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REVIEW ARTICLE

A Short Review on the Utilization of Pozzolan Material in Concrete

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ABSTRACT - This paper provides a short review on the agricultural waste and industrial waste as pozzolan material in concrete. The pre-treatment method, characterization process and the engineering properties of a suite of pozzolans used including rice husk ash (RHA), corn cob ash (CCA), sugarcane bagasse ash (SCBA), bamboo leave, oil palm shell (OPS), and others was studied. From the study, the agricultural waste which is Oil Palm Shell is highlighted to be use as pozzolan material in concrete apart for other used. Oil Palm Shell (OPS) ash as one of the agricultural based pozzolan gained less popularity due to its relatively low amorphous silica content after incineration process (< 50% silica). Therefore, an alternative approach was studied form the previous researcher to extract high proportion of amorphous silica from OPS ash that fulfils the minimum requirement of pozzolanic standard.

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1. INTRODUCTION

The cementitious materials of today were among the leading cementitious components of concretes created many centuries ago. These materials are generally called pozzolans and may form a durable binder. In accordance with ASTM C125-11b, the pozzolan is an aluminum and silica material that does not have any cementitious or zero characteristics; however, it comes to contact with humidity and begins chemical reactions with Ca $(OH)_2$ and appears to have cementitious material characteristics. These features render pozzolan perfect additions to mixtures of concrete Portland cement. They are made of similar materials and react with hydrated cement products to create a more cement binder. There are two types of cement replacement material sources: agricultural waste and industrial waste. Figure 1 shows the physical appearance of the cementitious materials from industrial waste like fly ash (Class C), metakaolin, silica fume, fly ash (Class F), slag, and calcined shale can be mixed with blended cement to enhance concrete strength [1].

Figure 1. Type of cementitious materials: From left to right, fly ash (Class C), metakaolin (calcined clay), silica fume, fly ash (Class F), slag, and calcined shale [1]

Class C ashes comprise calcium alumino-sulfate glass, tricalcium aluminate, quartz, and free lime. They are typically produced from sub-bituminous coals (CaO). While Class F ashes are generally made of bituminous and anthracite coals, they also contain quartz, mullite, magnetite, and alumino-silicate glass. When silicon and ferrosilicon alloys are produced in electric furnaces, high-purity quartz is reduced with coal to produce silica fume, a by-product. In addition to being formed by calcining native lateritic soils, metakaolin is typically made by thermally treating kaolin or paper sludge. Next, a considerable amount of steel slag is produced as a by-product when steel is made in a submerged electric arc furnace. Calcined shale comes from a lightweight aggregate producer and a kind of soft, stratified sedimentary rock that originated from the deposition of clay-sized particles in generally calm and murky seas.

1.1 Agricultural Waste

The high demand for natural resources due to rapid urbanization and the problem of disposal of agricultural wastes in developed countries have created agro-waste opportunities in the construction industry. Numerous researches in civil engineering have shown that agricultural wastes can be a great partial replacement for cement when making cement

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concrete [2, 3]. When appropriately processed and incinerated, agricultural wastes partially have enough silica and lime content to replace cement [4]. When adequately treated and designed, materials like rice husk ash, sugarcane bagasse ash, palm oil fuel ash, and bamboo lead ash can outperform cement concrete in terms of workability, durability, and heat of hydration [5]. Some agricultural waste materials are already used in concrete as replacement alternatives for other applications besides cement. Several types of agricultural waste are usually used as replacement material in concrete, like bagasse ash, Groundnut shell, Oyster shell, Giant reed ash, Giant reed fibre, and Tobacco waste [6]. Through the study, Bagasse ash, crushed nut shells, and tobacco waste are replaced for cement, while oyster shells, giant reed fibre, and ash are replaced for fine aggregate in concrete. Several studies also focus on the material like Oil palm shell (OPS), oil-palm-boiler clinker (OPBC), oil palm kernel shell (PKS), rice husk ash (RHA), corncob ash (CA), wheat straw ash (WSA), and olive ash (OA) that are also agricultural by-product [7]. Previous researcher has studied the effect of agricultural waste like rice husk ash (RHA), sugarcane bagasse ash (SCBA), palm oil fuel ash (POFA) and bamboo lead ash (BLA) on the strength of cement concrete [4]. All of this material was selected due to its high silica content [6]. Tables 1 and 2 show the physical properties and chemical composition of the four agricultural wastes and cement above.

Table 2. Physical properties of various agriculture waste [4]

Parameter	Cement	SCBA	RHA	POFA	BLA
Specific gravity	3.14	2.24	2.11	2.15	2.64
Colour	Grey	Black	Greyish black	Black	Grey
Particle size	25 µm	16.4 µm	10 µm	17.10 µm	58.1 µm

Table 2. Chemical composition of cement and various agricultural wastes [4]

Composition (in %)	Cement	SCBA	RHA	POFA	BLA
Lime (CaO)	63.170	6.5	1.39	5.23	5.06
Silica (SiO_2)	19.980	65.1	93.47	66.64	80.40
Alumina (Al_2O_3)	5.170	8.2	0.46	3.82	1.22
Iron Oxide (Fe_2O_3)	3.270	3.2	0.33	3.70	0.71
Magnesia (MgO)	0.790	2.1	0.08	2.29	0.99
Sulphur Trioxide (SO_3)	2.238	2.1	1.33	0.43	1.07
Alkalis (Na_2O, K_2O)	0.800	3.0	1.44	6.86	1.40
Loss on Ignition	2.500	12.1	8.50	2.32	8.04

1.2 Industrial Waste

Researchers have investigated the prospect of using industrial wastes as a component ingredient in concrete. By 2025, solid waste production from industry is predicted to reach up to 27 billion tonnes globally [8]. Waste from residential, commercial, institutional, industrial, and building and demolition projects is included in this estimate. Several industrial wastes are used as full or partial replacement of cement, coarse aggregate or fine aggregate. For example, the cement industry uses pozzolanic ingredients from industrial waste, such as fly ash and silica fume, to improve the performance of concrete and reduce its CO^2 contribution [9]. Several studies also reported that both materials give better particle packing [10], improve strength [11, 12], reduce the heat of hydration [13, 14], and enhance durability [15, 16].

[17] thoroughly assessed industrial waste substances that can be adequately utilized in concrete as a fine aggregate substitution. They reviewed some of these industrial wastes like waste foundry sand, steel slag, copper slag, imperial smelting furnace slag (ISF slag), blast furnace slag, coal bottom ash, ferrochrome slag, palm oil clinker, etc. Out of these materials, a maximum number of experiments have been conducted using waste foundry sand and copper slag as fine aggregate replacements [17]. However, the study suggests more examinations are required for other waste materials as replacement of sand in concrete. The various mechanical and physical characteristics of industrial waste and industrial waste concrete, which replaces natural sand, have been examined and compared in their study.

Table 4. Physical properties of GGBS, FA, RHA and POFA [18]

Components	GGBS	FA
Specific gravity	2.95	2.65
Bulk density (kg/m^3)	1185	1150
Specific surface area (cm^2/gm)	610	2450
Moisture contents (%)	17.8	16.6
Fineness modulus	2.39	1.97

Recent studies by [18] have examined ground granulated blast furnace slag (GGBS) and fly ash (FA) as replacements for cement in lightweight foamed concrete. It has been thought that FA and GGBS, as waste materials and industrial by-products, are suitable materials for making environmentally friendly and sustainable concrete [19, 20]. Tables 4 and 5 show the physical and chemical properties of GGBS and FA, respectively.

Table 5. Chemical compositions of GGBS, FA, RHA and POFA [18]

Components	GGBS	FA
Silicon dioxide (SiO_2)	32.95	53.25
Aluminum Oxide | (Al_2O_3)	12.82	30.12
Ferric Oxide (Fe_2O_3)	2.21	9.98
Calcium oxide (CaO)	40.85	3.83
Magnesium oxide (MgO)	8.57	1.56
Sulfur trioxide (SO_3)	1.83	0.35
Sodium Oxide (Na_2O)	0.29	0.33

2. PRETREATMENT METHOD

2.1 Silica Extraction and Incineration Process

Several previous studies have conducted the silica extraction method and incineration process to increase its pozzolanic reactivity and fulfil the minimum requirement of the pozzolanic standard. [21] intended to study the synthesized nano silica from sugarcane bagasse ash by sol-gel with reflux method and no pretreatment applied. Throughout the study, the incineration process was conducted by burning sugarcane calcined through a heating rate of 300 °C/h, 650 °C/2h, and constant burning temperatures at 500 °C and 650°C. By controlled-burning the sugarcane bagasse ash at 650 °C/2h, the researcher obtained 72 % silica content with some metallic impurities.

[22] carried out the extraction method by cleaning and drying the sugarcane bagasse ash for 24 hours before conducting the pretreatment process with low-concentration HCl at various concentrations of 0.1 M, 0.5 M, and 1.0 M to remove alkali metals from the bagasse. They were then tested at multiple soaking periods of 1, 2, 3, 4, 5, and 6 h. The incineration process for the treated sugarcane bagasse ash was conducted at various temperatures (600, 700, and 800 °C) and burning periods (1, 2, and 3 hours). Sugarcane bagasse ash was tested for its mineralogy and chemical oxide due to this incineration process. The researcher developed highly pozzolanic sugarcane-bagasse ash with the combination of 0.1 M HCl and 1-hour soaking duration through 800 °C incineration temperature for 1 hour burning duration that improves the use of SCBA as cement substitution material through experimental work.

Another researcher [23], conducted the only pretreatment without incineration process to extract silica from Palm Ashvia using the Citric Acid leaching treatment method. The strong acid leaching treatment aims to remove metallic impurities and organics in rice husk with optimum extracting conditions at 700 °C of solution temperature, 60 minutes of reaction time, and a concentration of citric acid of more than 2 %. The purity of silica extracted is more than 90 %.

3. CHARACTERIZATION OF MATERIAL

Material characterization is a process by which material structure and properties are probed and measured. The material's characteristics and performance depend on the preparation method and pretreatment conditions. The mechanical, thermal, chemical and electrical properties of the material also can be tuned for specific applications. Suitable characterization techniques are essential to make a correlation between the properties of the materials and their application. Varieties of techniques are available to characterize materials with specific properties and applications. [24] mentioned that all these techniques aim to determine the crystal structure, defect structure, phase, size, shape, crystal or grain size, composition, etc. The molecular structure, composition, and vibration frequencies of a substance can be better understood in spectroscopy. [25] found that it is also useful to find the concentration of reactants concerning time and find the reaction intermediates. A brief description of the various characterization techniques is discussed below.

3.1 Elemental Leaching

Inductively coupled plasma mass spectrometry (ICP-MS) is an elemental analysis technology capable of detecting most of the periodic table of elements at milligram to nanogram levels per litre. It is used in various industries, including, but not limited to, environmental monitoring, geochemical analysis, metallurgy, pharmaceutical analysis, and clinical research. Most analyses performed on ICP-MS instrumentation are quantitative; however, it also can serve as an excellent semi-quantitative instrument. Using a semi-quantitative software package, an unknown sample can be analyzed for 80 elements in three minutes, providing semi-quantitative data typically within ±30% of the quantitative values.

In a study conducted by [26], the concentration of Eu in coal, fly ash, and sedimentary rocks was measured by quadrupole-based inductively coupled plasma mass spectrometry (ICP-MS). The study used a Thermo Fisher (X-Series II) inductively coupled plasma mass spectrometer (ICP-MS) for Eu and Ba determination in coal and associated rock samples. The study shows that ICP-MS analysis combined with the AG50W-x8 cation exchange resin provides a reliable method for determining Eu concentrations in coal, fly ash, and sedimentary rocks. The AG50W-x8 cation exchange resin can effectively separate Ba from Eu and thus can diminish the interference of Ba on Eu during ICP-MS analysis. [27] conducted a case study to measure the uncertainty of heavy metal analysis in drinking water with inductively coupled plasma-mass spectrometry (ICP-MS). All measurements were carried out according to the EPA 200.8 method using a 7500a ICP-MS apparatus (Agilent, Santa Clara, CA, USA). The ICP-MS was equipped with a Babington nebulizer, a Peltier-cooled spray chamber, a peristaltic pump, a robust interface, omega lenses, two turbo motors, a hyperbolic rod quadrupole, and a dual-mode detector. The study concludes that all measurement uncertainty values were below 50% of the values given in the relevant environmental quality standards.

3.2 Chemical Oxide Composition

XRF (X-ray fluorescence) is a non-destructive analytical technique used to determine the elemental composition of materials. XRF analyzers determine a sample's chemistry by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source. Each element in a sample produces a set of characteristic fluorescent X-rays ("a fingerprint") unique for that specific element, which is why XRF spectroscopy is an excellent technology for qualitative and quantitative analysis of material composition. [28] shows the use of X-ray fluorescence (XRF) analysis in predicting the alkaline hydrothermal conversion of fly ash precipitates into zeolites. In this study, A Phillips 1404 XRF Wavelength Dispersive Spectrometer equipped with an array of six analyzing crystals and fitted with an Rh X-ray tube target was used. The co-disposal precipitates were analyzed by XRF spectrometry for the quantitative determination of SiO_2 and Al_2O_3. From this data, the [SiO_2]/[Al_2O_3] ratio was determined. [28] found that the analysis of the co-disposal precipitates by XRF spectrometry for the quantitative determination of SiO_2 and Al_2O_3 provides an effective technique to determine its appropriateness for hydrothermal zeolite synthesis.

3.3 Mineralogical Properties

X-ray Diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground and homogenized, and the average bulk composition is determined. Spectroscopy and photography are the two main classifications of X-ray diffraction methods. The spectroscopic technique, known as X-ray powder diffractometry, is the most widely used diffraction method. On the other hand, in his book, [29] notes that photographic techniques are not common but are used to determine unknown crystal structures. X-ray powder diffraction is widely used to identify unknown crystalline materials (e.g., minerals inorganic compounds). Choosing unknown solids is critical to geology studies, environmental science, material science, engineering, and biology. Other applications include the characterization of crystalline materials, identifying fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically, determining unit cell dimensions, and measuring sample purity.

In an investigation into Rice Husk Silica (RHS) using X-ray diffraction, [30] found that the micrograph shows the aggregates of particles in RHS. Besides that, the diffractograms in Figure 2 reveal that RHS is amorphous. It was observed that only a broad peak with 2 hours at 22˚, is a characteristic of amorphous silica. This form of silica is suitable for zeolite synthesis because it dissolves easily in a NaOH solution to form sodium silicate. In another study, X-ray diffraction was used to determine the crystallinity of the rice husk fibres (cellulose fibres and nanocrystals) after different treatments [31]. Each material in milled powder was placed on the sample holder and levelled to obtain total and uniform X-ray exposure. The samples were analyzed using an X-ray diffractometer (D8-Advance Bruker A.X.S. GmbH) at room temperature (RT) with a monochromatic CuK radiation source (λ= 0.1539 nm) in the step-scan mode with a two-angle ranging from 10˚to 50˚with a step of 0.04 and scanning time of 5.0 minutes characterize the rice husk. X-ray diffraction (XRD) analysis revealed that the crystallinity increased with successive treatments [31].

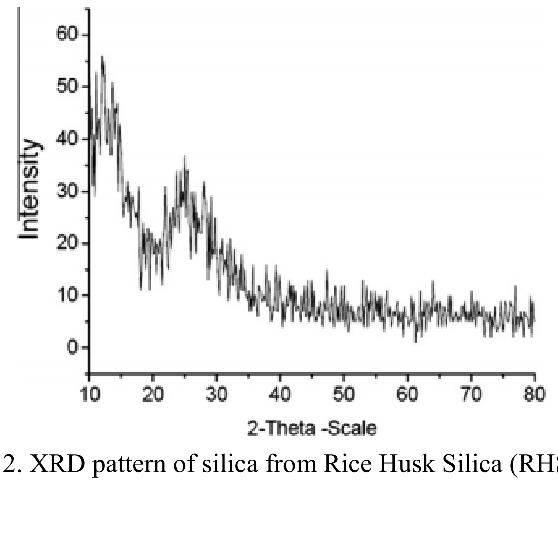

Figure 2. XRD pattern of silica from Rice Husk Silica (RHS) [30]

3.4 Pozzolanic Reactivity

Pozzolans are materials rich in amorphous silica and alumina, natural or artificial origin, used to optimize mortar and concrete durability. They contribute to more sustainable construction products, partially substituting mineral binders in durability, applicability, and energetic consumption. Using pozzolanic materials in lime mortars and concretes is well documented and has been studied intensively. However, some questions are still open: to know its reactivity, e.g., why a certain pozzolan reacts faster than others, what type of materials can be used as pozzolans, and what amounts of pozzolan are needed to achieve optimization. Several methods have been studied to measure the reactivity of pozzolans used in mortars and concrete. They are currently using, measuring, and analyzing different aspects of the lime/pozzolan and cement/pozzolan reaction, such as compression strength, reaction kinetics, and lime consumption [32].

In the previous studies, several methods such as the Chapelle test, saturated lime method, thermogravimetric analysis, and strength activity index were proposed to evaluate the pozzolanic activity of bamboo stem ashes for use as partial replacement of cement [33]. The study found that the Chapelle index results show that Bamboo ash (BA) presents similar pozzolanic activity as Sugar Cane Bagasse ash (SCBA) and other ashes (Table 6). Moreover, in the lime/ashes system, BA fixes less lime than SCBA but has a higher fixed lime (CaO) ratio than Natural Pozzolan (NP). In the cement/ashes system, the combination of NP and BA in a ternary matrix increases fixed lime for up to 90 [33].

Table 6. Chemical composition of materials [33]

Chemical composition (% by mass)	BA	SCBA	NP	OPC
SiO_2	96.74	57.70	43.54	22.84
Fe_2O_3	0.16	3.22	7.96	1.84
Al_2O_3	0.15	6.66	18.49	2.70
CaO	11.90	4.15	12.48	67.41
Na_2O	0.74	0.26	2.05	0.14
K_2O	0.54	13.63	0.39	0.23
MgO	5.83	4.16	4.56	0.81
Loss on ignition	0.42	0.17	0.86	1.72
Chapelle index (mg of CaO/g of material)	345 ± 6	352 ± 8	290 ± 10	-

3.5 Morphological Properties

Scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS or EDX) generates a clear image of the sample's microscopic surface features and comprehensive elemental composition data. The equipment is used in various applications for various material viewing and analysis tasks.

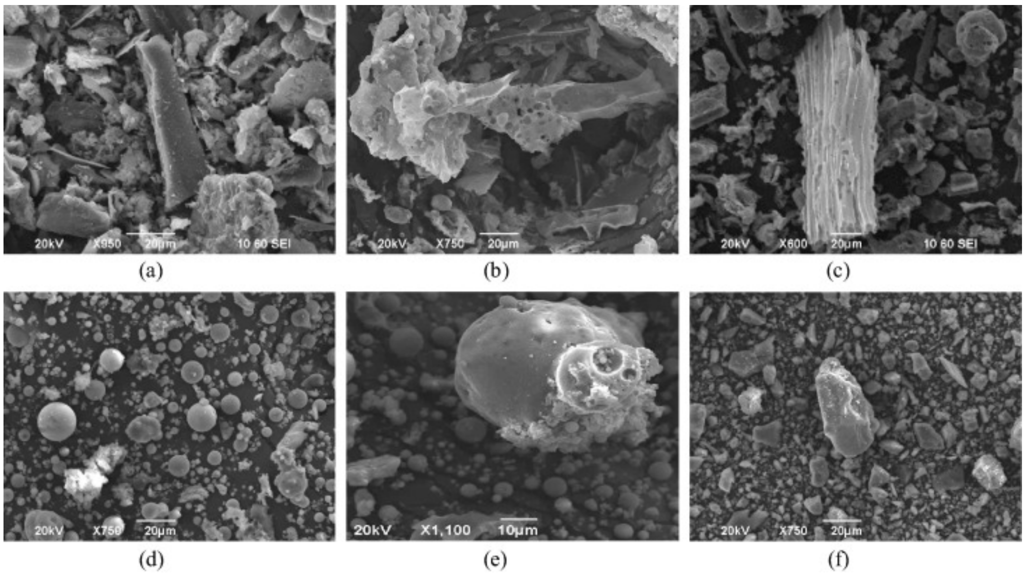

Figure 3. Morphology of the used materials: (a) (b) (c) = UtSCBA, (d) (e) = FA, (f) CPC [34]

In the previous research, they used SEM-EDX to characterize the untreated Mexican sugarcane bagasse ash (UtSCBA) as a supplementary cementitious material in concretes [34]. They aim to study the effects of adding a UtSCBA to binary concrete prepared with blended Portland cement (CPC) and fly ash (FA). Using a high vacuum JEOL® Scanning Electron Microscope (SEM.) Model JSM-6490LV in the secondary electron mode with an attached EDS microanalysis system examined the material particles' morphology and texture. According to the study, UtSCBA comprises heterogeneous particles that vary in size and shape [34]. Three particle forms are identified from the study: solid prismatic (Figure 3 (a)), porous irregular (Figure 3 (b)), and long porous cylindrical (Figure 3 (c)). An agglomeration of fine particles, which typically contains vitreous solid spherical particles, is seen in FA (Figure 3 (d)). Genospheres and plenospheres were also recognized (Figure 3 (e)). For CPC, irregular angular solid particles of varying sizes are seen, similar to UtSCBA (Figure 3 (f)).

A review by [35] demonstrates SEM-EDX patterns to study morphological properties of both the treated and untreated rice husk ash (RHA), palm oil fuel ash (POFA), and sugarcane bagasse ash (SCBA) blended concrete. Previous studies have shown that the amount of amorphous silica, unburned carbon, and morphology of RHA, POFA, and SBA can be controlled by the grinding technique and duration [35]. In contrast, the incineration temperature and duration can affect the morphology of TRHA, TPOFA, and TSBA (Jittin 2020). RHA, POFA, and SBA generally have low amorphous silica concentration, but TRHA, TPOFA, and TSBA have significant pozzolanic reactivity. However, amorphous SiO_2 in TRHA, TPOFA, and TSBA may convert to crystalline SiO2 at high combustion temperatures or over extended periods, lowering their pozzolanic activity [36]. RHA, POFA, and SBA have porous microstructures, as demonstrated by SEM images [37]. Meanwhile, the SEM picture in Figures 4 (a) and (b) shows that TRHA has a microporous cellular structure.

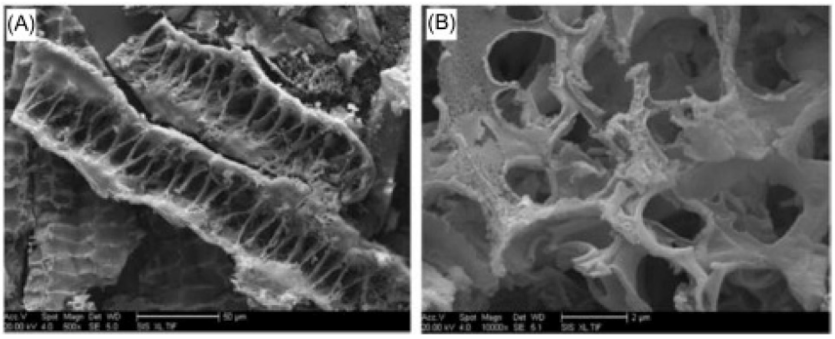

Figure 4. Scanning electron micrograph of RHA (a) 500 × magnification and (b) 1000 × magnification [37]

4. ENGINEERING PROPERTIES

Engineering characteristics, considered in general, are observable and measurable and affect the conduct. They are essential for analyzing and designing engineering and performance components, systems, or processes. The paper discussed several tests that will be used to observe the concrete performance with partial cement replacement.

4.1 Setting Time

The initial setting time test is essential for cement concrete's transportation, placement, and compaction. The initial setting time duration is required to delay the process of hydration or hardening, while the final setting time is when the paste completely loses its plasticity. According to ASTM C191 (2013), the initial and the finished time were taken with a Vicat needle [38]. Measurements of volumetric autogenous strain were carried out by monitoring the weight of cement paste samples stored in elastic membranes and immersed in buoyancy liquid (distilled water). Experiments were performed by volumetric in a climate room at 23 ± 0.2 ° C, and the detailed procedure was given by [39]. [40] experimentally investigated the properties of cement pastes and mortars with 2.5%, 5.0% and 7.5% of nano-MgO. The submerged weight of the paste sample was measured and reported automatically every 1 minute by controlling the program from the initial setting time after casting up to approximately 48 h [41, 42]. The effects of the initial setting, the final setting, and the time setting (the period between the final and the initial setting times) are shown in Figure 5.

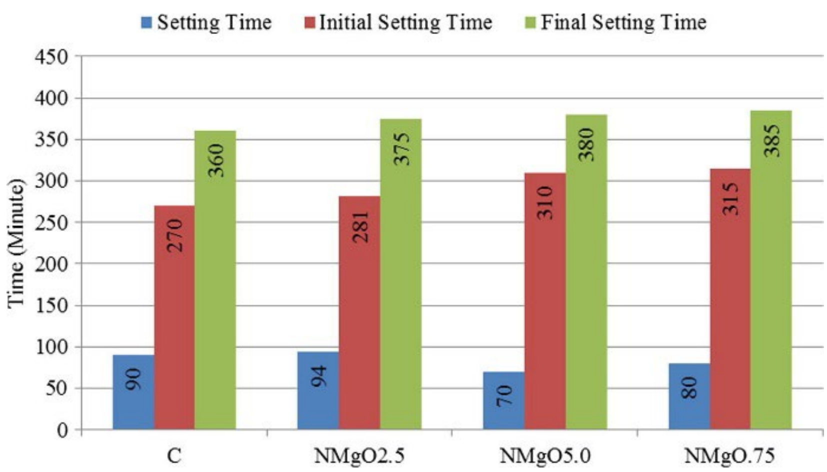

Figure 5. Setting time of pastes with 2.5%, 5.0%, and 7.5% nano-MgO compared to the control paste [40]

4.2 Compressive Strength

In their report [43], claimed that the time interval between the first and last settings (defined as ʈ) was a significant factor in determining the mechanical strength of cement mortar containing chemical accelerators. The relationship between crack and strength was shown in further microstructure analysis. Shorter timing caused the other crack and decreased mechanical strength during the set time, which also occurred during pre-healing. The chemicals can accelerate the hydration reaction of cement, although their reactions during the stated time are not reduced.

One of the most essential and valuable properties is the compressive strength of concrete. Concrete is used as a building material to withstand compressive stress. Although compressive strength is important in areas with tensile or shear strength, it is used to estimate the desired properties [43]. [40] conducted a compressive test on high-performance cement paste and mortar. Their research found that at the age of seven days, the concrete's compressive strength decreased when nano-MgO was used, but with the rise in nano-MgO of 7.5 % dosage, the strength was marginally increased [44]. The strengths of mortars at 7 and 28 days are shown in Figure 6. The result shows that the compressive strength of the MgO mortar remained lower at early ages (7 days) compared to the control specimen, but a higher compressive strength was observed at later ages. This is because the $Mg(OH)_2$ crystals formed as a result of a chemical reaction with water of nano-MgO accrete with curing time, and these crystals grow up over time, fill the pores of cement paste, and make the structure denser with time [44]. The addition of nano-MgO is generally said to increase the compressive strength for 28 days [44, 45]. [45] also report that the compressive strength of the w / b = 0.60 mortars increased to up to 28 days with rising concentrations of ultrafine MgO but decreased at 90 and 180 days due to seed and coating effect on cement particle surfaces. The high level of activity and seed effects could increase the mortar compressive strength containing ultrafine MgO at an early age [45].

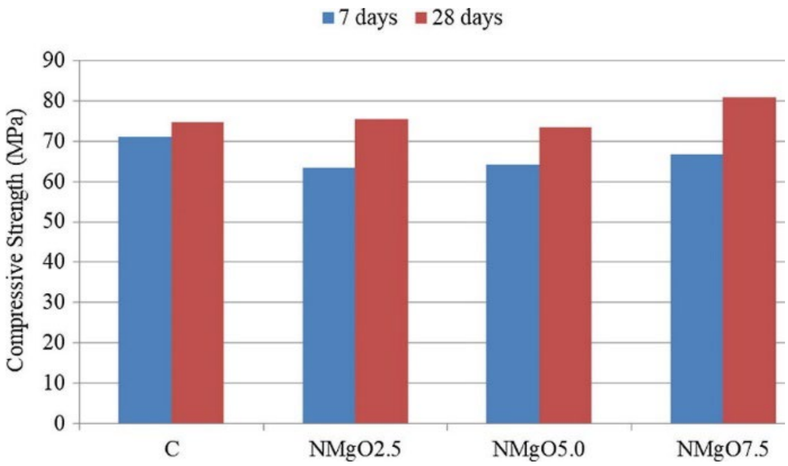

Figure 6. Compressive strength results for C, NMgO2.5, NMgO5.0, and NMgO7.5 samples

4.3 Water Permeability

[46] performed a water permeability test to characterize the saturated concrete's capacity to transport water in a hydraulic gradient [47]. The permeability tests based on Darcy's law are typically calculated in the laboratory and can be defined as follows:

$$k = \frac{\text{QL.}}{\Delta \text{ØS}} \tag{1}$$

In which ΔØS is the hydraulic potential difference, S (L^2) is the cross-sectional area perpendicular to the flow direction, L (L) is the length of the flow path, and Q (L^3 T^{-1}) is the flow rate. [48], through their study on the water permeability of mortar, incorporated with Rice Straw Ash prepared by burning at a controlled temperature of 600 ∘C. The study uses different percentages of replacement levels than the control specimen made by ordinary Portland cement only. A stable flow technique was applied to check the permeability of the mortar (as per the ASTM C1585-13). By calculating the amount of water through the sample, the water permeability coefficient was determined and measured using Darcy's law and equation of continuity. Figure 7 indicates the results at age 14 and 28 days and is divided into two regions [48]. Region I reflect mortar with compressive strength and permeability at the age of 14 days, and region II reflects mortar for 28 days.

The present study [49], investigated the impact of nano-silica inclusion on the mechanical properties and permeability characteristics of cementitious composites using a variety of accessible nanomaterials, including fly ash, silica fume, nano-alumina and nano-silica. For a nominal mix of concrete (Grade M20), cement replacement ranges from 0.5 per cent to 2.0 per cent. According to Indian Standards, experiments were conducted to gain an understanding of the mechanical and permeability properties of concrete. At 28 and 56 days, the reference mix's permeability was 6.197 × 10−10 m/s and 5.697 × 10−10 m/s, respectively. According to the experimental results (Figure 8), permeability is found to decrease at 28 and 56 days after the addition of nano-silica. This is because, in comparison to specimens with 0.0 per cent nano-silica composite, the size and quantity of pores have decreased in specimens containing 0.5 per cent nano-silica [49].

Additionally, specimens with replacement of 1.5% and 2.0% nano-silica exhibit comparable permeability and are the lowest. It could be because the inclusion of nano-silica appeared to have led to the development of more C-S-H gel. This implies that nano-silica functions as a filler to enhance the internal microstructure of cement paste and as a stimulant for cement hydration. Consequently, the microstructure of the nanocomposite became less porous and more uniform [49].

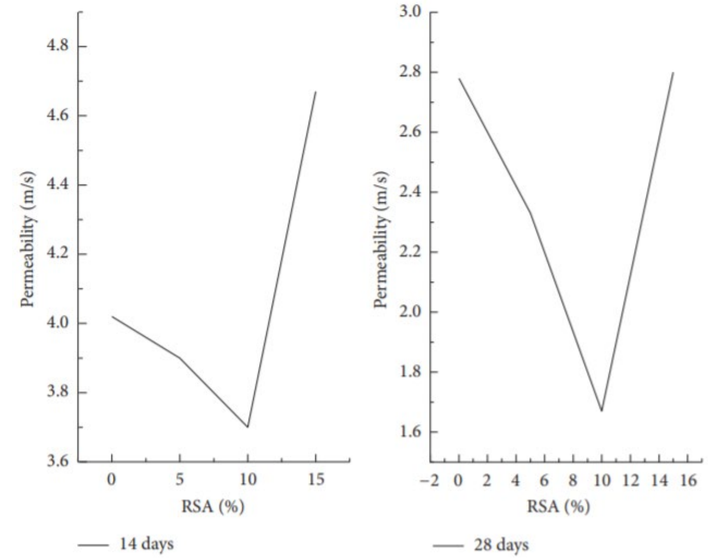

Figure 7. Permeability at 14 and 28 days [48]

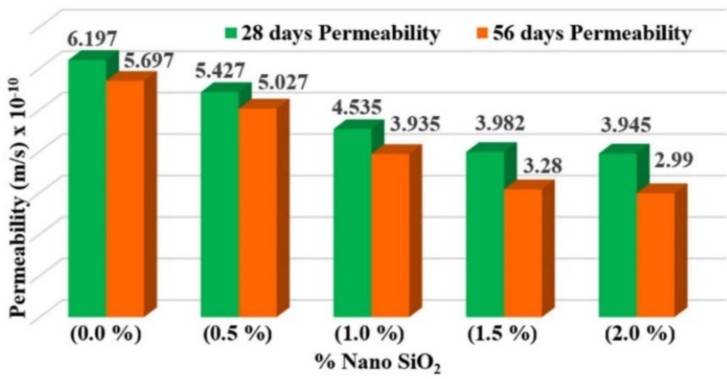

Figure 8. Permeability of cementitious composite with varying percentages of nano-silica [49]

4.4 Field Emission Scanning Electron Microscopes (FESEM)

[50] investigated the physical, chemical, and engineering properties of some tests to study the feasibility of utilizing palm oil clinker (POC) as fine aggregates in mortar. To make sure they meet the requirements for self-compacting mortar (SCM) and are in the high flowability mortar region, a fresh property test was conducted. Microstructure studies were performed on mortar specimens using SEM-EDX to learn more about the characteristics and POC interface with cement paste.

As observed in Figure 9, the porous nature of POC appears to be eminent at a larger magnification. Additionally, irregularities in the POC's shape can be seen. Many voids are on the surface, with an average size of 1 mm to 500 µm. Figure 10 shows the micro-pores that exist on the surface of the POC aggregate at 20 µm magnification. POC aggregate is light mostly due to the large number of voids and pores. The mortar's load-carrying capacity is significantly impacted by these pores. Furthermore, the aggregate-to-aggregate packing level and aggregate-paste border may be affected by the irregular form of the POC aggregate. Significant quantities of silica, aluminium, magnesium, and iron were found in the chemical composition of the graphs from the EDX investigation, which confirmed the chemical traces [50].

The magnification for SEM-EDX analysis is 10,000x for every specimen. Strength development is attributed to a dense structure made of C-S-H gel and increased calcium content in its oxide state, as seen in Figure 11 (a). Figures 11 (b) and 11 (c) demonstrate how glass powder fills the matrix as a filler. The dense and rich C-S-H gel that envelops the aggregates, cement, and glass particles increases the compressive strength. The development of pores and microcracks can be observed in Figures 11 (d), 11 (e), and 11 (f). This phenomenon may be attributed to the high silica content in glass, which promotes silica gel formation with a strong affinity for water. The water absorbed by the silica gel expands and exerts pressure on the interface between glass particles and cementitious materials, ultimately forming cracks and pores in the matrix. According to the study, EDX analysis shows a decrease in the calcium-to-silica ratio, an increase in the formation of C-S-H compounds, and an increase in the silica content [52]. These compounds are responsible for controlling the pozzolanic reaction, and the reduction in the calcium-to-silica ratio may reduce the concrete's overall strength [51]. Table 7 includes tabulations of the remaining elements of GPS0 and GPS1. The chemical composition of glass makeup results in a slightly higher calcium-to-aluminium ratio.

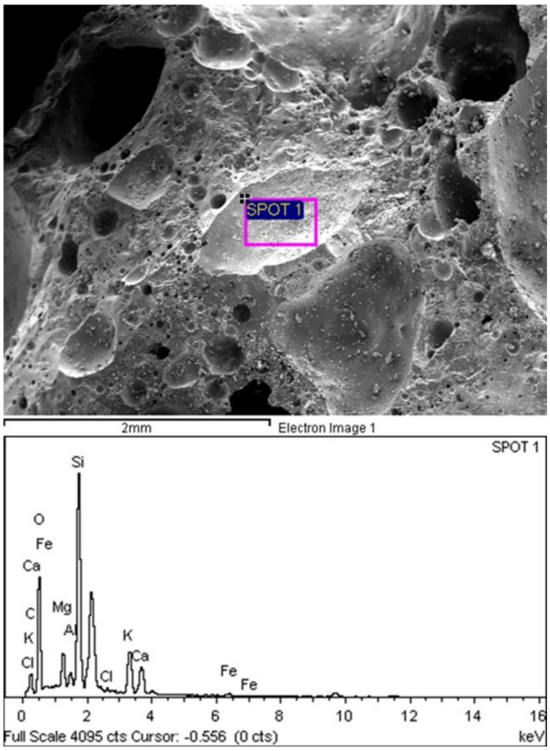

Figure 9. POC aggregate micrograph at a larger magnification [50]

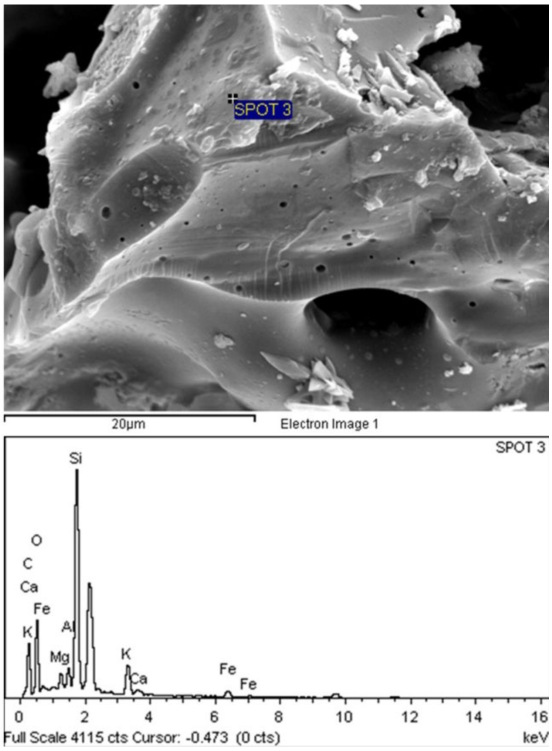

Figure 10. Presence of micro-pores within internal POC aggregate structure [50]

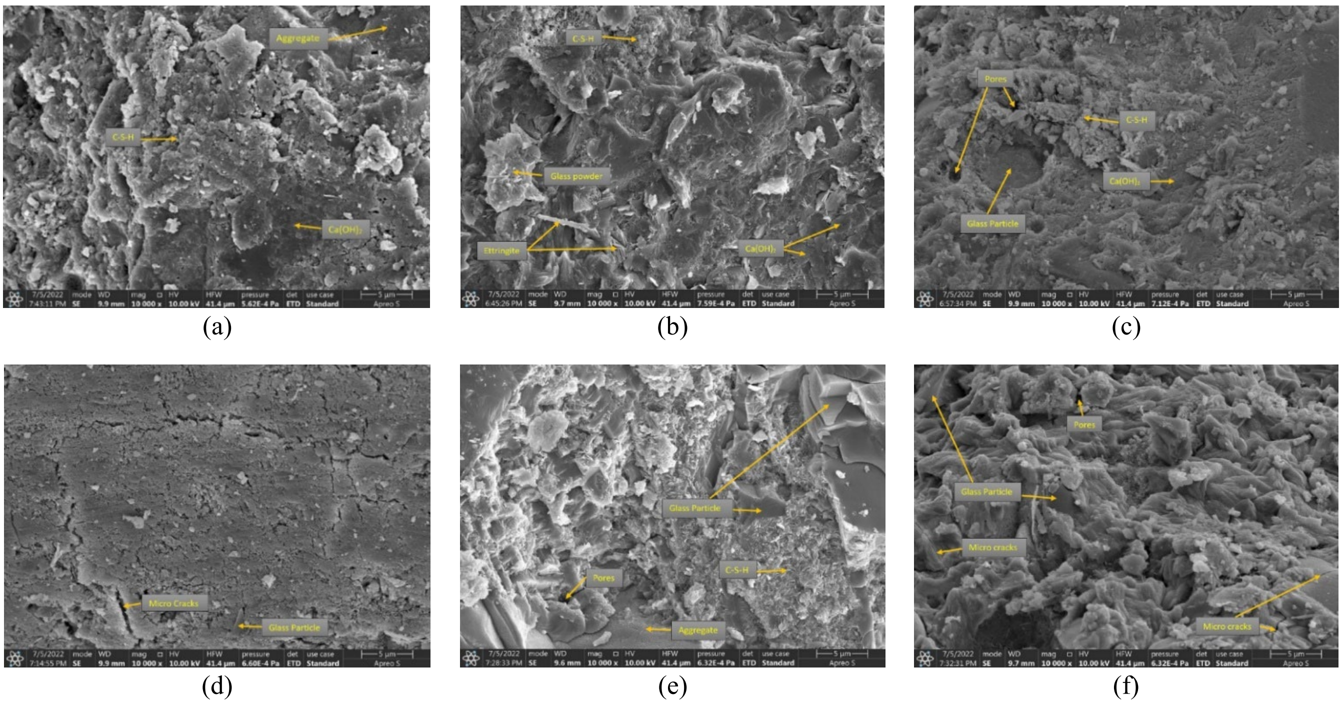

Figure 11. S.E.M. image of; (a) GPS0, (b) GPS1, (c) GPS2, (d) GPS3, (e) GPS4 and (f) GPS5 [51]

Table 7. Elemental composition through EDAX analysis for GPS0 and GPS1 [51]

Element	Atomic weight (%)	
	GPS0	GPS1
C	33.15	30.37
O	47.65	46.30
Na	0.07	0.03
Mg	0.26	0.11
Al	1.34	4.35
Si	4.57	13.22
K	0.15	0.21
Ca	11.51	5.20
Fe	1.32	0.41

5. SUMMARY OF PREVIOUS STUDY

An assessment of previous studies revealed that many types of agricultural and industrial waste were used as partial cement replacements in mortar or concrete. However, this paper will summarize the previous research that used replacement material from agricultural waste in mortar or concrete. The types of agricultural waste have been prepared through different treatment and extraction processes. Table 8 below shows the types of agricultural waste used as a cement replacement, including the treatment method and extraction process involved. Table 9 shows the types of replacement for OPS in concrete and mortar only.

Table 8. Types of agricultural waste used for cement replacement

Author/ Material	Treatment	Extraction process	Review
Rice Husk [53]	Pretreated with HCl. Remove silica with 15 wt. % Na2CO3 solution.	Continuous flash pyrolysis at 500 °C	Recover 88 % of silica content.
Corn Cob [54]	A 10.0 g sample of CCA was stirred in 60 mL of a 1 N NaOH solution.	Dried in an air oven at 110 °C for 24 hours. Burned inside a programmable furnace at 700 °C and heated at 5 °C/min.	Silica extracted from the residue.
Combination of rice husk, bamboo leaves, sugarcane bagasse and groundnut shell [55]	Treated with 1 M NaOH to form sodium silicate, then with 6 M H_2SO_4 to precipitate silica	Sintered at 900 °C for 7 hours	Silica varies between 52 % and 78 %.
Palm Ashvia [23]	-	Treated with Citric Acid leaching treatment method at 700 °C, 60 min reaction time, the concentration of citric acid of more than 2 %.	Silica extracted more than 90 %.

Table 9. Types of replacement for oil palm shell

Author	Type of Replacement	Treatment	Review
[56]	Aggregates for high-strength, lightweight concrete	Heat treatment at two temperature settings (60 and 150 °C) and duration of heat treatment (0.5 and 1 h).	The workability of the OPSC increases with an increase in temperature and duration of heat treatment of the OPS aggregate. It can be used as a new eco-friendly alternative method to enhance HSLWC.
[57]	Natural aggregate in lightweight concrete	Five treatments on physical and mechanical properties of concrete to reduce the hydrophilic behaviour of OPS or to modify the OPS surface.	Lime treatment (CH) on OPS showed good improvement in the mechanical properties of concrete compared to untreated OPS.
[58]	Oil palm shell ash as cement mortar	No treatment	The deflection, load to failure, time to failure of compressive strength and flexural strength of all specimens have significantly been improved.
[59]	As coarse aggregate in lightweight concrete	The treatment method by Okafor et al. was followed.	OPS concrete fulfils the requirements of structural concrete, has good workability with superplasticizers, and meets the requirements of BS and ASTM for compressive strength.

Table 9. (cont.)

Author	Type of Replacement	Treatment	Review
[60]	As a concrete aggregate replacement	Treated raw OPS with 20 % PolyVinyl Alcohol (PVA.)	PVA-treated OPS concrete significantly improved its strength compared to raw OPS concrete. The 28-day compressive strength for PVA-treated OPS can reach up to 33.53 MPa and shows good potential as an aggregate alternative for construction purposes.

6. CONCLUSIONS

This study examined earlier research on the pozzolan material and technique for enhancing the pozzolanic performance of industrial and agricultural wastes in concrete. The following conclusions can be made:

a) Using agricultural and industrial waste as replacement material in concrete will reduce the high demand for natural resources.
b) Previous research used several types of pre-treatment methods to increase silica and lime content and the pozzolanic reactivity to fulfil the minimum requirement of the pozzolanic standard.
c) The engineering and mechanical properties of concrete are essential in measuring the strength and durability of the modified concrete for an extended period.

7. RECOMMENDATIONS

The following recommendations can be made:

a) Replace a higher percentage of replacement material in concrete by investigating the pretreatment method to improve silica content and pozzolanic reactivity without sacrificing the strength of concrete.
b) Diversifying the use of agricultural and industrial waste material to replace concrete material.

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AUTHOR CONTRIBUTIONS

Shuhaimi Shaedon: Data curation, Writing - Original draft preparation
Azrina Abd Aziz: Supervision, Writing - Reviewing and Editing
Rahimah Embong: Supervision

DATA AVAILABILITY STATEMENT

The data used to support the findings of this study are included in the article.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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