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Assessment of Microstructural Characteristics in Cement Matrices with Volcanic Ash and Nano-Silica Substitutions

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Abstract

This study explores the microstructural behavior of cementitious matrices incorporating volcanic ash (VA) and nano-silica (NS) as partial replacements for ordinary Portland cement, with the aim of enhancing both sustainability and material performance. Three mortar mixes—control (45C), VA (45CVA30), and NS (45CNS2)—were analyzed using nitrogen gas adsorption, scanning electron microscopy (SEM), and thermogravimetric analysis (TGA). Results showed that NS significantly improved matrix densification and reduced total porosity, while VA demonstrated less refinement in pore structure. SEM images revealed a denser morphology in the NS mix with well-developed calcium silicate hydrate (C-S-H) phases, whereas the VA sample exhibited a more porous microstructure. TGA results confirmed consistent decomposition of calcium hydroxide ($Ca(OH)_2$) across all mixes, indicating similar hydration progress, though the NS-modified mix showed greater thermal stability and reduced mass loss associated with portlandite decomposition. Additionally, BET surface area analysis revealed a notable reduction in the NS mix over time, highlighting a refined and compact pore network. These findings demonstrate the potential of nano-silica as an efficient supplementary cementitious material for developing sustainable, high-performance cement matrices with improved microstructural integrity.

Keywords: calcium silicate hydrate; gas adsorption; SEM; supplementary cementitious materials; thermogravimetric analysis

1. Introduction

Concrete production contributes significantly to global CO_2 emissions, largely due to the high demand for ordinary Portland cement (OPC). To mitigate this impact, supplementary cementitious materials (SCMs) such as volcanic ash (VA) and nano-silica (NS) are increasingly employed (Lothenbach et al., 2011; Lea & Hewlett, 2004). These materials can refine pore structures, improve hydration products, and enhance durability (Hasan & Nihal, 2014; El-Didamony et al., 2017). Microstructural studies using advanced characterization techniques provide insights into how SCMs influence the morphology and stability of hydrated cement

matrices (García Juenger & Jennings, 2001; Xu et al., 1999). This study focuses on the microstructural performance of three mixes: control OPC (45C), volcanic ash replacement (45CVA30), and nano-silica modification (45CNS2). Analytical techniques include scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and nitrogen gas adsorption (Blaine & Valis, 1949; Bodor et al., 1970).

2. Objectives and Hypothesis

The objective of this study is to evaluate the microstructural characteristics of cement matrices modified with volcanic ash (VA) and nano-silica (NS) through three representative mixes: control (45C), VA replacement (45CVA30), and NS modification (45CNS2). The study aims to assess how these supplementary cementitious materials influence pore structure, hydration products, and overall morphological development using nitrogen gas adsorption, scanning electron microscopy (SEM), and thermogravimetric analysis (TGA). This work provides early-age microstructural evidence to support the use of VA and NS as sustainable additives capable of enhancing the performance of cementitious systems.

It is hypothesized that nano-silica, due to its nano-scale fineness and high pozzolanic reactivity, will significantly refine the pore structure, increase matrix densification, and reduce total porosity compared to both the control and VA-modified mixes. Volcanic ash, being a slower-reacting natural pozzolan, is expected to exhibit only moderate refinement in early ages but may demonstrate stronger improvement at extended curing periods beyond 28 days.

3. Background

The demand for sustainable and advanced concrete materials has increased significantly in recent years. Supplementary cementitious materials (SCMs) are increasingly utilized in the construction industry as partial or complete replacements for ordinary Portland cement (OPC) to produce durable and environmentally friendly concrete. Several studies, investigated the use of volcanic ash (VA) as a partial substitute for OPC and examined its influence on the mechanical and microstructural properties of cementitious systems. The findings revealed that replacing OPC with up to 30% VA by mass improved concrete strength due to the formation of calcium silicate hydrate (C-S-H) and calcium alumino-silicate hydrate (C-A-S-H) gels. However, mixes incorporating VA exhibited lower early-age strength compared to OPC mixes (Lothenbach et al., 2011; Al-Bahar et al., 2017). Furthermore, results from the KISR research project EU089C: Sustainability of Kuwait's Built Environment Thrust Area A: Nano-Engineered Construction Materials for Durability in Aggressive Environments in Kuwait demonstrated enhanced matrix densification when OPC was partially replaced with VA and/or nano-silica (NS). These additions were found to significantly influence the mechanical and microstructural behavior of cement

paste matrices at the macro, micro, and nano-scale (Kupwade-Patil et al., 2016). In addition, NS and fly ash have been used as additives to enhance the early strength of concrete, acting as filler materials that densify the matrix and accelerate hydration reactions. The reaction between NS and calcium hydroxide promotes additional formation of C-S-H, leading to improved strength and microstructural refinement.

4. Literature Review

Several studies have focused on using the performance of concrete by incorporating multi-scaled ratios of cementitious materials such as nano-silica (NS) and volcanic ash (VA) to achieve enhanced performance with reduced cement content. Properties like porosity, pore size, and pore morphology have been extensively investigated at the nano-scale, while nano-indentation techniques were applied to determine nano-mechanical characteristics **(**Al-Bahar et al., 2017). The influence of replacing ordinary Portland cement (OPC) with VA on pore structure and microstructural properties has been analyzed at various curing stages. Researchers found that the primary hydration products resulting from VA addition include Calcium Silicate Hydrate (C-S-H), Calcium Alumino Silicate Hydrate (C-A-S-H), and Magnesium Silicate Hydrate (M-S-H). However, when the replacement level of VA exceeds 30%, the porosity and void connectivity in the cementitious matrix tend to increase (Kupwade-Patil et al., 2016).

Recent findings also indicate that incorporating 10% silica fume (SF) and 1% nano-silica (NS) enhances the chemical and mechanical performance-particularly density and compressive strength-up to 90 days of curing (Kuwait Institute for Scientific Research, 2015). This improvement is attributed to the formation of additional hydrated silicate compounds resulting from the reaction between NS and calcium hydroxide, which increases the C-S-H content.

Furthermore, researches have studied studied the effect of using ultra-fine sand (UFS), a micro-scale crystalline silica, as a partial cement replacement. Their work showed that substituting 5% of cement with UFS improved the flexural, compressive, and tensile strengths of cement composites (Shebl et al.,2013; El-Didamony et al., 2017). The physical effect of UFS contributes to a denser and more homogeneous matrix through enhanced interfacial bonding and improved particle packing **(**Al-Bahar et al., 2017).

Researchers introduced a methodology that combines SEM–EDS elemental mapping with image-based analysis to directly quantify nanosilica dispersion within hardened cement paste. Their results showed that dispersion measured in solution does not necessarily reflect particle behavior in the cement matrix, where the alkaline environment and ongoing hydration can alter nanosilica distribution. By isolating regions with low Ca/Si ratios and converting them into binary segmentation maps, the authors distinguished pozzolanic C–S–H gel from unreacted phases and nanosilica clusters. Quantitative indicators such as fractal dimension, Delaunay triangulation, and dispersion indices confirmed that poorly dispersed nanosilica forms

agglomerates that limit nucleation and reduce pozzolanic efficiency, whereas the use of a surfactant (Triton X-405) significantly improved dispersion and promoted more uniform secondary C–S–H formation. The study reinforces that nanosilica performance is highly dependent on its micro-scale dispersion state and highlights the importance of rigorous characterization methods in NS-modified cement systems (Sargam & Wang, 2021).

Researchers recently provided a comprehensive explanation of how supplementary cementitious materials (SCMs) interact synergistically with limestone in ternary blended cement systems, offering clear relevance to studies involving SCMs such as volcanic ash or nano-silica. The paper highlights that limestone contributes both physical effects (filler and packing improvement) and chemical effects, primarily through the formation of carboaluminate phases, which stabilize ettringite and refine pore structure. These mechanisms accelerate early hydration, improve matrix density, and enhance long-term durability. It also shows that SCMs with high alumina content such as metakaolin, slag, and certain fly ashes exhibit strong synergy with limestone, significantly increasing early strength and carbonation resistance, while silica-rich SCMs benefit more from improved packing and dilution compensation. Overall, the findings reinforce that ternary systems combining limestone and SCMs can reduce clinker content and CO_2 emissions while enhancing mechanical and durability performance, aligning with modern sustainability requirements (Campagiorni et al., 2024).

Recent study further emphasizes the role of nano-silica in enhancing both the mechanical and durability performance of cementitious materials. Their synthesis of nearly one hundred studies found that nano-silica consistently refines pore structure, reduces water absorption, and improves resistance to chloride and sulfate attack due to its high pozzolanic reactivity and micro-filling capability. These improvements were attributed to accelerated C–S–H gel formation, densification of the interfacial transition zone (ITZ), and reduced connectivity of harmful capillary pores. Importantly, the authors also highlight practical implications relevant to sustainable construction—namely the potential for nano-silica to reduce cement demand through improved binder efficiency and to enhance long-term durability performance, aligning with modern CO_2 reduction strategies in cement composites (AlTawaiha et al., 2023).

5. Scope of Work

Several tasks have been conducted to achieve the objectives of this study:

Task 1. Mobilization and Selection of Materials

This task included performing literature review on partial replacement of OPC with different pozzolanic materials (nano silica and volcanic ash) and their effect on the microstructural properties of cement paste and mortar samples. Collecting and reviewing the relative standard specifications and standard test methods. ordering of required equipment, tools, and materials

when necessary. Insuring that the machines required for this study are calibrated and functioning properly and that tools and moulds are available and in good condition. Mix proportions were decided to perform tests on cement mortar with different percentages partially replacing OPC with nano silica and volcanic ash.

Task 2. Physical and Chemical Characterization of Starting Materials.

This task involves the physical, chemical, and mineralogical characterization of the starting materials such as OPC, NS, and VA using Particle size analysis, and Scanning Electron Microscope (SEM).

Task 3. Mix Design

Following ASTM C 305 (ASTM C305-06, 2016) for mixing using w/c 0.45, OPC was partially replaced with nano and micro materials in different percentages as explained in the following Table 1.

Table 1: Mix Design Details

Mix Number	Mix Description	Mix ID
1	Control (OPC)	45C
2	70% OPC + 30% VA	45CVA30
3	98% OPC + 2% NS	45CNS2

Task 4. Sample Casting and Curing

Cement mortar specimens were cast in standard molds following ASTM C305, demolded after 24 hours, and cured in saturated limewater at 23 ± 2 °C for 7 and 28 days prior to testing.

Task 5. Microstructural Studies

The effect of SCMs on the microstructural and cement hydration behaviour of selected hardened cement pastes and mortar samples were analysed by N2 adsorption, Thermogravimetry (TGA), and Scanning Electron Microscopy (SEM). The prepared specimens were allowed to cure for the desired periods, then keeping them in acetone in an air tight container to stop the hydration process. Samples were kept in desiccators after removing the solvents from the specimens using a vacuum pump.

6. Experimental Work and Material Preparation

Standard procedure for mixing cement mortar ASTM C 305 was followed using different percentage of Pozzolanic materials and added as a percentage from the cement weight. Sample

prepared in molds and cured in water mixed with lime at different ages as shown in Figure 1 according to ASTM C 305 (ASTM C305-06, 2016).

Nano-silica was introduced in a slurry form and dispersed in the mixing water using a high-shear mixer for approximately 2 minutes to prevent agglomeration. Volcanic ash was dry-blended with OPC for 60 seconds before adding water to ensure uniform particle distribution. The volcanic ash used in this study had a median particle size of approximately 18–22 μm. Specimens were demolded after 24 hours and cured in saturated limewater at 23 ± 2 °C until the designated testing ages (7 and 28 days). Hydration of paste samples was stopped by immersing them in acetone immediately after curing, followed by storage in a vacuum desiccator. All microstructural tests (SEM, TGA, and BET) were conducted on three replicate specimens per mix, and mean values were reported.

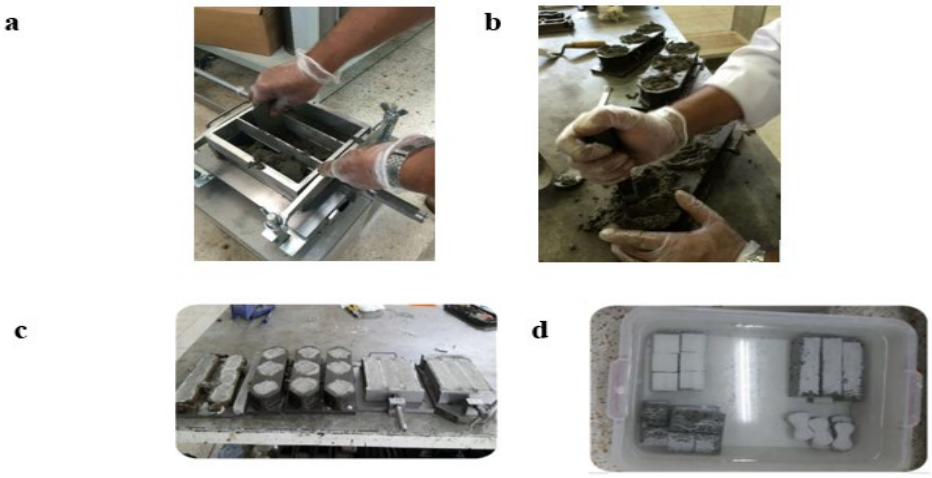


Figure 1: (a) molds preparation, (b) molding, (c) demolding, and (d) curing.

6.1 Microstructural Testing Protocols

Thermogravimetric analysis (TGA) was performed under a nitrogen atmosphere at a heating rate of 10 °C/min from room temperature to 800 °C using approximately 20–30 mg of powdered sample.

Nitrogen adsorption–desorption measurements were conducted after degassing samples at 105 °C for 24 hours under vacuum. BET surface area was calculated over a relative pressure range (P/P_0) of 0.05–0.30, and total pore volume was obtained at $P/P_0 \approx 0.99$.

SEM imaging was performed using a JEOL JSM-6010LA microscope at accelerating voltages of 15–20 kV, with magnifications ranging from 2,000× to 20,000×. Samples were gold-coated prior to imaging.

Nano-silica dispersion was controlled by introducing NS as an aqueous slurry and dispersing it using high-shear mixing for approximately 2 minutes prior to cement addition.

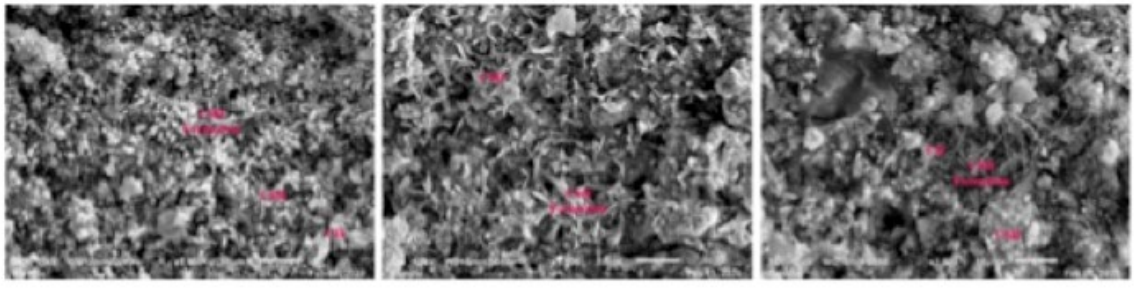
7. Result and Discussion

7.1 Microstructural Studies

The effect on the microstructural and cement hydration behaviour of hardened cement pastes samples (45C, 45CVA30, and 45CNS2) after 7 and 28 days of curing were analysed by Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), and N_2 Adsorption. The prepared cement paste specimens were allowed to cure for the desired periods followed by keeping these samples in acetone in an air tight container to stop the hydration process. Samples were stored in desiccators after removing the solvents using a vacuum.

7.1.1 SEM Analysis

The microstructure of cement paste samples with different percentages of mineral admixtures (0%, 30% VA, & 2% NS) after 7 and 28 days of curing were studied using Scanning Electron Microscopy (SEM) (Figure 2 & Figure 3). SEM analysis was carried out using JEOL analytical scanning electron microscope, JSM6010LA. Samples were placed on carbon tape attached to the sample holder with gold coating. Various hydration products like C-S-H, C-H, and ettringite were identified after 7 and 28 days of curing (Elena & Lucia, 2011; Elena & Lucia, 2012; Lea & Hewlett, 2004).



a b c

Figure 2: SEM images of (a) 45C, (b) 45CV30, and (c) 45CNS2 after 7 days of curing.

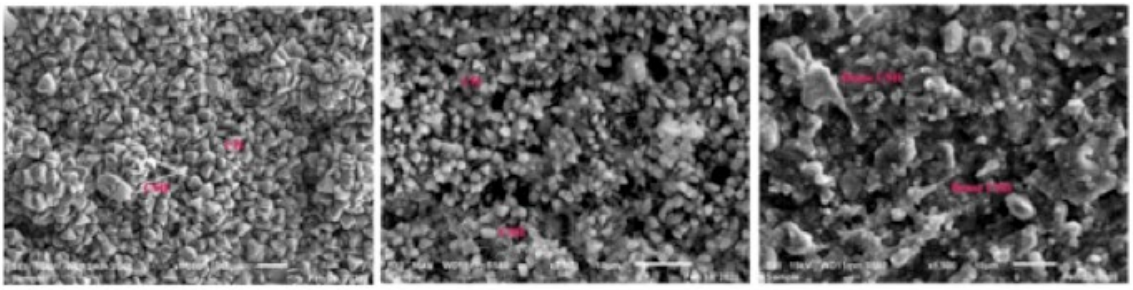


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Figure 1: SEM images of (a) 45C, (b) 45CV30, and (c) 45CNS2 after 28 days of curing.

As the time progresses (after 28 days of curing), much denser morphology was observed for all the samples except for the one with 30% volcanic ash replacement (45CVA30). The addition of Nano silica decreases the pore size and increases the densification of the matrix. These mineral admixtures not only act as nucleation sites for the C-S-H molecules to grow (filler effect) but also react with C-H to form more C-S-H (pozzolanic reaction).

7.1.2 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is a method of thermal analysis that measures changes in mass in relation to changes in temperature (at a constant heating rate). Mass, temperature, and time are considered as base measurements in thermogravimetric analysis, while many additional measures may be derived from these three base measurements. The TGA curve gives information on the changes in sample composition, thermal stability, and kinetic parameters for chemical reactions in the sample. The differential thermal analysis (DTA) curve can be used to show the point at which mass loss is most apparent. During cement hydration, the components present in the cement react with water to form calcium silicate hydrate (C-S-H) and calcium hydroxide ($Ca(OH)_2$) as the major reaction products.Typical TGA graphs of the cement paste samples after 7 and 28 days of curing (0.45 w/c) are shown in Figure 4 From the TG curves it can be seen that, each curve consists of three different phases corresponding to three different mass losses. The first mass loss is between 125 to 380 ^oC corresponding to the dehydration of calcium silicate hydrate (C-S-H), ettringite (Aft) and calcium aluminate hydrate. The second phase is noticed between 415 and 490 ^oC attributed to the decomposition of calcium hydroxide ($Ca(OH)_2$). $Ca(OH)_2$ (a) decomposition peak can be seen at (b) the same temperature range for all samples, which indicates that there is no major change in the phase composition. Finally, the third mass loss is noticed between 560 and 735 ^oC due to the decomposition of carbonates presented in the cement paste sample (Hasan & Nihal, 2014; Patil et al., 2016).

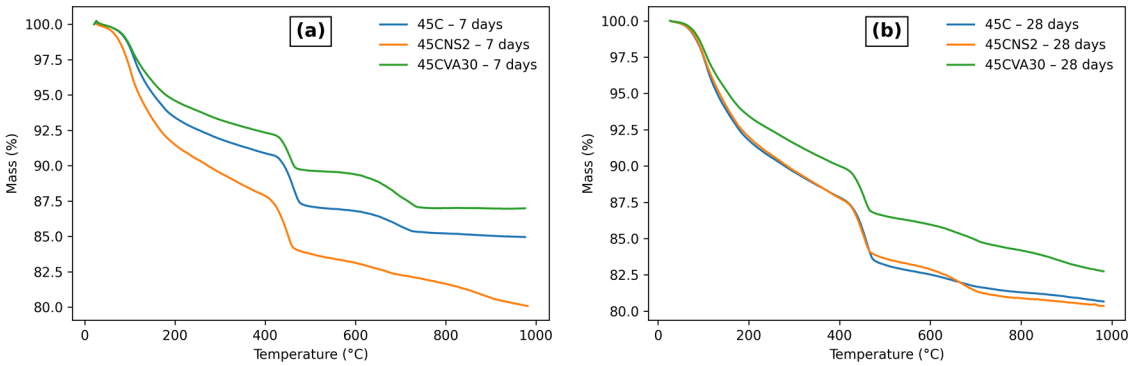

Figure 4: TG curves of cement paste samples (0.45 w/c ratio) after (a) 7 and (b) 28 days of curing.

Mass loss values were calculated from the TG curves as the difference between the sample mass at the beginning and end of each characteristic temperature interval. The ranges 125–380 °C, 415–490 °C, and 560–735 °C correspond to dehydration of C–S–H and aluminate hydrates, decomposition of calcium hydroxide, and decomposition of carbonates, respectively.

As summarized in Table 2, the nano-silica-modified mix (45CNS2) exhibited a lower mass loss associated with $Ca(OH)_2$ decomposition compared to the control, indicating increased pozzolanic consumption of portlandite, while the volcanic ash mix showed a moderate reduction consistent with its slower reactivity.

Table 2: Mass loss (%) from thermogravimetric analysis (TGA) of cement paste samples at 28 days.

Mix	125–380 °C	415–490 °C	560–735 °C
45C	7.01	4.19	1.2
45CNS2	7.35	3.7	2.04
45CVA30	6.03	3.1	1.61

7.1.3 Gas (N_2) Adsorption Analysis

The physical properties of a substance of almost all materials can be defined by microstructure. Concrete, one of the most used building materials, known to have a highly complex microstructure. The microstructure of porous materials can be defined by several parameters such as specific surface area, cumulative pore volume and pore size distribution. There are different techniques and experimental methods that can be used to determine such parameters. One of them is Nitrogen adsorption, which is commonly used to determine the pore properties of cementitious material by measuring the adsorbed nitrogen by applying varying nitrogen pressure in the form of the adsorption-desorption isotherm.

Gas adsorption analysis was performed using Nitrogen gas on solid chunks (~0.4 g) that were taken from hardened cement paste samples. Brunauer-Emmett-Teller (BET) surface area, adsorption, and desorption isotherms were calculated using the data from the gas adsorption experiment. The samples were weighed before and after analysis. The adsorption-desorption isotherms of 45C, 45CVA30, and 45CNS2 after 7 and 28 days of curing are shown in Figure 5. All the isotherms are of pseudo Type II with H3 hysteresis loop characteristics of open slit, parallel plates, and panel shaped mesoporous pores (Rouquerol et al., 2013; Blaine, & Valis, 1949). This type of isotherms is normally associated with monolayer adsorption followed by multilayer adsorption that starts at point 'B' in the Figure 5 Due to an increase in the thickness of the adsorbed layer, a relatively slow increase in the N_2 adsorption can be observed after the point 'B'. However, the accelerated increase in the adsorption isotherm from point 'C' is due to the well-known pore capillary condensation phenomenon.

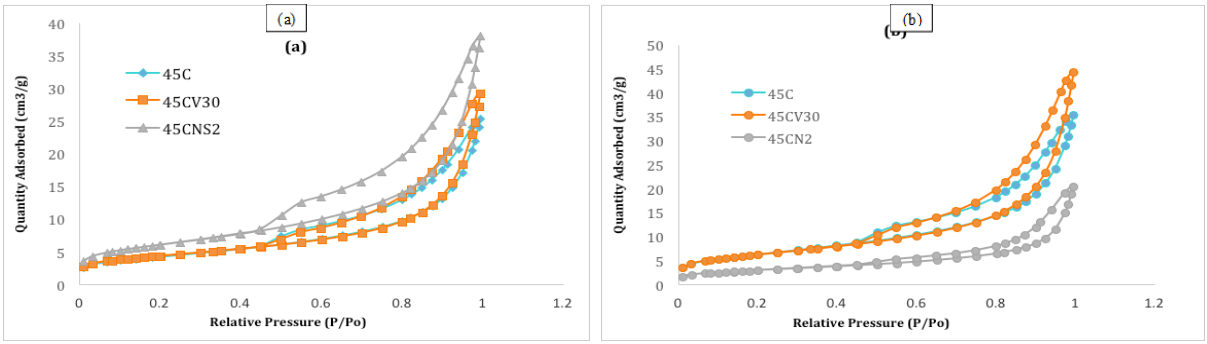

Figure 5: Nitrogen adsorption-desorption isotherms of 45C, 45CVA30, and 45CNS2 after (a) 7 and (b) 28 days of curing.

BET surface area of cement paste samples after 7 and 28 days of curing is shown in Figure 6. For all the samples, except 45CNS2, an increase in the surface area with curing age was observed. A decrease in the surface area from 21.3648 m^2/g after 7 days to 10.3514 m^2/g was noticed for the sample with 2% nano-silica replacement after 28 days of curing. An increase in the surface area of cement paste samples is attributed to the formation of microstructural characteristics of C-S-H gels (Odler, & Chen, 1995; Garci, & Jennings, 2001; Bodor, et al., 1970).

BET surface area was calculated from the linear portion of the BET plot (P/P_o = 0.05–0.30). Total pore volume was taken from the adsorption branch at $P/P_o \approx 0.99$. All adsorption–desorption isotherms were processed using identical software parameters to ensure consistency across all mixes.

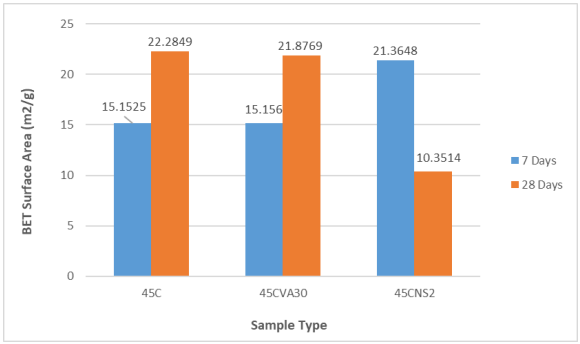

Figure 6: BET surface area for cement paste samples after 7 and 28 days of curing.

8. Conclusions & Recommendations

This study evaluated the microstructural behavior of cement matrices incorporating volcanic ash and nano-silica. Results showed that nano-silica significantly enhanced matrix densification, reduced porosity, and increased thermal stability, while volcanic ash showed limited refinement of pore structure. The results highlight nano-silica's potential as a sustainable additive to improve microstructural integrity in cementitious systems. Despite this, VA remains a valuable additive; being a slower reacting pozzolan, its microstructural and durability behavior shows after 56 to 90 days of curing. To obtain more comprehensive understanding of VA's long term performance, it is recommended that future investigations include testing samples at later curing ages, provided that specimens receive adequate moist curing, proper fineness, and appropriate dosage levels.

The microstructural results obtained in this study provide clear practical value for developing more sustainable cementitious materials. The partial replacement of OPC with volcanic ash and the incorporation of nano-silica both contribute to reducing clinker content, which can directly decrease embodied CO_2 emissions in cement-based products. The refined pore structure observed in the NS-modified mix suggests improved durability performance, including reduced permeability and better resistance to moisture-related deterioration. For volcanic ash, although early-age microstructural refinement is limited due to its slower reactivity, its long-term pozzolanic activity indicates potential strength gain and durability improvements at extended curing ages, supporting its feasibility as a sustainable replacement material.

This study also presents several limitations. The investigation focused on early-age performance (7 and 28 days), limiting the ability to fully assess the long-term benefits of volcanic ash, which typically develops strength and microstructural refinement at later ages. The evaluation was also limited to a single water to cement ratio and specific replacement levels, which may not capture the full range of possible mix optimizations. Additionally, performance indicators such as mechanical strength and durability parameters were inferred from microstructural analysis rather

than measured directly within this paper. Future work should include longer curing periods, direct mechanical and durability testing, and exploration of varied SCM replacement levels to balance early-age performance with long-term sustainability goals.

Building on these findings, this preliminary work should be extended toward the development of nano-concrete composite technology as a practical approach to reducing cement usage in concrete mixes. Such advancement will not only enhance the durability of concrete structures but also contribute to mitigating the global environmental impact of cement production.

From a sustainability perspective, replacing 30% of ordinary Portland cement with volcanic ash can theoretically reduce clinker-related CO_2 emissions by approximately 25–30%, assuming an average emission factor of 0.85–0.90 kg CO_2 per kg of clinker. Although nano-silica is used at a low dosage (2%), its ability to refine the microstructure and enhance binder efficiency may enable further reductions in cement content at the mix-design level. These combined effects highlight the potential role of supplementary cementitious materials in reducing the embodied carbon of cement-based materials.

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