Response Surface Methodology (RSM), SCHPCNP1 emerged as the optimal design mix, delivering the highest performance.

(iv) Fine NaPOFA particles, particularly when replacing 1% of cement, decreased voids in the concrete structure.

In conclusion, NaPOFA shows promise for improving concrete properties, with SCHPCNP1 offering superior performance. This innovation holds potential for sustainable construction, including high-rise buildings, bridges, and infrastructure projects, as well as repairs of existing structures.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial, or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS’ CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: **study conception and design**: Muhd Norhasri Muhd Sidek, Nuradila Izzaty Halim; **data collection**: Nuradila Izzaty Halim, Aidan Newman; **analysis and interpretation of results**: Nuradila Izzaty Halim, Muhd Afiq Muhd Fauzi; **draft manuscript preparation**: Nuradila Izzaty Halim, Muhd Norhasri Muhd Sidek, Aidan Newman, Hamidah Mohd Saman, Nurul Huda Suliman, Mohd Afiq Mohd Fauzi. All authors reviewed the results and approved the final version of the manuscript.

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