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Enhancing the Physico-Mechanical Properties of Concrete via Hydrochloric Acid Treatment on Palm Oil Fuel Ash Additives

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Abstract

Concrete faces significant sustainability challenges due to its reliance on ordinary Portland cement (OPC), which contributes to high CO_2 emissions and environmental degradation. Drying and heat contraction can cause conventional concrete to shrink and crack early in its life. This study explores the use of chemically treated palm oil fuel ash (POFA) as a sustainable alternative to OPC, presenting a novel approach to enhancing the physico-mechanical properties of concrete while addressing environmental concerns. The POFA underwent grinding, sieving, heat treatment at 1000°C, and acid treatment with 2M HCl before being incorporated into concrete samples at replacement levels of 0%, 10%, 20%, and 30%. After curing for seven and twenty-eight days, the samples were evaluated for density, porosity, compressive strength, and water absorption. The sample with 20% POFA replacement demonstrated the most significant improvements, achieving an optimal density of 2.172 g/cm^3, the lowest water absorption (0.84%), smallest porosity (1.78%), and optimal compressive strength (1097.00 MPa). These enhancements are attributed to improved pozzolanic reactions, which densified the concrete matrix by reducing voids. While the study highlights the potential of POFA in sustainable construction, limitations such as the availability and processing cost of POFA warrant further investigation. Future research should explore the long-term durability of POFA concrete and its performance under varying environmental conditions, paving the way for its broader application in eco-friendly building practices.

1. Introduction

Concrete, one of the most widely used construction materials globally, is essential for modern infrastructure. However, the production of ordinary Portland cement (OPC), a primary component of concrete, is associated with significant carbon emissions and environmental degradation [1]. These challenges have prompted research into sustainable supplementary cementitious materials (SCMs) to enhance concrete properties while reducing its environmental footprint. Palm oil fuel ash (POFA), a by-product of palm oil production, has shown great potential as an SCM due to its high silica content and pozzolanic properties [2]. Concrete containing POFA shown enhanced resistance to acid and sulphate attacks, making it appropriate for hard environmental conditions [4].

POFA is produced from the combustion of oil palm shells and fibers during biomass energy generation in palm oil mills, resulting in ash that contains high amounts of silica, alumina, and ferric oxide [5][6]. Combustion temperatures between 700–1000°C yield approximately 5% POFA from the burned solid waste [7]. Due to its pozzolanic properties, POFA has been shown to enhance concrete characteristics such as reduced porosity, improved workability, and increased resistance to gas, water, and chloride infiltration when treated properly [8][9]. However, untreated POFA often contains impurities and unburned carbon, leading to reduced reactivity and performance issues, such as lower slump values [17]. Studies suggest that chemical treatments, such as acid treatment and heat treatment, can mitigate these issues by enhancing pozzolanic activity and binder compatibility [18].

Previous research has demonstrated the benefits of incorporating POFA into concrete. For example, Ganesan et al. [12] found that replacing OPC with 10–30% POFA improved concrete's physico-mechanical properties, including durability and compressive strength. Additionally, POFA-modified concrete has shown enhanced resistance to acid and sulphate attacks, making it suitable for aggressive environments [4][13]. Kumar et al. [14] and Azeez et al. [15] reported that chemically treated POFA reduces permeability and increases durability against chemical deterioration. However, despite these findings, gaps remain in understanding the optimal treatment processes and replacement levels required to maximize POFA's performance. This includes addressing challenges such as impurities, variability in chemical composition, and the effects of treatment on concrete's hydration process.

This study investigates the potential of chemically treated POFA as a sustainable additive to enhance the physico-mechanical properties of concrete. Chemically treated POFA is produced through grinding, sieving, heat treatment at 1000°C, and acid treatment with 2M HCl. By addressing the limitations of untreated POFA, this research evaluates its effectiveness in concrete mixtures with replacement levels of 0%, 10%, 20%, and 30%. Laboratory tests are conducted to assess key properties such as density, porosity, water absorption, and compressive strength. The findings of Ahmad et al. [17] and other researchers [18][19] highlight the importance of optimizing POFA treatment to achieve superior mechanical performance and structural durability. This research examines the environmental and economic implications of utilizing POFA, particularly in countries like Indonesia, which accounts for 49.39% of global palm oil production [11]. POFA is often discarded in open fields, causing environmental pollution and health risks, such as pulmonary ailments [10]. Transforming this waste into a valuable construction material offers a dual benefit: enhancing concrete performance while reducing the ecological impact of palm oil waste. By addressing the impurities and compositional inconsistencies of untreated POFA through chemical modification, this study aims to establish its potential as a reliable additive for concrete applications.

The research evaluates the performance of chemically treated POFA compared to untreated POFA in concrete mixtures, focusing on improvements in durability, workability, and compressive strength. Laboratory testing identifies the optimal replacement levels to achieve maximum compressive strength while examining CTPOFA’s overall impact on concrete properties. The chemical treatment process enhances POFA's pozzolanic activity, improves compatibility with cement, and optimizes its role in the hydration process. These findings contribute to the effective use of CTPOFA in sustainable construction practices, building on the work of Ganesan et al. [12] and Ahmad et al. [17], and advancing the global push for environmentally friendly building materials [20].

2. Material and Methods

The materials employed in this study were chosen with care to ensure the production of superior concrete with improved qualities. Since ordinary OPC has a track record of dependability and compatibility in construction applications, it was used as the main binder. The best possible particle size distribution and workability were achieved by using fine aggregate that complied with ASTM C33 requirements. The mixing agent of choice was distilled water in order to remove any contaminants that can potentially obstruct the hydration process and chemical reactions. The pozzolanic reactivity and applicability of palm oil fuel ash (POFA), an industrial byproduct obtained from nearby palm oil mills, were improved by careful chemical treatments such as calcination and acid leaching.

An initial phase of treatments was applied to POFA in order to improve its viability as a partial cement substitute. Raw POFA was ground using ball mill machine laboratory into a fine powder that could fit through a 50 µm filter after being dried at 100°C to eliminate moisture. The POFA was then subjected to heat treatment in a Protherm high temperature chamber furnaces PLF series, where it was heated to 1000°C at a regulated rate of 5°C/min, maintained there for two hours, and then cooled at 10°C/min. By using a 2M hydrochloric acid solution in a 1:5 ratio, the POFA was further processed by using magnetic stirrer with hot plate, stirring it for six hours at 25°C and 500 rpm. This chemically treated POFA was filtered, neutralized with distilled water, and dried at 105°C before final grinding. Four mix designs with chemically treated POFA as a cement replacement at 0%, 10%, 20%, and 30% were used to prepare concrete samples. The basic volume for each sample is 6.28 mm^3 of cement. Chemically treated POFA replaces a proportion of the cement by weight in the following ranges: 0%, 10%, 20%, and 30%, depending on the mix design. For example, 11.88 grams of cement and 1.32 grams of chemically treated POFA will make up a 10% chemically treated POFA mix as shown in Table 1. Of the 20 samples that were cast, 10 were given a 7-day curing period and 10 were given a 28-day curing period. A water-to-cement ratio of 0.5 was part of the mix design, and all elements had to be precisely weighed before batching. Before progressively adding water to create a consistent blend, the dry components were manually mixed for three minutes to guarantee even distribution. The prepared concrete mixture was poured into cylindrical molds with a diameter and height of 20 mm. Tamping and vibration techniques were used to remove air spaces and produce proper compaction. In order to preserve ideal hydration conditions, the samples were then cured in distilled water at a regulated temperature range of 15°C to 25°C. The Mettler Toledo Density Kit XS64 was utilized to accurately evaluate the physical properties of the concrete samples, including density, porosity, and water absorption. This precision equipment ensured reliable measurements critical for assessing the material's quality and performance. A Universal Testing Machine (UTM) with a gauge length of 20 mm, a maximum load capacity of 1000 kN to 2000 kN, a pressure applied of approximately 1.159 GPa, and a compression speed of 5 mm/min was used to perform compressive strength testing. The compressive strength was determined using maximum load data, which offered important information about how well CTPOFA performed in improving the mechanical properties of concrete. As stated in table 1, the samples are designated as follows: CWSGP10% (Sample 1) represents a mix of cement (C), water (W), sand (S), gravel (G), and 10% POFA (P); CWSGP20% (Sample 2) contains 20% POFA; and CWSGP30% (Sample 3) includes 30% POFA in the mix.

Table 1 Mixtures design for POFA-CTPOFA samples

Materials per batch	POFA (g)	Cement (g)	Sand (s)	Gravel (g)	Water-cement ratio
Sample 1	1.32	11.88	4.2	8.4	0.5
Sample 2	2.64	10.53	4.2	8.4	0.5
Sample 3	3.96	9.24	4.2	8.4	0.5

3. Results and Discussion

3.1 Heat and Acidic Treatment of POFA

Heat treatment of POFA enhances its pozzolanic properties by increasing the amorphous silica phase, reducing carbon content, and altering the physical and chemical characteristics of the powder before and after the treatment process. By enhancing POFA's reactivity, this procedure needs to increase its potential as a cementitious component in concrete [12]. Fig. 1 represents the powder of POFA which is (a) untreated POFA (UTPOFA), (b) heat treated POFA (HTPOFA) and (c) chemically treated POFA (CTPOFA).

(a) (b) (c)

Fig. 1 The samples of POFA which are (a) untreated POFA (UTPOFA), (b) heat treated POFA (HTPOFA), and (c) chemically treated POFA (CTPOFA)

It clearly shows that these three samples differ from one another in significant ways. The comparison of the three conditions reveals that the powder's colour before treatment is darker than after undergoing three treatments. This variation in colour is linked to the amount of unburned carbon in the POFA. Studies concluded that heat treatment modifies the colour of POFA particles by decreasing their carbon content, which is responsible for their black appearance [21]. POFA underwent heat treatment at 1000°C, causing its colour to shift to an orange-pink hue. This contrasts with the typical transition from dark black to grey, which occurs due to decarbonation as calcium carbonate decomposes at temperatures between 400°C and 600°C [22]. By eliminating impurities and enhancing surface area, acid treatment is anticipated to further purify POFA and improve bonding within the cement matrix. By improving POFA's overall quality and reactivity, this treatment helps the concrete's strength and durability [23].

3.2 Crystallographic analysis of POFA

X-ray diffraction (XRD) was used to perform the specimen's crystallographic examination. The study examined different approaches of POFA replacement, including heat treatment of untreated POFA for two hours at 1000°C. Following a 6 hours treatment with 2M hydrochloric acid (HCl), it was dried for 24 hours at 105°C to eliminate any remaining moisture. The XRD profiles of four samples UTPOFA, HTPOFA, and CTPOFA are shown in Fig. 2.

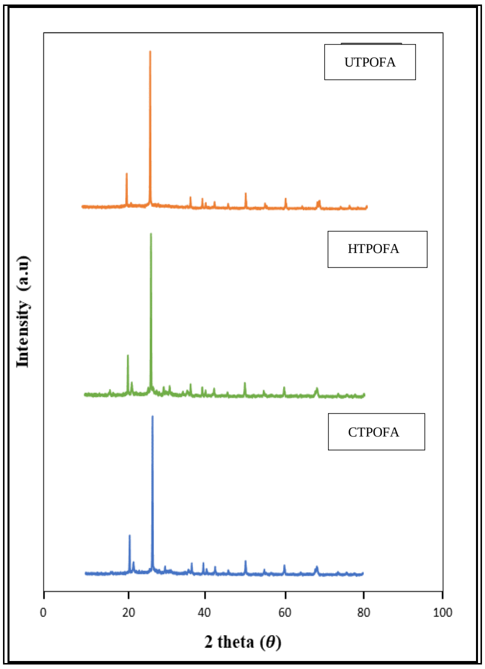
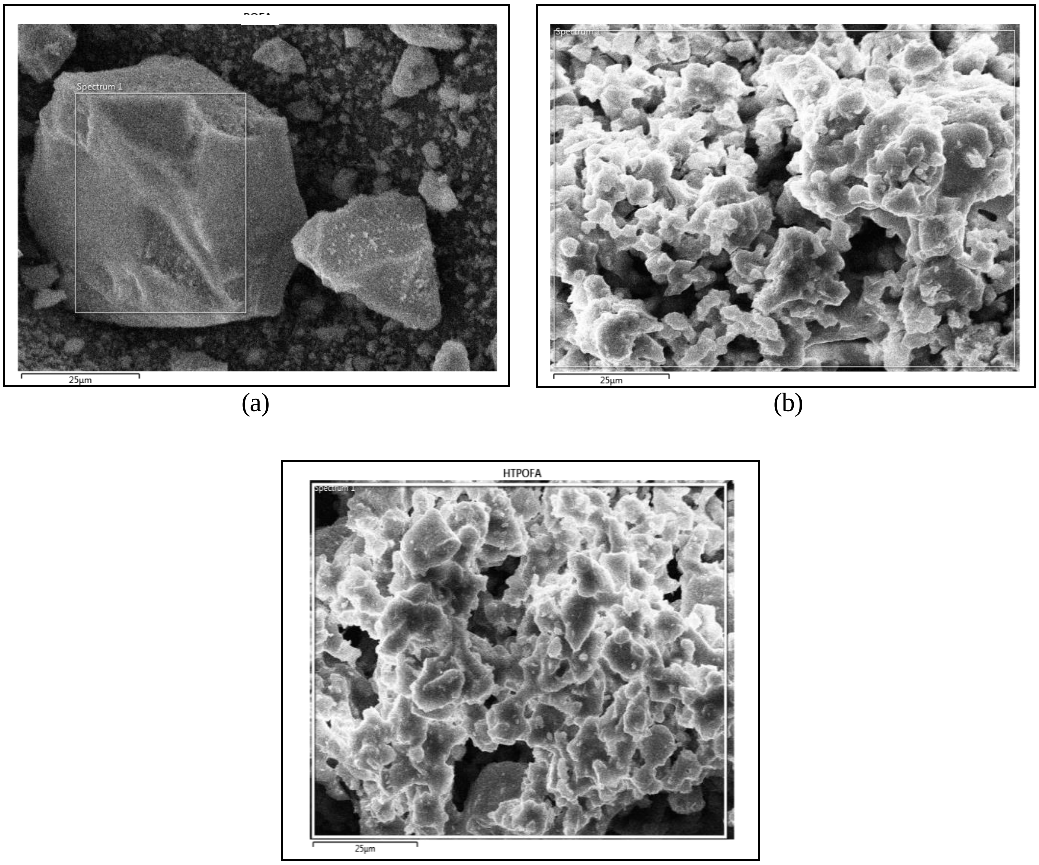


Fig. 2 XRD profile of four samples, namely UTPOFA, HTPOFA and CTPOFA

Significant alterations in the crystalline phases of chemically treated POFA were discovered by X-ray diffraction (XRD) analysis. The efficacy of chemically treated POFA in improving the qualities of concrete was convincingly demonstrated by XRD analysis. Together with a lower crystalline proportion in chemically treated POFA, the patterns verified the existence of amorphous silica, a crucial element promoting pozzolanic activity. As evidenced by the reduced diffraction peaks for quartz and mullite, this reduction highlights the effectiveness of the chemical treatment procedure. Interestingly, once the carbonaceous residues were removed, the heat-treated chemically treated POFA showed sharper peaks and more crystalline phases, indicating a clear change in the structure. Another indication that CH was actively utilized during the pozzolanic process was provided by the reduced calcium hydroxide (CH) peaks in concrete samples that included chemically treated POFA substitution. The production of more calcium silicate hydrate (C-S-H), which is essential for improving the density of the matrix and fine-tuning the microstructure, was made easier by this reaction. These findings establish chemically treated POFA as a game-changing material in sustainable building practices by demonstrating that, at ideal replacement levels, it greatly enhances the microstructural integrity and physico-mechanical performance of concrete.

3.3 Surface Morphology of POFA

The microstructure analysis of the specimen was conducted using SEM. The studies focused on samples sintered at 1000 °C, with varying percentages of POFA replacement, namely 0%, 10%, 20%, and 30%. Fig. 3 illustrates SEM micrographs of three samples: (a) UTPOFA, (b) HTPOFA, and (c) CTPOFA.



(a) (b)

(c)

Fig. 3 SEM micrographs of three samples: (a) UTPOFA, (b) HTPOFA, and (c) CTPOFA

The morphological differences between UTPOFA, HTPOFA, and CTPOFA, each with unique characteristics, are clearly depicted by the SEM examination. Due of unburned organic residues, UTPOFA exhibits angular and irregular particle morphologies with jagged and porous surfaces, which lowers reactivity. The heat treatment method that removes carbonaceous residues from HTPOFA, on the other hand, results in smoother, more compact surfaces with lower porosity, achieving a balance between moderate reactivity and structural integrity. chemically treated POFA, by contrast, is characterized by a highly porous and fractured surface with visible microcracks caused by acid leaching, which significantly enhances its reactivity but compromises its structural strength. These differences underline how unburned residues in UTPOFA hinder its reactivity, while HTPOFA benefits from improved particle compactness, offering a trade-off between strength and reactivity. The acid treatment in CTPOFA introduces microstructural defects, greatly boosting pozzolanic behaviour but limiting its durability. Depending on the desired application, HTPOFA might be favoured for structural reliability, whereas chemically treated POFA could be prioritized for high reactivity demands.

3.4 Physical Properties: Density, Water Absorption & Porosity

This study encompassed the sample's physical properties following testing, including its density, water absorption, and porosity. Fig. 4 illustrates the graph depicting the relationship between density and the percentage of chemically treated POFA for cement + water + sand + gravel + POFA (CWSGP). Sintering temperature and acid treatment significantly enhance the material properties of POFA by improving silica purity and density through the removal of impurities [24][25]. Density measurements were conducted using a Mettler Toledo density kit based on Archimedes' principle, with samples preheated at 105°C to eliminate moisture for accurate results. The study evaluated the density of various POFA compositions (10%, 20%, and 30%) subjected to different heat treatments and curing times, revealing critical insights into their physical properties. Chemically treated POFA incorporation, curing time, and material composition all have a significant impact on the bulk density of concrete-POFA samples, especially when cement, water, sand, and gravel are mixed together. The cement + water + sand + gravel mix, which reflects ideal particle packing and compaction, has the optimal density of 2.172 g/cm^3 at 28 days with 20% CTPOFA replenishment. This is explained by chemically treated POFA's pozzolanic activity, which densifies the microstructure and lowers porosity by reacting with calcium hydroxide to produce more calcium silicate hydrate (C-S-H) [26][27]. Higher chemically treated POFA concentration introduces lightweight particles, which causes density to significantly fall to 2.35 g/cm^3 at 20% replacement. However, this effect is lessened by adding sand and gravel, which increases packing density. Density further decreases to 2.29 g/cm^3 with 30% replacement, suggesting that too much cement replacement has reduced compactness. The steady rise in density from 7 to 28 days for all compositions emphasizes chemically treated POFA's contribution to better long-term matrix densification. According to these results, for concrete mixtures containing aggregates, 20% chemically treated POFA offers the optimal balance between sustainability, compactness, and structural integrity.

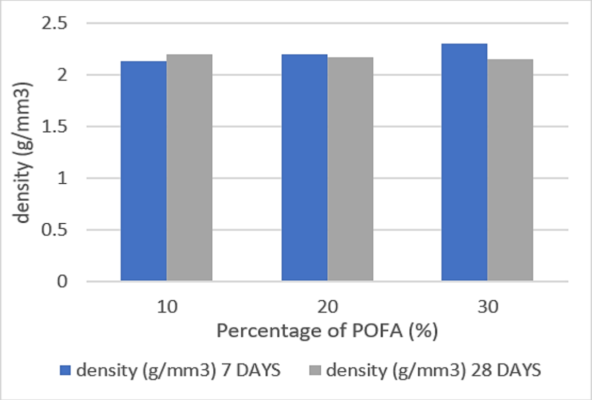


Fig. 4 Density vs the percentage of POFA for cement + water + sand + gravel + POFA (CWSGP)

By strengthening its microstructure through improved pozzolanic reactions that generate more calcium silicate hydrate (C-S-H), chemically treated POFA dramatically lowers the water absorption of concrete. While aggregates like sand and gravel further improve compactness and reduce water infiltration, chemically treated POFA, which fills gaps and reduces porosity, has the lowest water absorption (0.835% after 28 days) at a 20% replacement level. The addition of more porous particles causes a modest increase in water absorption at 10% (1.021%) and 30% (1.165%) replacement; nevertheless, permeability is reduced by the pozzolanic activity and aggregate inclusion as shown in Fig. 5. According to these results, 20% chemically treated POFA is the ideal composition for improving the durability of concrete, lowering moisture intrusion, and encouraging environmentally friendly building practices by utilizing agricultural waste.

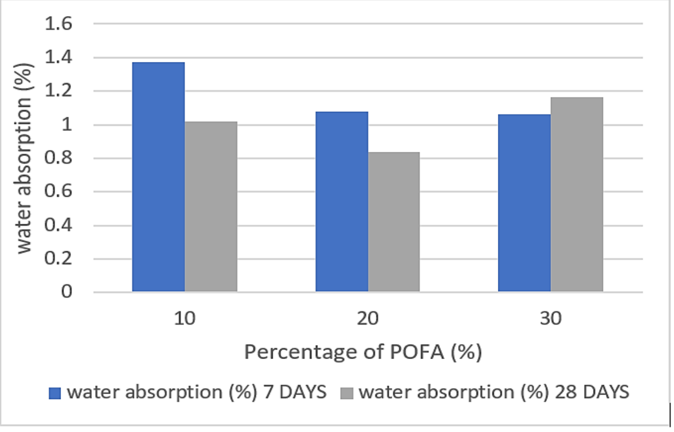


Fig. 5 Water absorption vs the percentage of POFA for cement + water + sand + gravel + POFA (CWSGP)

Fig. 6 depicts the porosity vs the percentage of POFA for cement + water + sand + gravel + POFA (CWSGP). Concrete's porosity is greatly decreased by adding chemically treated POFA, which densifies the microstructure of the concrete through improved pozzolanic reactions that generate more calcium silicate hydrate (C-S-H). The interfacial transition zone between the aggregate and cement paste is strengthened and capillary holes are reduced as a result of this reaction, which enhances durability and impermeability. With the lowest porosity (1.782% after 28 days) at a 20% replacement level, chemically treated POFA efficiently fills gaps and lowers porosity. By increasing particle packing density, aggregates like sand and gravel reduce water infiltration paths and improve compactness. The pozzolanic reaction and the inclusion of aggregates, which support the maintenance of a relatively dense and impermeable structure, counteract the minor increase in porosity at higher replacement levels 2.193% for 10% chemically treated POFA and 2.318% for 30%. According to these results, 20% chemically treated POFA is the ideal replacement amount because it provides the optimum compromise between decreased porosity, increased durability, and sustainability through the use of agricultural waste. This encourages the production of environmentally friendly concrete that performs better over time in harsh conditions and has better resistance to moisture infiltration.

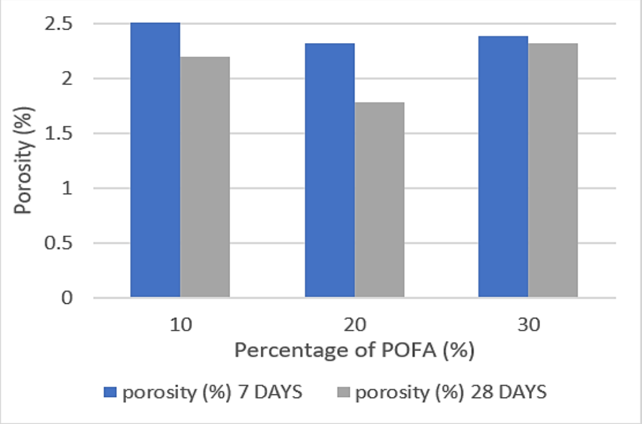


Fig. 6 Porosity vs the percentage of POFA for cement + water + sand + gravel + POFA (CWSGP)

The relationship between density, water absorption, and porosity in concrete is interconnected, as these properties collectively determine the material's compactness, durability, and resistance to external factors. Because a denser concrete matrix contains fewer spaces, which means fewer paths for water to pass through, higher density is generally linked to lower porosity. As seen in samples containing chemically treated POFA, where the pozzolanic reaction produces more calcium silicate hydrate (C-S-H), filling voids and enhancing the microstructure of the concrete, this decrease in porosity directly correlates to decreased water absorption. For example, concrete containing 20% chemically treated POFA had the lowest water absorption (0.835%) and the optimal density (2.172 g/cm^3), suggesting lowest porosity (1.782%) and ideal particle packing. On the other hand, adding lighter chemically treated POFA particles results in a minor reduction in density at higher replacement levels, such 10% and 30%, while increasing porosity and water absorption. However, by making the mix more compact, aggregates like sand and gravel counteract these impacts. This interaction shows that improving concrete's physico-mechanical qualities requires striking the ideal balance between density, porosity, and water absorption; 20% chemically treated POFA is the most efficient in preserving structural integrity and durability while advancing sustainability.

3.5 Mechanical Properties: Compressive Strength

Chemically treated POFA is added to concrete to increase its compressive strength by encouraging pozzolanic processes that produce more calcium silicate hydrate (C-S-H), which creates a denser and more robust matrix [28]. As shown in Fig. 7, with a compressive strength of 1097.00 MPa after 28 days, the cement, water, sand, gravel, and 20% chemically treated POFA mixture outperformed the other samples tested. This was in stark contrast to the control mix without chemically treated POFA, which only registered 633.67 MPa. Sand and gravel improve particle packing in this mix, reducing voids and producing a more compact structure, while chemically treated POFA improves the microstructure and speeds up hydration. While the 30% chemically treated POFA mix shows much lower strength (968.79 MPa), perhaps as a result of increased porosity and excessive cement content reduction, the 20% chemically treated POFA mix gives reasonable strength (1158.69 MPa at 28 days) and may be appropriate for non-structural applications. Accordingly, the 20% chemically treated POFA composition balances sustainability and performance, making it perfect for structural applications needing high strength and durability [27].

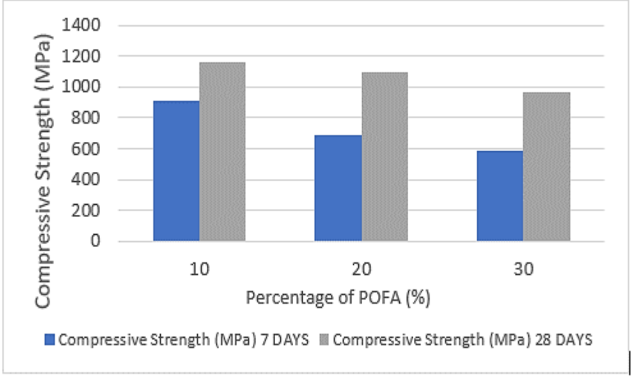


Fig. 7 Compressive strength vs the percentage of POFA for cement + water + sand + gravel + POFA (CWSGP)

3.6 Optimal Composition of Chemically Treated POFA for Enhanced Concrete Performance

Since POFA has improved pozzolanic reactivity and compatibility with the cement matrix, its ideal content of 20% was found to offer the greatest combination of physico-mechanical properties. By efficiently reacting with calcium hydroxide during the hydration process, the silica concentration of chemically treated POFA at this level creates more calcium silicate hydrate (C-S-H) gel, which densifies the concrete matrix. Results from experiments show that this enhanced microstructure results in lower porosity and higher compressive strength. In addition to offering an adequate quantity of reactive material without appreciably reducing the cement content, the 20% replacement guarantees a strong matrix with outstanding mechanical performance.

Conversely, lesser replacement levels, like 10% POFA, might lead to inadequate matrix densification since there wouldn't be enough reactive material to fully utilize the available calcium hydroxide, which would restrict pozzolanic activity. Conversely, larger replacement levels, such 30% POFA, can increase porosity and decrease compressive strength because of an overabundance of POFA particles that might serve as fillers rather than active hydration players. The concrete structure may be weakened by this excess since it may break up the cement matrix and prevent C-S-H gel from forming. Furthermore, voids caused by an excess of unreacted POFA might result in increased porosity and decreased durability.

Concrete that has been altered with chemically treated POFA offers a lot of potential for practical uses, especially in a variety of environmental settings. Structures exposed to harsh environments, including industrial or coastal locations, where resistance to sulphate and chloride attacks is crucial, might benefit from its increased durability and decreased permeability. The enhanced compressive strength and reduced porosity of concrete treated with chemically treated POFA, also make it appropriate for high-performance applications that require long-term structural integrity, like bridges, dams, and high-rise structures. Its reduced ability to absorb water also reduces the possibility of freeze-thaw damage in cold conditions, and its environmentally friendly manufacturing fits well with the objectives of sustainable building in tropical areas where waste from palm oil is plentiful. These characteristics demonstrate chemically treated POFA's adaptability and worth in advancing long-lasting and sustainable infrastructure.

4. Conclusion

This study demonstrated the outstanding potential of chemically treated POFA as a sustainable concrete addition, with the most balanced and optimal results obtained at a replacement level of 20%. After 28 days of curing, concrete containing 20% chemically treated POFA, sand, and gravel had a density of 2.172 g/cm^3, meaning it was 16.8% better than the control mix. When compared to control samples, water absorption dropped by 38.4%, with a minimal rate of 0.835% in the same composition. Similar reductions were seen in porosity, which was significantly lowered by 25.78% from 2.401% in the control to 1.782% in the 20% chemically treated POFA mix. Compressive strength testing indicated that chemically treated POFA enhances mechanical properties, with the 20% chemically treated POFA mix achieving a compressive strength after 28 days of curing, representing a 20% improvement over the control sample. The chemically treated POFA-modified concrete's compressive strength also demonstrated encouraging outcomes, suggesting higher mechanical performance as a result of the pozzolanic reaction and increased matrix density. However, scalability challenges such as sourcing and processing chemically treated POFA in large quantities and the economic implications of its adoption, including initial processing costs, must be addressed to enable widespread implementation. These results support chemically treated POFA's ability to enhance concrete's physico-mechanical characteristics while resolving environmental issues, and future studies should focus on its scalability, economic feasibility, and long-term performance under diverse environmental conditions to validate its role in sustainable construction practices.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Siti Nurain Shukor;* ***solve the governing equation:*** *Siti Nurain Shukor, Mohamad Zaky Noh;* ***data collection:*** *Siti Nurain Shukor;* ***analysis and interpretation of results:*** *Siti Nurain Shukor, Mohamad Zaky Noh;* ***draft manuscript preparation:*** *Siti Nurain Shukor, Mohamad Zaky Noh. All authors reviewed the results and approved the final version of the manuscript.*

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